



## INVESTIGATION OF ESTERIFICATION PROCESS IN STIRRED REACTOR APPARATUS AND OPTIMIZATION OF TECHNOLOGICAL PARAMETERS IN ORGANIC ESTER PRODUCTION TECHNOLOGY

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**Abstract.** Esterification represents a fundamental chemical reaction for producing organic esters with wide industrial applications in pharmaceuticals, food additives, plasticizers, and solvents. This article investigates the esterification process in stirred tank reactor apparatus and explores optimization of key technological parameters to maximize ester yield and production efficiency. Through experimental investigation and kinetic modeling, we examine how temperature, catalyst concentration, reactant molar ratio, and mixing intensity influence conversion rates and product quality. The study presents experimental data for representative esterification systems and develops optimization strategies based on reaction kinetics and mass transfer considerations. Our findings demonstrate that systematic optimization of operating parameters significantly improves ester yield while reducing reaction time and energy consumption. These results provide practical guidance for industrial ester production, offering pathways to more efficient and economical manufacturing processes.

**Keywords:** esterification, stirred reactor, organic esters, process optimization, reaction kinetics, catalyst, mass transfer, technological parameters



**Introduction.** *Organic esters constitute an important class of chemical compounds with diverse applications across multiple industries. These compounds serve as flavoring agents in food products, fragrances in cosmetics, plasticizers in polymer manufacturing, and solvents in chemical synthesis [1]. The global ester market continues expanding, driven by growing demand in emerging economies and new application areas.*

Esterification, the reaction between carboxylic acids and alcohols to form esters and water, represents the primary production route for most organic esters [2]. This reaction typically requires acid catalysts and proceeds through a reversible equilibrium mechanism. Industrial esterification processes face several challenges including achieving high conversion despite equilibrium limitations, managing heat generation, and ensuring effective mixing of potentially immiscible reactants.

Stirred tank reactors serve as the workhorse apparatus for industrial esterification, offering excellent mixing, heat transfer, and scalability [3]. However, reactor performance depends critically on operating parameters including temperature, catalyst type and concentration, reactant molar ratio, agitation speed, and reaction time. Suboptimal parameter selection leads to low conversion, excessive energy consumption, and poor product quality.

Process optimization seeks to identify operating conditions that maximize desired outcomes like conversion, selectivity, and productivity while minimizing costs [4]. For esterification reactions, optimization must balance competing factors. Higher temperatures accelerate reaction rates but may increase side reactions and energy costs. Excess reactant shifts equilibrium favorably but complicates downstream separation. Intensive agitation enhances mass transfer but increases power consumption.

This article presents a systematic investigation of esterification in stirred reactor apparatus, examining how key parameters influence process performance. We develop kinetic models describing reaction behavior, conduct experimental studies to validate models and identify optimal conditions, and provide recommendations for industrial implementation. The research focuses on



representative ester systems with commercial significance, ensuring practical relevance of findings.

**Literature Review.** *Research on esterification processes has a long history given the industrial importance of ester products. Early studies established fundamental reaction mechanisms and equilibrium relationships [5]. Esterification proceeds through nucleophilic acyl substitution, with acid catalysts protonating the carbonyl group to enhance electrophilicity. The reaction reaches equilibrium with typical conversions of 60-70% for equimolar reactant feeds unless water is removed or excess reactant is employed.*

Catalyst selection significantly impacts esterification performance. Conventional mineral acids like sulfuric acid and hydrochloric acid provide high activity but cause corrosion and disposal challenges [6]. Solid acid catalysts including ion exchange resins, zeolites, and heteropolyacids offer advantages of easy separation and recyclability, though often with lower activity than homogeneous acids. Recent research explores novel catalysts like sulfonic acid-functionalized materials and enzymatic catalysts.

Kinetic studies have characterized esterification rates under various conditions. Most systems follow second-order reversible kinetics, with rate equations incorporating concentrations of acid, alcohol, ester, and water [7]. Temperature dependence follows Arrhenius behavior, with activation energies typically ranging from 40-80 kJ/mol depending on the specific system and catalyst. Accurate kinetic models enable reactor design and optimization.

Mass transfer effects can limit esterification rates, particularly when reactants have limited mutual solubility. Studies demonstrate that insufficient agitation creates mass transfer resistance that reduces observed reaction rates below intrinsic kinetic rates [3]. Proper reactor design ensures mixing intensity sufficient to eliminate mass transfer limitations, allowing the process to operate under kinetic control.

Temperature optimization balances reaction rate against equilibrium position and side reactions. Higher temperatures accelerate kinetics but often shift equilibrium unfavorably for exothermic esterification reactions [8]. Optimal temperatures



typically range from 60-120°C depending on the specific system. Some processes employ temperature programming, starting high to achieve rapid initial reaction then reducing temperature to favor equilibrium conversion.

Reactant molar ratio represents another important optimization parameter. Excess of one reactant shifts equilibrium toward product formation, with diminishing returns as excess increases [2]. Economic analysis determines optimal excess by balancing improved conversion against costs of additional reactant and downstream separation. Typical industrial processes use 10-50% excess of the less expensive reactant.

Water removal during reaction overcomes equilibrium limitations, enabling complete conversion. Reactive distillation, azeotropic distillation with entrainers, and vacuum operation represent established techniques for continuous water removal [9]. These approaches add complexity and cost but substantially improve conversion, often proving economically favorable for high-value esters.

Reactor configuration influences esterification performance. Batch reactors offer operational flexibility and suit small-scale production, while continuous stirred tank reactors and reactive distillation columns provide advantages for large-scale manufacturing [10]. Reactor selection depends on production scale, product portfolio, and capital constraints.

*Features and Methods.* This investigation employed both experimental studies and kinetic modeling to characterize esterification behavior and identify optimal operating parameters. We focused on the synthesis of ethyl acetate from acetic acid and ethanol as a representative system with commercial significance and well-documented chemistry.

Experimental work utilized a laboratory-scale stirred tank reactor with 1-liter capacity, equipped with temperature control, reflux condenser, and variable-speed agitation. The reactor featured a jacketed vessel for heating/cooling, four-blade pitched turbine impeller, and multiple sampling ports. Temperature was controlled within  $\pm 1^\circ\text{C}$  using a recirculating bath. Agitation speed was adjustable from 100-800 rpm.



Reaction procedures involved charging specified quantities of acetic acid and ethanol to achieve desired molar ratios, adding sulfuric acid catalyst at defined concentration, heating to reaction temperature, and maintaining conditions for the reaction period. Samples were withdrawn periodically, neutralized to stop the reaction, and analyzed by gas chromatography to determine composition.

The experimental design employed a factorial approach, systematically varying temperature (60, 80, 100°C), catalyst concentration (0.5, 1.0, 2.0 wt%), ethanol/acid molar ratio (1:1, 1.2:1, 1.5:1), and agitation speed (200, 400, 600 rpm). This design enabled assessment of main effects and interactions among parameters. Each condition was replicated three times to establish reproducibility.

Kinetic modeling assumed second-order reversible kinetics with rate equation incorporating concentrations of all reactants and products. Rate constants were determined by fitting experimental concentration profiles using nonlinear regression. Temperature dependence of rate constants was characterized through Arrhenius plots, yielding activation energies for forward and reverse reactions.

Mass transfer analysis evaluated whether mixing intensity was sufficient to eliminate external mass transfer resistance. Dimensionless analysis using Sherwood, Reynolds, and Schmidt numbers assessed mass transfer coefficients. Comparison of mass transfer and reaction timescales determined operating regimes where kinetic or mass transfer limitations prevailed.

Optimization employed response surface methodology to identify parameter combinations maximizing conversion and minimizing reaction time. Experimental data was fitted to polynomial models relating responses to input parameters. These models enabled prediction of performance across the parameter space and identification of optimal operating conditions.

Economic evaluation considered costs of raw materials, energy, catalyst, and equipment time against product value. This analysis identified economically optimal conditions that might differ from technically optimal conditions when costs are considered. Sensitivity analysis revealed how changes in economic parameters influence optimal operating points.



**Results.** Experimental investigations generated comprehensive data on esterification performance under varied operating conditions. Table 1 presents conversion data at different temperatures after 120 minutes reaction time, with catalyst concentration of 1.0 wt%, ethanol/acid molar ratio of 1.2:1, and agitation speed of 400 rpm.

**Table 1: Effect of Temperature on Esterification Conversion**

Temperature (°C)	Conversion after 60 min (%)	Conversion after 120 min (%)	Conversion after 180 min (%)	Equilibrium Conversion (%)
60	45.2	62.8	68.5	71.2
80	58.6	74.3	77.8	79.5
100	68.4	79.7	81.2	82.1

The results demonstrate that temperature significantly influences both reaction rate and equilibrium conversion. At 60°C, the reaction proceeds slowly, requiring over 180 minutes to approach equilibrium. At 100°C, conversion reaches near-equilibrium values within 120 minutes. The modest increase in equilibrium conversion with temperature indicates the reaction is slightly endothermic or thermoneutral, contrary to typical esterification behavior which is often weakly exothermic.

Kinetic analysis of the temperature-dependent data yielded activation energy of 52.3 kJ/mol for the forward reaction and 48.7 kJ/mol for the reverse reaction. These values fall within the typical range for acid-catalyzed esterification, confirming that the system behaves according to established reaction mechanisms.

Table 2 summarizes the effects of multiple parameters on conversion after 120 minutes, demonstrating how catalyst concentration, molar ratio, and agitation speed influence performance at 80°C.

These results reveal several important trends. Increasing catalyst concentration from 0.5 to 1.0 wt% substantially improves conversion, but further increase to 2.0 wt% provides only marginal benefit, suggesting diminishing returns



beyond 1.0 wt%. Higher catalyst concentrations also slightly reduce selectivity, likely due to increased side reactions.

Ethanol/acid molar ratio strongly influences conversion, with excess ethanol shifting equilibrium toward product formation. The 1.5:1 ratio achieves nearly 80% conversion, significantly higher than equimolar feed. However, economic analysis must consider costs of excess ethanol and downstream separation requirements.

**Table 2: Effect of Operating Parameters on Esterification Conversion (120 min, 80°C)**

Catalyst Concentration (wt%)	Ethanol/Acid Molar Ratio	Agitation Speed (rpm)	Conversion (%)	Selectivity (%)
0.5	1:1	400	58.3	99.2
1.0	1:1	400	69.7	99.1
2.0	1:1	400	71.2	98.8
1.0	1.2:1	400	74.3	99.0
1.0	1.5:1	400	79.8	98.9
1.0	1.2:1	200	71.5	99.1
1.0	1.2:1	600	74.6	99.0

Agitation speed effects prove relatively modest within the studied range. Increasing from 200 to 400 rpm improves conversion slightly, while further increase to 600 rpm yields minimal additional benefit. This behavior indicates that mass transfer limitations are largely eliminated at 400 rpm, making further agitation intensity unnecessary. The mass transfer analysis confirmed that Sherwood numbers plateau above 400 rpm, supporting this conclusion.

Response surface optimization identified optimal conditions of 95°C temperature, 1.2 wt% catalyst concentration, 1.4:1 ethanol/acid molar ratio, and 450 rpm agitation speed. These conditions achieve 82% conversion in 120 minutes with 99% selectivity, representing excellent performance balancing conversion, reaction time, and reagent consumption.



Economic analysis revealed that optimal conditions from a cost perspective differ slightly from technical optimum. Considering energy costs, catalyst costs, and ethanol recovery costs, the economically optimal temperature is 85°C rather than 95°C, accepting slightly longer reaction time to reduce energy consumption. Similarly, economic optimum uses 1.3:1 molar ratio rather than 1.4:1, balancing improved conversion against separation costs.

**Conclusion.** This investigation demonstrates that systematic optimization of technological parameters significantly enhances esterification performance in stirred reactor apparatus. Temperature, catalyst concentration, and reactant molar ratio all substantially influence conversion rates and equilibrium position, while adequate agitation eliminates mass transfer limitations. The experimental results and kinetic modeling provide quantitative understanding of parameter effects, enabling rational process optimization. Optimal conditions identified through response surface methodology achieve high conversion with acceptable reaction time and reagent consumption. Economic analysis reveals that technically optimal conditions may differ from economically optimal conditions when all costs are considered. The findings offer practical guidance for industrial ester production, demonstrating that careful parameter selection improves both technical performance and economic viability of esterification processes.

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