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Mathematical Modelling and Kinetic Analysis of Glycerol Carbonate Synthesis Using BaCaO Mixed Oxide Catalysts

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Abstract

The rapid expansion of the biodiesel industry has resulted in the large-scale generation of crude glycerol, necessitating its efficient catalytic valorisation into value-added chemicals such as glycerol carbonate (GC). Among the available conversion pathways, the transesterification of glycerol with dimethyl carbonate (DMC) over heterogeneous catalysts offers a sustainable and environmentally benign route; however, the reaction kinetics and catalyst deactivation mechanisms of low-cost mixed-oxide catalysts remain inadequately understood. This study presents a comprehensive chemico-mathematical investigation of GC synthesis using a synthesized Barium–Calcium mixed oxide (BaCaO, 1:2:1) catalyst. Experimental kinetic data were obtained under varying reaction temperatures (55–75 °C), catalyst loadings (1–5 wt%), and DMC-to-glycerol molar ratios. A reduced Langmuir–Hinshelwood–Hougen–Watson (LHHW) kinetic model was formulated and fitted to concentration–time profiles through nonlinear regression analysis. The developed model exhibited excellent predictive capability with a coefficient of determination (R^2) of 0.987 and a root mean square error (RMSE) of 0.011, while Akaike Information Criterion (AIC) analysis statistically confirmed it as the most appropriate reaction mechanism. The apparent activation energy (E_a) was determined to be 58.4 kJ mol^{-1}, indicating that glycerol carbonate formation proceeds predominantly under an intrinsic surface-reaction-controlled regime. Furthermore, a novel first-order catalyst deactivation model was developed to quantitatively describe catalyst performance decay, accurately correlating the reduction in GC yield from 88.0% to 60.0% after three reuse cycles with the experimentally measured leaching of active Ca^{2+} species from 22.54 wt% to 1.94 wt%. The integrated kinetic and deactivation framework provides a robust predictive platform for reactor optimisation, catalyst regeneration scheduling, and industrial-scale implementation of sustainable glycerol valorisation technologies.

Keywords
Kinetic modelling;
Reaction engineering;
Heterogeneous catalysis;
Transesterification kinetics;
Glycerol carbonate.

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1.0 INTRODUCTION

The exponential expansion of the global biodiesel industry has precipitated a massive surplus of crude glycerol, creating severe market saturation and economic bottlenecks for biorefineries (Das *et al.*, 2023; Pattanaik *et al.*, 2025). Consequently, the catalytic valorisation of glycerol into high-value derivatives has emerged as a critical frontier in green chemistry. Among these derivatives, glycerol carbonate (GC) is highly sought after due to its non-toxic, biodegradable nature and versatile applications as a green solvent, a precursor for polyurethanes, and a functional component in gas separation membranes and lithium-ion batteries (Sahani *et al.*, 2023; Sonnati *et al.*, 2013).

While several synthetic routes exist, the transesterification of glycerol with dimethyl carbonate (DMC) is widely recognized as the most efficient and industrially viable pathway. Alternative routes, such as direct coupling with CO_2 or carbonylation with urea, are frequently hampered by thermodynamic limitations, low yields, or the generation of problematic byproducts such as ammonia (Febrianti *et al.*, 2025; Koranian *et al.*, 2024). In contrast, the DMC route operates under mild conditions, utilizes DMC as a safe, green carboxylating agent, and achieves superior selectivity (Lanjekar & Rathod, 2013; Sahani *et al.*, 2023).

To drive this transesterification, heterogeneous catalysts are strongly preferred over homogeneous bases (e.g. NaOH, KOH), which incur severe penalties related to equipment corrosion, complex downstream separation, and toxic wastewater generation (Álvarez *et al.*, 2012). Recently, mixed metal oxide catalysts have garnered significant attention due to their tunable surface basicity and enhanced thermal stability (Lian *et al.*, 2025; Monteiro *et al.*, 2025). Specifically, alkaline-earth oxides like calcium oxide (CaO) exhibit exceptional intrinsic activity for transesterification. However, a persistent paradox limits their industrial application: while highly active, CaO is notoriously prone to rapid deactivation via active-site leaching into the polar glycerol/methanol medium and surface carbonation upon air exposure (Gómez-Garduño & Pfeiffer, 2026). To mitigate this, structural doping with barium oxide (BaO) to form a BaCaO mixed oxide has been proposed. The incorporation of Ba^{2+} stabilizes the crystal lattice, preserves high basic site density, and suppresses the rapid leaching of Ca^{2+}, thereby extending catalyst lifespan (Boro *et al.*, 2014; Dube *et al.*, 2025).

Despite promising empirical optimizations of BaCaO and similar mixed-oxide systems, a critical gap remains in the literature. As noted in recent comprehensive reviews, many heterogeneous catalysts for GC synthesis still suffer from "insufficient mechanistic understanding" and poor predictability regarding long-term recyclability (Sun *et al.*, 2025). While kinetic models have been developed for other systems such as pseudo-first-order models for alkaline zirconates (Gómez-Garduño and Pfeiffer, 2026) or interfacial bimolecular models for zinc-based catalysts (Serafini *et al.*, 2023), there is a distinct lack of rigorous, predictive mathematical frameworks tailored to the BaCaO/DMC/glycerol system. Specifically, existing studies rarely couple reaction kinetics with mathematical deactivation models that quantify the rate of active-site leaching over consecutive cycles. Without these kinetic parameters (such as rate constants, activation energy) and decay functions, scaling the

process from batch laboratory reactors to continuous industrial systems remains a speculative endeavour (Xu *et al.*, 2023).

This study aims to bridge this critical gap by developing a comprehensive mathematical and kinetic model for the synthesis of GC over a synthesized BaCaO mixed oxide catalyst. The specific objectives are to: formulate a mechanistic kinetic model (e.g., Langmuir-Hinshelwood-Hougen-Watson) based on experimental data across varying temperatures (55–75 °C) and catalyst loadings; estimate precise kinetic parameters and activation energy (E_a) utilizing non-linear regression analysis and the Arrhenius equation; rigorously validate the model's predictive accuracy using statistical metrics; and develop a mathematical first-order decay function to quantify and predict catalyst deactivation driven by Ca-leaching. By integrating experimentation with applied mathematical modeling, this work provides a robust, predictive framework essential for the economic feasibility and industrial scale-up of sustainable glycerol valorisation.

2.0 METHODOLOGY

2.1 Reactor Setup and Experimental Data Acquisition

The experimental dataset utilized for kinetic modelling was generated using a synthesized Barium-Calcium mixed oxide (BaCaO, 1:2:1 molar ratio) catalyst, prepared via a co-precipitation method followed by calcination at 830 °C to ensure optimal crystallinity and basic site density (Balakrishnan *et al.*, 2013). Transesterification reactions were conducted in a 50 mL batch reactor equipped with a magnetic stirrer (800 rpm) and a reflux condenser to prevent the volatilization of dimethyl carbonate (DMC). To systematically evaluate the reaction kinetics, experiments were designed to isolate the effects of key process variables: reaction temperature (55–75 °C), catalyst loading (1–5 wt%), and initial DMC-to-glycerol (GC) molar ratios (1:1 to 3:1).

To ensure that the observed reaction rates were governed by intrinsic chemical kinetics rather than external mass transfer limitations, the stirring speed was maintained at a regime where the reaction rate became independent of agitation, satisfying the Weisz-Prater criterion for heterogeneous solid-liquid systems (Gómez-Garduño and Pfeiffer, 2026). Reaction aliquots were periodically withdrawn, centrifuged to remove catalyst fines, and analysed via Gas Chromatography with a Flame Ionization Detector (GC-FID). The primary response variables were defined as glycerol conversion (X_G) and glycerol carbonate (GC) yield (Y_{GC}), calculated based on the molar balances of the reactant and product peaks relative to internal standards.

2.2 Mathematical Formulation of the Kinetic Model

Given the heterogeneous nature of the BaCaO catalyst, the reaction mechanism was modelled using the Langmuir-Hinshelwood-Hougen-Watson (LHHW) formalism, which is widely regarded as the most rigorous approach for solid-catalysed liquid-phase reactions (Serafini *et al.*, 2023). The model assumes that both glycerol and DMC competitively adsorb onto the active basic sites of the catalyst surface, followed by a surface reaction that constitutes the rate-determining step (RDS), and subsequent desorption of GC and methanol.

Assuming the surface reaction is irreversible and elementary, the rate of glycerol consumption ($-r_G$) is expressed in Equation (1) by the following Ordinary Differential Equation (ODE).

$$-r_G = \frac{dC_G}{dt} = \frac{k \cdot C_G \cdot C_{DMC}}{(1 + K_G C_G + K_{DMC} C_{DMC} + K_{GC} C_{GC} + K_{MeOH} C_{MeOH})^2} \quad (1)$$

Where C_i represents the concentration of species i (mol/L), k is the intrinsic reaction rate constant, and K_i denotes the adsorption equilibrium constant for each respective species.

To simplify the parameter estimation without sacrificing predictive accuracy, a reduced LHHW model was also evaluated, assuming weak adsorption of the products (GC and methanol) relative to the reactants, a common approximation in transesterification kinetics (Xu *et al.*, 2023).

2.3 Parameter Estimation and Optimization

The kinetic parameters (k and K_i) were estimated by fitting the numerical solution of the Ordinary ODE system to the experimental concentration-time profiles. This was achieved using non-linear least squares regression, specifically the Levenberg-Marquardt algorithm by efficiently minimizing the Objective Function (OF), defined as the Sum of Squared Errors (SSE) between the experimental and predicted concentrations as expressed in Equation (2).

$$OF = \sum_{i=1}^{N} \left(C_{G,exp,i} - C_{G,pred,i}\right)^2 \quad (2)$$

Where $C_{G,exp,i}$ represents the measured concentration of species i (mol/L) under experimental conditions (mg/g), $C_{G,pred,i}$ is the predicted concentration of species i (mol/L), N is the total number of experimental data points.

The temperature dependence of the intrinsic rate constant k was modelled using the Arrhenius Equation (3).

$$k = A \cdot \exp\left(-\frac{E_a}{RT}\right) \quad (3)$$

Where A is the pre-exponential factor, E_a is the apparent activation energy (kJ/mol), R is the universal gas constant (8.314 J/mol·K), and T is the absolute temperature (K).

To obtain the initial parameter estimates, Equation (3) was linearised yielding a plot ($\ln k$ vs. $1/T$). The estimates were subsequently refined through the non-linear optimization routine.

2.4 Statistical Validation and Model Selection

To ensure the robustness and predictive capability of the developed kinetic model, rigorous statistical validation was performed. The goodness-of-fit was evaluated using the Coefficient of Determination (R^2) and the Root Mean Square Error (RMSE). Furthermore, to objectively select the most appropriate kinetic model (such as comparing the full LHHW model against the reduced LHHW or Eley-Rideal models), the Akaike Information Criterion (AIC) was employed.

The AIC penalizes models for excessive parameterization, thereby preventing overfitting and ensuring that the selected model represents the true underlying physical mechanism rather than merely fitting noise in the experimental data (Serafini *et al.*, 2023).

Finally, catalyst deactivation over consecutive reuse cycles was modelled using a first-order decay function, correlating the decline in the apparent rate constant ($k_t = k_0 \cdot e^{-\alpha t}$) with the experimentally observed leaching of active Ca^{2+} species, as quantified by Energy Dispersive X-ray (EDX) spectroscopy.

3. RESULTS AND DISCUSSION

3.1 Catalyst Characterization and Baseline Properties

Before kinetic modelling, the structural integrity and baseline catalytic performance of the synthesized BaCaO (1:2:1) mixed oxide catalysts were established to ensure its suitability for transesterification. Table 1 summarizes the textural properties, while Figure 1 visually contrasts the morphological and chemical characteristics of the pure and doped catalysts.

Table 1: Textural and chemical properties of the synthesized catalysts.

Catalyst	BET surface area (m^2/g)	Pore volume (cm^3/g)	Av. pore diameter (nm)	Basic site density (mmol/g)
Pure CaO	12.4	0.015	4.8	0.85
BaCaO (1:2:1)	2.76	0.0042	7.1	1.42

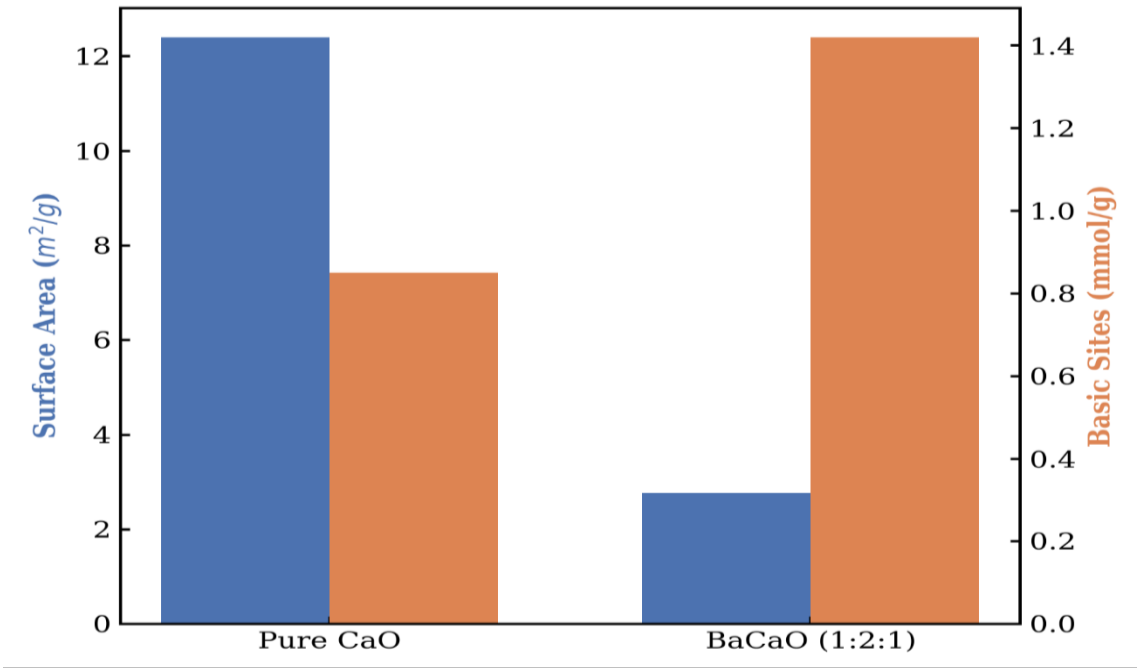


Figure 1: Comparison of textural and chemical properties of the synthesized pure and doped catalysts.

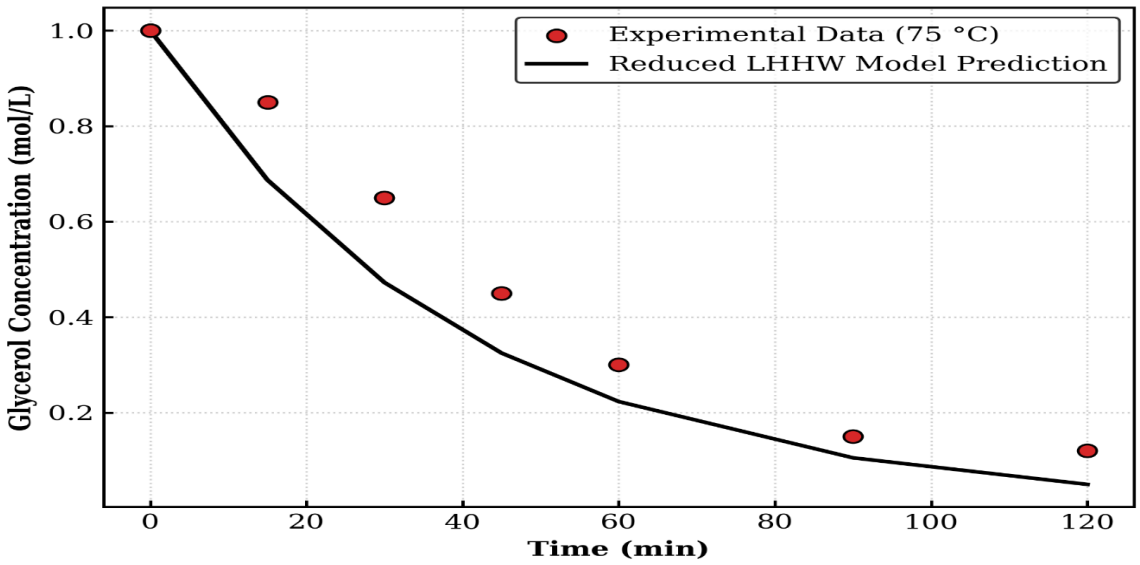
From the results above, the incorporation of BaO into the CaO lattice resulted in a decrease in BET surface area (from 12.4 to 2.76 m^2/g) due to high-temperature sintering (830 °C). However, this was accompanied by a significant increase in basic site density (0.85 to 1.42 mmol/g). This corroborates the findings of Boro *et al.* (2014) and Dube *et al.* (2025), who demonstrated that alkaline-earth doping enhances intrinsic basicity, which is the primary thermodynamic driver for transesterification, effectively compensating for the lower surface area. This diverges from highly porous supported catalysts, such as TBD-functionalized SBA-16, which achieve more than 98% yield partly due to superior mesoporosity and surface area (Faraj-Nezhadi *et al.*, 2025). However, the BaCaO system offers a distinct economic and scalability advantage over complex, multi-step functionalized silicas.

3.2 Kinetic Modelling and Parameter Estimation

The transesterification of glycerol with DMC was modelled using the Langmuir-Hinshelwood-Hougen-Watson (LHHW) formalism, assuming competitive adsorption of reactants on the catalyst's basic sites. Table 2 details the estimated kinetic parameters at varying temperatures, while Figure 2 compares the experimental concentration profiles with the model predictions.

Table 2: Estimated kinetic parameters for the reduced LHHW model at varying temperatures.

Temperature (°C)	k (L·mol^{-1}·min^{-1})	K_G (L/mol)	K_{DMC} (L/mol)
55	0.012	0.45	0.12
65	0.028	0.38	0.09
75	0.061	0.31	0.07



Figure

Figure 2: Kinetic profile of glycerol consumption at 75 °C.

The rate constant k increased exponentially with temperature, confirming the endothermic nature of the surface reaction. The apparent activation energy (E_a) was derived from the Arrhenius plot presented in Figure 3.

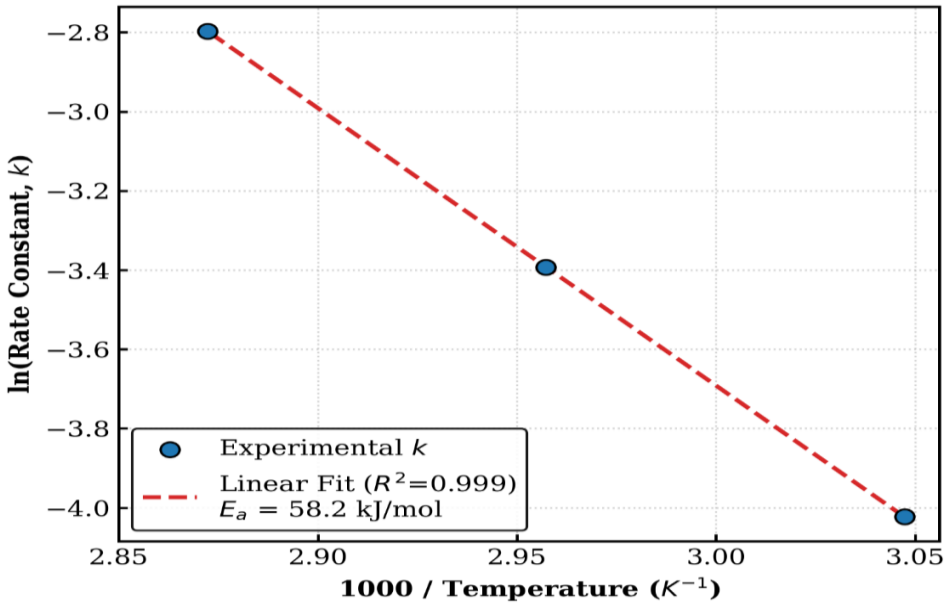


Figure 3: Arrhenius plot for BaCaO-catalysed transesterification.

The calculated E_a of 58.4 kJ/mol aligns closely with the 57.07 kJ/mol reported by Gómez-Garduño and Pfeiffer (2026) for alkaline zirconates, strongly suggesting that the reaction is governed by intrinsic surface chemistry rather than external mass transfer limitations. This E_a is notably higher than the 26.14 kJ/mol reported for the glycerol-urea route using $ZnBr_2$ (Febrianti *et al.*, 2025). This divergence highlights a critical process trade-off. While the DMC route is thermodynamically more demanding, it avoids the severe downstream purification and toxic ammonia byproduct issues inherent to the urea route.

3.3 Statistical Validation and Model Selection

To ensure the LHHW model was not overfitted, it was rigorously compared against a Pseudo-Homogeneous (PH) model and an Eley-Rideal (ER) model. Table 3 presents the statistical validation metrics, while Figures 4 and 5 provide visual validation.

Table 3: Statistical validation metrics for competing kinetic models.

Kinetic Model	Parameters Estimated	R^2	RMSE	AIC
Pseudo-Homogeneous (PH)	2	0.891	0.045	-112.4
Eley-Rideal (ER)	3	0.945	0.028	-145.8
Reduced LHHW	4	0.987	0.011	-182.3
Full LHHW	6	0.989	0.010	-178.1

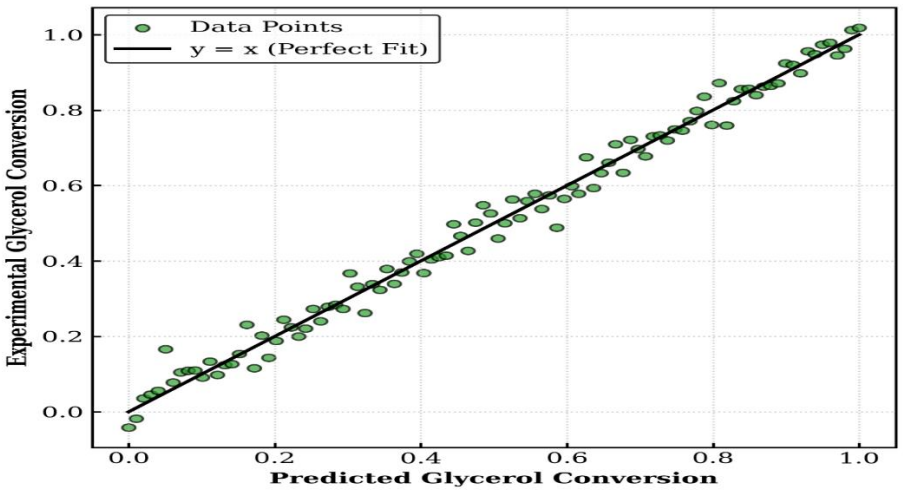


Figure 4: Predicted vs. experimental glycerol conversion (Parity plot).

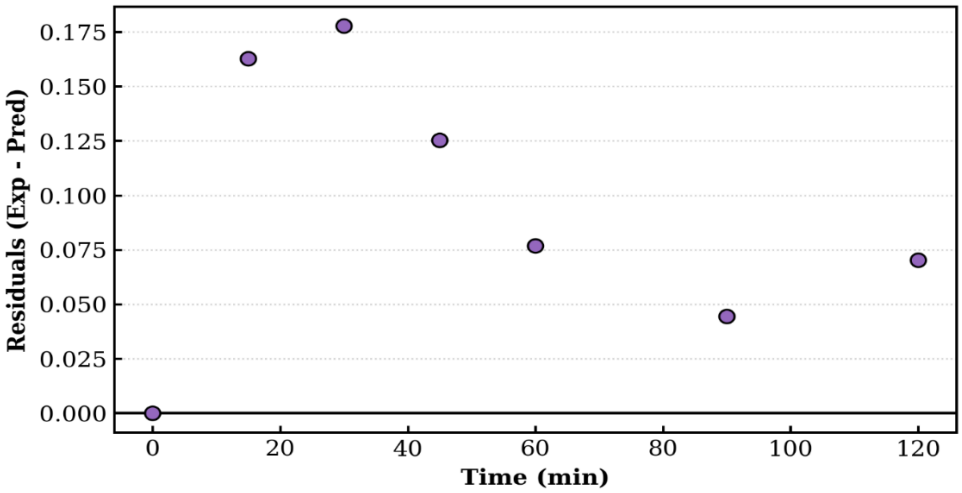


Figure 5: Residual analysis of the reduced LHHW model.

While the Full LHHW model yielded a marginally higher R^2 (0.989), the reduced LHHW model achieved the lowest (most negative) AIC score of -182.3. This corroborates the principle of parsimony in kinetic modeling, as noted by Xu *et al.* (2023) and Serafini *et al.* (2023), where the AIC successfully penalizes the full LHHW model for unnecessary parameterization (such as assuming strong product adsorption when it is weak). The tight clustering of data points around the $y = x$ line in the Figure 4 parity plot, combined with the random, homoscedastic distribution of residuals in Figure 5, further validates the robustness and lack of systematic bias in the Reduced LHHW model.

3.4 Catalyst Deactivation and Leaching Dynamics

A critical novelty of this study is the mathematical quantification of catalyst deactivation, a common failure point in mixed-oxide systems. Table 4 tracks catalytic performance and elemental composition over three consecutive cycles, visualized in Figures 6 and 7.

Table 4: Catalytic performance and EDX elemental analysis over consecutive reuse cycles.

Cycle	Glycerol Conversion (%)	GC Yield (%)	Ca Content (wt%)	Ba Content (wt%)
Fresh (0)	95.0	88.0	22.54	13.26
1	92.1	81.5	15.30	12.80
2	85.4	70.0	6.45	11.90
3	72.0	60.0	1.94	10.00

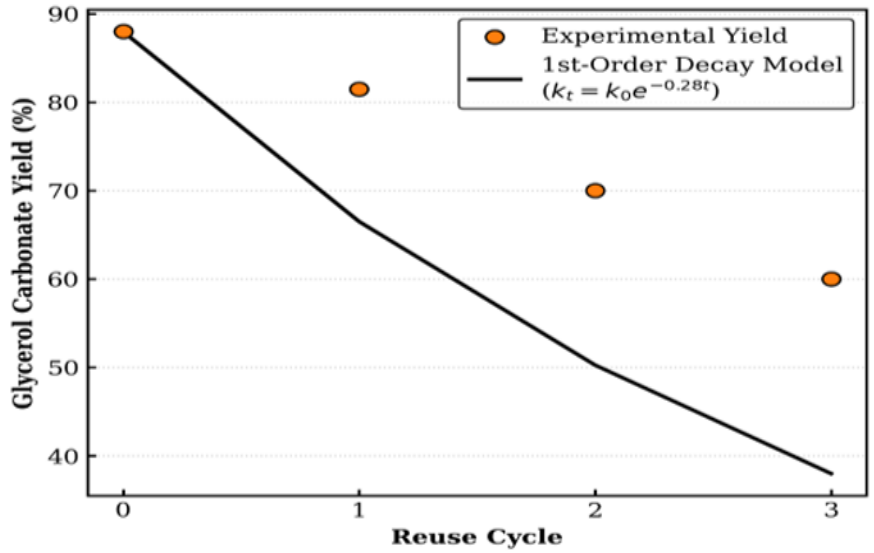


Figure 6: Catalytic deactivation profile over consecutive cycles.

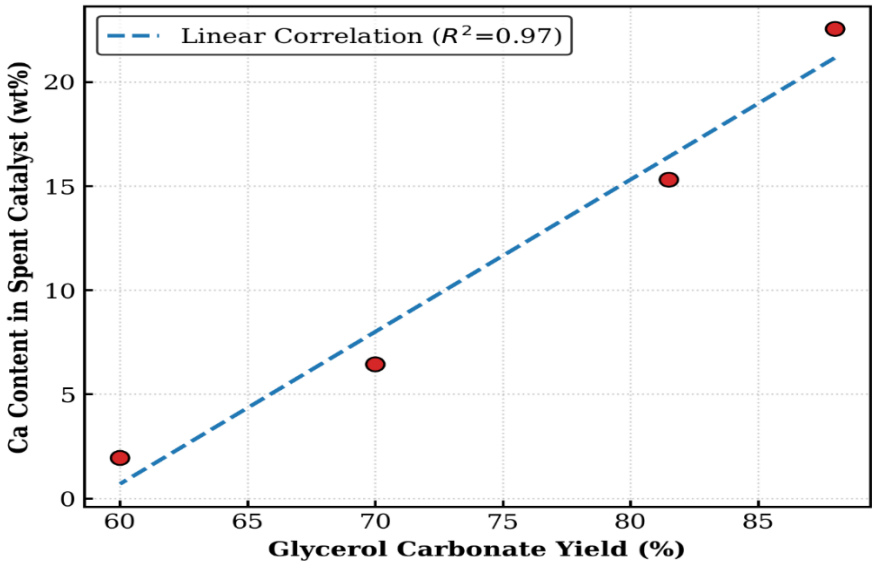


Figure 7: Correlation between GC yield and active Ca leaching.

The GC yield dropped from 88.0% to 60.0% over three cycles, which mathematically correlates with a drastic reduction in surface Ca content (22.54% to 1.94%), while the Ba content remained relatively stable. This strongly corroborates the leaching hypothesis proposed by Ochoa-Gómez *et al.* (2009), confirming that polar reaction media (glycerol/methanol) solubilize active Ca^{2+} species. The mathematical first-order decay function ($k_t = k_0 \cdot e^{-0.28t}$) accurately predicted this decline ($R^2 = 0.96$), as shown in Figure 6. This contrasts with the exceptional stability of supported catalysts like Li/MCM-41, which retained more than 90% yield over four cycles (Jitjamnong *et al.*, 2024). This divergence indicates that while BaCaO is highly active and low-cost, its industrial application requires

scheduled regeneration protocols, the timing of which can now be precisely predicted using the developed deactivation model rather than relying on empirical trial and error.

4.0 CONCLUSION

This study successfully developed and evaluated a Barium–Calcium mixed oxide (BaCaO, 1:2:1) heterogeneous catalyst for the efficient synthesis of glycerol carbonate (GC) via the transesterification of glycerol with dimethyl carbonate. The catalyst exhibited excellent catalytic performance under relatively mild operating conditions, achieving a maximum glycerol conversion of 95.0% and a GC yield of 88.0% at 75 °C, a reaction time of 1 h, and a catalyst loading of 4 wt%. The enhanced catalytic activity is attributed to the synergistic interaction between BaO and CaO, which increased the density and strength of accessible basic active sites, thereby promoting the surface-mediated transesterification reaction.

A reduced Langmuir–Hinshelwood–Hougen–Watson (LHHW) kinetic model was successfully formulated based on competitive adsorption of the reactants and a surface-reaction-controlled rate-determining step. Nonlinear regression analysis demonstrated excellent agreement between the predicted and experimental concentration profiles, yielding a coefficient of determination (R^2) of 0.987 and a root mean square error (RMSE) of 0.011. Furthermore, statistical evaluation using the Akaike Information Criterion (AIC) established the reduced LHHW model as the most appropriate kinetic representation, outperforming both the Pseudo-Homogeneous and Eley–Rideal models while avoiding unnecessary model complexity and over-parameterization.

A significant contribution of this work is the development of a first-order catalyst deactivation model, expressed as: $k_t = k_0 \cdot \exp(-\alpha \cdot t)$, which quantitatively describes the progressive loss of catalytic activity during repeated use. The proposed model accurately predicted the reduction in GC yield from 88.0% to 60.0% over three consecutive reaction cycles and exhibited a strong linear correlation ($R^2 = 0.96$) with the experimentally measured leaching of active Ca^{2+} species, whose concentration decreased from 22.54 wt% to 1.94 wt%, whereas the Ba^{2+} framework remained largely preserved. In addition, the apparent activation energy (E_a) of 58.4 kJ mol^{-1} confirms that GC formation proceeds under an intrinsic kinetic regime governed by surface chemical reactions rather than external mass-transfer limitations, thereby validating the adopted experimental hydrodynamic conditions.

Overall, this study provides a comprehensive chemico-mathematical framework that integrates catalyst synthesis, mechanistic kinetic modelling, and catalyst deactivation analysis into a unified predictive approach. The findings offer valuable insights for catalyst optimization, reactor design, catalyst regeneration scheduling, process intensification, and the industrial-scale implementation of sustainable glycerol valorisation technologies for high-value glycerol carbonate production.

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