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Theoretical Study of the Esterification of Carboxylic Acids with Glycerol in the Presence of Organic Calcium Salts

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Esterification of carboxylic acids with glycerol is an important route to glycerides with potential applications in bio-based fuels and fuel additives, lubricants, emulsifiers, and specialty chemicals. Because glycerol contains three hydroxyl groups, esterification generally proceeds through a sequential reaction network in which monoacylglycerols can form diacylglycerols and, subsequently, triacylglycerols. Product distribution depends on catalyst properties and operating variables, including temperature, reactant ratio, water management, phase behaviour, and reaction time. This review examines the available evidence concerning organic calcium catalysts, with particular emphasis on calcium glycerate and calcium carboxylates. Published studies identify calcium glycerate as a promising catalyst for the esterification of oleic acid with glycerol and report acid conversion of approximately 58–86% after 10 hours under the conditions investigated. The reported induction period and differences in performance relative to calcium oleate suggest that catalyst effects, interfacial phenomena, and product-related changes in the reaction medium influence the observed kinetics. However, the evidence should be interpreted cautiously: structural studies of calcium glycerate and related calcium–glycerol compounds do not, by themselves, establish the identity of the active phase under esterification conditions. The literature on organic calcium salts is also considerably less developed than that on solid acids, zeolites, and enzyme catalysts, particularly with respect to catalyst reuse, deactivation, and scale-up. This review summarises the reaction network, discusses plausible catalytic and transport effects, compares calcium-containing systems with alternative catalytic platforms, and identifies the experiments needed to establish activity, selectivity, and process viability on a comparable basis.

Keywords: *glycerol; carboxylic acids; esterification; calcium glycerate; calcium carboxylates; monoacylglycerols; heterogeneous catalysis; selectivity.*

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I. Introduction

Glycerol is a versatile platform molecule that can be converted into products with properties substantially different from those of the starting material. Its three hydroxyl groups enable the formation of glycerol esters with carboxylic acids, while glycerol's polarity and viscosity also affect reaction engineering. Glycerol esterification is important for producing mono-, di-, and triacylglycerols, as well as oxygenated compounds investigated for fuel-additive, lubricant, and specialty-chemical applications [1–3]. The value of the process depends not only on overall acid conversion, but also on which ester products are formed, how readily they can be separated, and whether the catalyst can be recovered and reused.

The reaction is inherently sequential. A carboxylic acid can react with one hydroxyl group of glycerol to form a monoester and water; further esterification can produce diesters and triesters. These steps do not necessarily proceed at the same rates, and the product distribution at any given time reflects both the forward reaction rates and the removal or accumulation of water. Therefore, high acid conversion does not automatically imply high selectivity towards the desired monoester. This distinction is especially important when monoacylglycerols are the target products, because prolonged reaction times or severe conditions can favour more highly esterified products [4–7].

Catalyst selection is complicated by the physical properties of the reactants. Glycerol is polar, whereas many higher fatty acids and their glycerides are less polar. Depending on temperature, composition, and conversion, the reaction mixture may be biphasic or may change its phase behaviour as surface-active glycerides accumulate. Consequently, measured rates may reflect chemical kinetics, interfacial area, mixing, and mass transfer. Studies of glycerol–oleic acid systems have specifically considered by-product formation and phase-transfer-related autocatalytic effects, illustrating why observed kinetics cannot always be interpreted solely in terms of intrinsic catalyst activity [8].

Calcium-containing materials are of interest because they may be readily available, inexpensive, and chemically related to glycerol and carboxylate species present in the reaction mixture. Studies of calcium-containing compounds in glycerol esterification have compared several calcium precursors and reported promising activity for calcium glycerate [9,10]. Separate structural studies have characterised calcium glyceroxide and examined calcium glycerate as an active phase in other glycerol-conversion chemistry [11–13]. These studies provide a rationale for focused investigation, but their findings should not be conflated: evidence about structure or catalytic activity in transesterification or glycerol polymerisation does not, by itself, establish the mechanism of carboxylic-acid esterification.

The aim of this review is to critically assess the available evidence rather than to claim a universal catalyst ranking. It focuses on (i) the chemistry and selectivity of glycerol esterification, (ii) direct evidence concerning calcium glycerate and related calcium species, (iii) plausible catalyst-speciation and mass-transfer effects, (iv) comparison with more extensively studied catalyst families, and (v) the experimental work needed to establish practical performance.

II. Literature Review

This article is a review based on the supplied bibliographic record and the studies represented in it. The principal works include reports on calcium-containing catalysts for the esterification of glycerol with higher carboxylic acids, structural investigations of calcium glycerate, and studies of active phases in calcium-mediated glycerol conversion. Additional research on homogeneous acid catalysis, zeolites, supported solid acids, mesoporous materials, and immobilised lipases is used for contextual comparison [1–13].

The studies considered are divided into three groups. The first consists of direct esterification studies used to discuss conversion and compare catalysts. The second comprises structural or mechanistic investigations of calcium–glycerol chemistry. The third includes studies of alternative catalyst families suitable for process assessment and benchmarking.

Esterification Chemistry

The overall reaction begins with the formation of a monoacylglycerol (MAG), followed by conversion to a diacylglycerol (DAG), and then to a triacylglycerol (TAG). The simplified sequence can be written as follows:

Glycerol + RCOOH ⇌ MAG + H_2O

MAG + RCOOH ⇌ DAG + H_2O

DAG + RCOOH ⇌ TAG + H_2O

These equations describe the main esterification sequence, but actual product mixtures may be more complex. Depending on the acid and the analytical method, positional isomers, unreacted glycerol, residual acid, and by-products may also be present. Product distribution therefore depends on reaction time and the relative rates of the individual steps, not simply on the initial rate of acid consumption [4–7,14].

Esterification is reversible, and water is formed during the reaction. Water accumulation may reduce the attainable product yield and promote hydrolysis of ester products. Removing water can shift the equilibrium towards ester formation, although the removal method must be compatible with the volatility and phase behaviour of the reactants and products. In solvent-free systems, water removal, temperature, mixing, and the glycerol-to-acid ratio are interdependent design variables rather than independent parameters [5,6,15].

An excess of glycerol is often used when targeting monoester formation because it reduces the likelihood that a newly formed monoester will encounter another acid molecule under conditions favourable to further acylation. This is a useful process principle, not a guarantee: selectivity also depends on catalyst type, reaction time, acid structure, and phase behaviour. Increasing the temperature may accelerate the reaction, but it can also affect equilibrium, viscosity, miscibility, and the relative rates of successive esterification steps.

Aromatic acids and fatty acids present different reaction environments. Aromatic acids, such as cinnamic acid, have been studied under solvent-free homogeneous-catalysis conditions, with kinetic modelling used to describe glyceride formation [16,17]. Fatty acids, including oleic and lauric acids, introduce hydrophobicity and potentially substantial phase limitations. Chain length and unsaturation can affect viscosity, solubility, interfacial behaviour, and the relative accessibility of esterifiable groups. It is therefore unsafe to interpret conversion differences between different acids as a pure measure of catalyst activity.

For higher fatty acids, the ester products themselves may affect the reaction medium. Glycerides can exhibit surface activity and improve contact between glycerol-rich and acid-rich phases [8].

Organic Calcium Salts as Esterification Catalysts

Calcium glycerate is the most prominent catalyst in the studies reviewed. Structural investigations have examined its composition and properties using different characterisation approaches, including studies proposing a chain-like polymeric structure and distinguishing the material from a simple calcium monoglyceride formula [11,12]. This matters because the label "calcium glycerate" should refer to a characterised material rather than to an assumed mixture of calcium species, glycerol, and hydroxide.

However, structural identity is not the same as catalytic identity. A solid present before the reaction may undergo hydration, ligand exchange, dissolution, aggregation, or reaction with carboxylic acids under operating conditions. The active catalytic entity may remain a surface site on the original solid, arise from a transformed calcium-glycerate phase, or include species in the liquid phase. These possibilities require direct investigation during or after esterification rather than inference from the catalyst name alone.

The two studies most directly relevant to the topic investigate oleic-acid esterification promoted by calcium glycerate and compare calcium-containing compounds as catalysts for higher fatty acids [9,10]. The supplied summary reports acid conversion of approximately 58–86% after 10 hours for calcium glycerate under the conditions considered. This range is useful evidence that the catalyst may be active, but it should not be presented as a universal yield or intrinsic activity constant. Conversion depends on the exact reaction temperature, initial acid-to-glycerol ratio, catalyst loading, mixing regime, water management, and analytical method used in each experiment.

In the reported comparison, calcium glycerate becomes more active than calcium oleate after an induction period. The available summary also gives the following activity order for the calcium-containing materials investigated: $CaO < Ca(OH)_2 <$ calcium oleate < calcium ethoxide < calcium butoxide < calcium glycerate [10]. This sequence should be interpreted strictly as the reported ranking in the system studied. It cannot be generalised to every acid, reaction state, or catalyst preparation without matched comparative experiments.

One possible explanation is that organocalcium compounds interact more favourably with glycerol-rich media or form catalytically significant species more readily than oxide and hydroxide precursors. Another possibility is that precursor transformation and interfacial behaviour differ across the series. The observed ranking cannot distinguish between these explanations. Nor does it demonstrate that the most active material will be the easiest to recover, the most selective for MAG, or the most stable over repeated cycles.

Studies of calcium-based catalysts for glycerol polymerisation have highlighted calcium glycerate as a possible active phase [13]. Related studies of calcium oxide and calcium hydroxide in glycerol-containing systems suggest that the initially added precursor may transform under reaction conditions. These findings support a testable hypothesis for esterification: a glyceroxide- or glycerate-like phase may form in situ and contribute to activity.

When applied to carboxylic-acid esterification, however, this hypothesis remains an inference. The presence of free carboxylic acid and the water generated during esterification may affect calcium coordination and phase composition. Accordingly, the active phase must be identified under actual reaction conditions, preferably by complementary analyses of both the solid and solution phases. A single ex situ characterisation may be misleading if the catalyst changes during cooling, washing, drying, or exposure to air.

Carboxylic-acid esterification is commonly promoted by acid catalysis, which facilitates activation of the carbonyl group and subsequent nucleophilic attack by the alcohol. Calcium-containing compounds may display Lewis-acid, basic, or mixed acid–base behaviour depending on their structure and surroundings. It would be premature to assign a single mechanism to calcium glycerate without direct kinetic and spectroscopic evidence. Calcium coordination with glycerol, carboxylate formation, and changes in water concentration may all affect the overall rate.

A further complication is that calcium carboxylates may form through acid–base interactions between a calcium-containing material and the free acid. The resulting species may be less active, more active, or simply differ from the starting catalyst in solubility and interfacial behaviour. A complete mechanistic study should therefore track both organic products and calcium-containing phases rather than relying exclusively on acid conversion.

An induction period is an interval during which the measured rate is initially low before increasing. In glycerol–oleic acid systems, an increase in the concentration of amphiphilic glycerides may improve interfacial contact and reduce resistance to mass transfer. The phase-related autocatalytic effect discussed in the literature provides a plausible basis for interpreting such behaviour [8]. If calcium glycerate exhibits a delayed acceleration relative to calcium oleate, the cause may involve the evolution of interfacial properties, formation of a new catalytic phase, or both.

These explanations should not be treated as mutually exclusive. To distinguish chemical induction from transport effects, experiments should vary agitation rate and interfacial area while keeping the chemical

composition constant. If the apparent induction period changes substantially with mixing, transport is likely to be important. If it persists when external mass-transfer limitations are minimised, catalyst transformation or intrinsic kinetic effects become more plausible. Time-resolved sampling and phase characterisation would help establish the sequence of events.

In heterogeneous and multiphase reaction systems, the measured rate may depend on external mass transfer, internal diffusion, catalyst wetting, and changes at the liquid–liquid interface. A catalyst ranking obtained at one stirring rate or reactor geometry may reflect transport properties as much as chemical activity. This is especially important for viscous glycerol mixtures and long-chain fatty acids.

A reliable kinetic study should test whether the observed rate changes with stirring speed, particle size, catalyst concentration, and phase ratio. It should also report a complete mass balance and determine conversion, yield, and selectivity separately. For example, acid conversion measures the fraction of the initial acid consumed, whereas MAG selectivity describes the fraction of converted acid or formed glyceride assigned to monoester products, depending on the definition used. Without harmonised definitions, numerical comparisons can be misleading.

Temperature affects reaction rate, equilibrium, viscosity, phase behaviour, and catalyst stability. Higher temperatures may accelerate esterification, but they may also increase the rate of subsequent conversion from MAG to DAG and from DAG to TAG. The optimum temperature depends on the target product and catalyst. Reaction time should be considered similarly: the time that maximises acid conversion may not provide the maximum MAG yield or selectivity.

Studies of cinnamic acid and methoxycinnamic acid esterification have investigated solvent-free operation and kinetic modelling at elevated temperatures [16,17]. The supplied source summary reports conditions of approximately 150–200 °C and acid-to-glycerol ratios ranging from 1:3 to 1:9 in one set of examples, with high conversion and substantial monoester selectivity. These values apply to the specific systems reported and should not be transferred directly to calcium-catalysed fatty-acid esterification without verification.

An excess of glycerol may favour MAG formation by reducing the effective probability of further acylation, while water removal favours the forward reaction. Practical implementation is not straightforward. Strong water removal at high temperature may also remove other volatile components, change the local composition, or complicate energy integration. In addition, water may affect calcium speciation, so a condition that improves equilibrium conversion may simultaneously alter the catalyst.

Future studies should report the initial molar ratio, water content of the feed, whether water is removed continuously or only during work-up, and the final water content. These details are necessary for reproducibility and for assessing whether catalyst performance is attributable to the material itself or to a particular process configuration.

Because glycerol and higher fatty acids may form separate phases, the mixing regime determines the available interfacial area. Impeller type, stirring speed, reactor dimensions, fill volume, and viscosity can affect apparent kinetics. A process optimised in a small laboratory vessel may not scale linearly if mixing and heat transfer change. Reporting only the temperature and reaction duration is insufficient for meaningful comparison of multiphase systems.

For practical assessment, the minimum set of operating data should include reactor geometry, stirring speed, catalyst particle-size range, catalyst loading, reactant ratio, temperature profile, sampling procedure, and analytical method. These details are particularly important when comparing calcium glycerate with calcium oleate or with solid-acid and enzyme catalysts.

Comparison with Alternative Catalysts

The literature on glycerol esterification includes homogeneous acids, heterogeneous solid acids, zeolites, supported acid species, mesoporous materials, and lipases. These platforms provide useful benchmarks for evaluating a newer or less extensively studied calcium catalyst. They differ in catalytic mechanism, reaction conditions, product selectivity, sensitivity to water, separation requirements, and potential for reuse.

Homogeneous acid catalysis has been investigated for glycerol esterification with aromatic acids, including kinetic modelling of cinnamate glyceride formation [16,17]. Such systems can provide useful mechanistic and kinetic benchmarks, but separation of the dissolved catalyst from products and neutralisation requirements may add downstream operations. Corrosivity and waste generation may also affect process sustainability. Any comparison with calcium-containing catalysts should consider not only conversion, but also catalyst loading, selectivity, work-up, and waste treatment.

Solid acids offer the practical advantage that the catalyst can be separated from the liquid product by filtration or other solid–liquid operations. Studies have investigated niobium-containing acid catalysts, mesoporous silicas, supported heteropolyacids, and porous polymeric Brønsted acids in glycerol-related

esterification chemistry [18–21]. Zeolites have also been investigated for monoglyceride formation with shape selectivity, illustrating how pore structure and accessibility can affect product distribution [22,23].

These studies do not prove that solid acids are universally superior to organic calcium salts. Rather, they show that catalyst performance should be assessed using multiple metrics: activity, product selectivity, stability, recoverability, resistance to leaching, and feedstock compatibility. The available data on calcium glycerate indicate promising activity, but the supplied evidence does not provide an equally broad basis for process evaluation or scale-up.

Lipases can catalyse glycerol esterification under conditions that may provide useful selectivity, including monoglyceride synthesis using immobilised enzyme systems [24,25]. Enzymatic systems may be sensitive to water activity, temperature, substrate composition, and mass transfer, so the cost and lifetime of the biocatalyst must be considered. Their relevance here is methodological: they demonstrate that high selectivity and catalyst reusability are performance measures distinct from overall conversion.

A fair comparison of calcium catalysts and lipases should use the same acid substrate and clearly report the basis for catalyst loading, reaction time, water activity, product analysis, and number of reuse cycles. Comparisons involving unrelated substrates or different definitions of selectivity should be regarded as qualitative rather than quantitative.

Reliable product analysis is essential because glycerol esterification produces mixtures whose components may have similar physical properties. Acid-value titration can estimate residual free acid, but it does not by itself determine the distribution of MAG, DAG, and TAG. Chromatographic methods should be selected and validated to resolve the relevant products, and reports should indicate whether positional isomers are distinguished. Where necessary, complementary methods such as NMR or mass spectrometry can support structural assignments.

At a minimum, a publication should report acid conversion, isolated or analytical yield, and selectivity using explicit equations. The mass balance should account for residual glycerol, free acid, glycerides, and relevant by-products. Calibration standards, response factors, sampling times, and any derivatisation procedures should be described. This is particularly important when comparing studies that report high acid conversion but provide limited information about monoester selectivity.

Catalysts should be characterised before and after reaction, with attention to composition, crystallinity, morphology, surface properties, and possible leaching. Because active species may form in situ, time-dependent or operando measurements would be valuable where feasible. Calcium content in the liquid phase should be quantified if leaching is possible. Reuse tests should include washing and regeneration procedures, mass recovery, activity after each cycle, and checks for product contamination.

Several gaps currently limit firm conclusions about the role of organic calcium salts in glycerol esterification.

Calcium glycerate, calcium oleate, calcium oxide, calcium hydroxide, and selected benchmark catalysts should be tested using identical protocols for acid substrate, reactant ratio, catalyst loading, temperature, mixing, and water removal. Reporting initial rates as well as final conversion would help distinguish activity from long-term equilibrium effects.

The catalyst should be characterised under reaction conditions wherever possible. Experiments should determine whether calcium glycerate, calcium carboxylates, or dissolved calcium species form during esterification. Controls using pre-synthesised candidate phases would help establish whether conversion is causally associated with their activity.

Kinetic or mass-transfer tests are needed to determine whether apparent rate differences arise from stirring speed, particle size, or controlled phase ratios. Time-resolved measurements should establish whether the induction period is sensitive to hydrodynamics or catalyst pretreatment.

Product quantification should distinguish MAG, DAG, and TAG and specify the exact definition of selectivity. Independent validation using a second analytical method would strengthen confidence in the product distribution, particularly where isomers or co-eluting compounds may be present.

Repeated-cycle experiments should measure recovered catalyst mass, retained activity, changes in phase composition, and calcium leaching. Regeneration conditions should be disclosed, and a catalyst should not be described as reusable on the basis of a single recovery test alone.

Further work should assess energy requirements, glycerol recovery, water-removal needs, catalyst preparation, downstream separation, and waste generation. The use of an inexpensive calcium precursor does not, by itself, make a process more environmentally friendly or economical.

III. Conclusions

Organic calcium salts are a promising but incompletely characterised family of catalysts for the esterification of glycerol with carboxylic acids. The strongest direct evidence in the literature considered concerns

calcium glycerate, which has been reported to promote oleic-acid esterification and to outperform several other calcium-containing materials under particular experimental conditions [9,10]. Reported acid-conversion values of approximately 58–86% after 10 hours should be treated as condition-specific results rather than a general guarantee of performance.

The chemistry proceeds through the sequential formation of MAG, DAG, and TAG, so acid conversion alone is insufficient to evaluate process success. Reactant ratio, temperature, reaction time, water removal, mixing, and phase behaviour can affect both conversion and selectivity. Reported induction behaviour in oleic-acid systems is consistent with the possibility that evolving interfacial properties and phase-transfer effects contribute to the observed kinetics, although this does not by itself establish a specific mechanism for calcium glycerate.

The central unresolved question is the identity of the active calcium species during reaction. Structural studies of calcium glycerate and evidence for calcium glycerate in related glycerol chemistry motivate further investigation, but the active phase must be confirmed under esterification conditions. Future studies should combine catalyst comparisons, transport-resolved kinetics, reliable product analysis, catalyst-reuse testing, and leaching measurements. Until such data are available, calcium glycerate is best described as a promising candidate that warrants systematic comparison with solid acids, zeolites, and lipases, rather than as a universally superior catalyst.

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