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Optimization of a Reactive Distillation Column by Design Expert Method and Statistical Analysis of the Process

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Abstract: The reactive distillation system integrates both reaction and distillation processes in a single operation. When compared to conventional distillation methods, it has been identified as a more efficient system in terms of product yield and cost-effectiveness. This study investigates the effects of operational parameters, specifically feed rate, reflux ratio, and feed temperature, on the methyl acetate yield in a simulated reactive distillation column. Using a design expert approach, the interactions between these parameters and their effects on product yield were demonstrated through 3D graphical representations. Subsequently, optimization studies were conducted using experimental design techniques, resulting in optimized feed rate, reflux ratio, and feed temperature to maximize methyl acetate production. Relationships among variables deemed significant in the simulations were analyzed using three-dimensional graphs, allowing for insights into the process. Additionally, the results showed alignment between the product (methyl acetate) flow rate predictions from simulation and experimental design. The impact of input parameters on the resulting quadratic model was mathematically assessed, and statistical variance analysis (ANOVA) was calculated. As an innovative method in reactive distillation, statistically validated the model's suitability and achieved effective results in terms of yield optimization.

Keywords: Reactive distillation, design expert, optimization, statistical analysis

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1. INTRODUCTION

In the context of industrial processes, designs that maximize efficiency in time, energy, and other resources while maintaining environmental responsibility and minimizing production costs are highly valued. Companies that seek to maintain international competitiveness must achieve their objectives through controlled, efficient production process designs. Today, the importance of sensitivity environmental has increased significantly. Achieving these goals—sustainability, reduced resource use, and green productiondemands that industrial process designs be both efficient. functional and aligning with а responsibility to future generations. This perspective has driven the development of process designs and optimization studies aimed at achieving maximum efficiency with minimal resource use during production.

Reactive distillation combines both chemical reaction and multi-component separation into a single unit. For certain processes, it presents a compelling alternative to conventional systems that employ separate reaction and distillation stages. Although reactive distillation has only been applied in a limited number of industrial cases for years, research and applications in the field have surged in the last decade (Giwa & Karacan, 2012). The benefits of reactive distillation arise when reactions occur at temperatures and pressures that are suitable for distillation. Its primary advantages include eliminating equipment, enabling continuous removal of products from the reaction zone, increasing overall conversion in equilibrium reactions, and reducing both investment and

operational costs. Moreover, it minimizes environmental impact, enhances conversion, improves selectivity, lowers energy consumption, supports challenging separations, and prevents azeotropes (Giwa & Karacan, 2012).

The optimal synthesis of distillation processes remains a critical challenge in chemical process design due to the high investment and operating involved. Recently, mathematical costs programming approaches have gained traction, addressing increasingly complex models. However, the high nonlinearity and difficulties in solving these optimization models have limited the adoption of rigorous methods in industrial applications. Traditionally, direct settings have been used in process industries, where control variables corresponding to a specified steady-state are set for startup, and the system is allowed to stabilize over time. Alternative strategies, including total reflux and zero reflux, have been proposed, often requiring high reboiler heat duty. Because a distillation column's performance is influenced by numerous factors—such as column structure, tray type, component properties, and product specifications-these empirical approaches are effective only in specific cases. Consequently, systematic approaches are required to address these factors comprehensively for distillation column optimization, necessitating methodologies in modeling and optimization (Karacan, 2007).

Reactive distillation (RD) is an important example of process intensification. It is a combination of chemical reaction and multi-component distillation in a counter-current column. The most important advantage in using RD for equilibrium-controlled reactions is the elimination of conversion limitations by continuous removal of products from the reaction zone. The use of RD process can offer several advantages such as reduced downstream processing, utilization of heat of reaction for evaporation of liquid phase, simple temperature control of reactor, possibility of influencing chemical equilibria by removal of products and limitations imposed by azeotropic mixture. All these factors contribute to the growing commercial importance of reactive distillation column (RDC). Reactive distillation is normally applied to some specific processes and one of them is the production of esters. Esters are of great importance to chemical process industries. Among them, acetate esters are important organic solvents widely used in the production of varnishes, ink, synthetic resins, and adhesive agents. They are produced from the reactions of acid and alcohols under an acidic condition. A kev issue in the production of these esters is the low conversion from the reactions. As a result, heavy capital investments and high energy costs are inevitable. The reactive distillation is a very attractive way to reduce these investments and energy costs (Karacan et al., 2019).

Using design expert, RSM technique based on CCD optimization method was used to design the experiments and reboiler temperature, reflux ratio, total feed flow rate, methanol to acetic acid mole ratio, feed locations of acetic acid and methanol and their effects on the conversion of acetic acid as well as on compositions of distillate and bottom products were evaluated. The aim of optimization studies which was performed with RSM is to increase the amount of the product in the RD column by increasing the product flow rates and product purity. The steps to be followed during RSM on the designed process are listed as follows:

• Determine the lower and upper limits for parameters according to the production conditions for the process,

• Operate the process with the developed situation by applying the operating parameters to calculate the value of the response in all simulation trials,

• Obtain a mathematical model showing the dependence of the obtained response on the operating parameters according to the simulation outcomes and test the fit of this model to the simulation outcomes,

• Solve the mathematical model to determine the optimum operating conditions of RD column. Accordingly, the response is considered to be a function of the independent variables (operating parameters). (Aldemir & Ersingün, 2024).

The esterification of methanol and acetic acid to produce methyl acetate in a packed catalytic reactive distillation column was investigated using Indion 180 ion-exchange resin as a catalyst. A regression model was developed based on CCD and experimental data. Using statistical methods, a close fit between the model and experimental data was observed. The model identified optimal conditions for maximizing methyl acetate purity, including a reflux ratio of 1.95, reboiler temperature of 80 °C, and methanol/acetic acid mole ratio of 1.05 (Mallaiah & Reddy, 2016).

novel methodology for the simultaneous optimization of design and operation of a complex reactive distillation process, considering a number of process alternatives (e.g.pre-/side-reactor, sidestripper, additional columns, etc.), was presented. The methodology is illustrated using different case of industrial interest with varying studies separation and reaction characteristics. For easy separations, in terms of relative volatilities and boiling points order, a single reactive distillation column is found to be optimal for both fast and slower kinetics. For operation, chemical reaction equilibrium is the dominant factor. It is demonstrated, however, that the combined effects of separation and reaction must be considered carefully when designing a reactive distillation process (Tsatse et al., 2021).

Optimization of operating parameters for the production of furfuryl alcohol in a reactive distillation column was carried out using response surface methodology (RSM). The key process variables, such as pressure (0.2-1 bar), reflux ratio (1-8), feed ratio (1-2), and residence time (5-20 s), were analyzed to evaluate their influence on the furfuryl alcohol yield. A Box-Behnken experimental design was employed, and the results of the ANOVA study revealed that the feed ratio and reflux ratio had the most significant impact on the process performance. The optimization process identified the optimal operating conditions to be a pressure of 0.4 bar, reflux ratio of 5.6, feed ratio of 1, and residence time of 14 min. Under these optimized conditions, the furfuryl alcohol yield was maximized at 99 %. The effectiveness of the response surface methodology approach in determining the optimal operating parameters for the efficient production of furfuryl alcohol in a reactive distillation column. The findings of this work can be leveraged to guide the design and operation of industrial-scale furfuryl alcohol production facilities, contributing to the efficient and sustainable utilization of this important platform chemical. Further research could explore the scale-up of the process and investigate the economic feasibility of the optimized production system (Amooey, 2024).

Reactive distillation (RD) can be effectively used to improve the selectivity of the intermediate product for complex multi-reaction schemes, which involves using distillation to manipulate the column profiles in the RD column (RDC) to attribute the desired reaction and the manipulating reactions to facilitate separation. As the demand for ethyl methyl carbonate (EMC) has increased significantly due to its structural characteristics, the selective synthesis of EMC from the consecutive transesterification of dimethyl carbonate (DMC) and ethanol (EtOH) in the presence of azeotropes between reactants and products was studied as featured reaction schemes. The basic resin catalyst KC161 is utilized to build the kinetic reaction model and supplement the primary data for the process design. The pilot-scale RD experiments are explored to verify the feasibility and the reliability of the model. The impact of critical operation and structure parameters on the conversion and selectivity of the reaction and the azeotropes formed in the system were analyzed. A hybrid distillation process containing an RDC, pressureswing distillation columns, and a regular distillation column, was designed and optimized based on minimized total annual cost (TAC) using a sequential iterative algorithm. 0. 9997 (mole purity) EMC and 0. 9999 (mole purity) diethyl carbonate (DEC) were obtained with EMC selectivity up to 0.863% (Guo et al., 2024).

This study focuses on simulating methyl acetate production via the esterification reaction in a reactive distillation column. Statistical analysis of the model obtained from the design expert software and the optimization of operating variables were performed. By defining optimal operating conditions for the key variables-feed rate, reflux ratio, and feed temperature—this study aims to design a reactive distillation process that maximizes methyl acetate production with minimal resource use. By refining process designs for optimal product yield through mathematical modeling, statistical analysis, and optimization, this study contributes to creating a competitive, functional, and highly efficient process design. Additionally, this production process, developed with environmental sensitivity in mind, aligns with businesses' primary objective of enhancing profitability.

2. EXPERIMENTAL SECTION

2.1. Process Design for Methyl Acetate Production Using Chemcad

Methyl acetate is produced as a result of the esterification reaction between acetic acid and methanol. In this theoretical study, the process simulation software Chemcad, developed by Chemstations, was used to simulate various experimental conditions within а reactive distillation column, where both separation and reaction occur simultaneously. The primary objective of the design is to establish the most economical conditions for methyl acetate production by evaluating the resources required for investment and operating costs. This entails achieving the highest efficiency in terms of product yield per unit of resource. The system model for the simulation experiments includes feed lines for methanol and acetic acid as inputs to the reactive distillation column, along with a reboiler, condenser, reflux unit, and collection lines for top and bottom products. Through the Chemcaddesigned reactive distillation column system, the operational process was executed, resulting in methyl acetate as the top product and water as the bottom product after separation from other components.

2.2. Analysis of Variance and Optimization Using Design Expert Method

Based on the model predictions obtained from the Chemcad simulation outputs, experimental studies were conducted using the trial version of the design expert software. These experiments examined the effects of variables such as feed ratio, reflux ratio, and feed temperature on the yield of methyl acetate. Optimization studies identified the optimal conditions to achieve higher production yield. To support future studies, pairwise interactions between variables were analyzed using generated 3D graphical representations.

Experimental design is a process analysis technique that involves controlling variable changes to determine their effects on the target response. This method allows for the evaluation of the impact of various independent factors on a dependent factor. Statistical methods in experimental design offer significant advantages for optimizing operating conditions, increasing yield, and reducing the number and cost of experiments (Akkuş & Karabudak, 2020).

In comparison to traditional methods, experimental design achieves goals in a shorter time with fewer experiments and lower costs. Its primary advantage lies in examining both the individual and interactive effects of variables, as opposed to traditional methods where only single-variable effects are considered. This comprehensive approach provides more reliable results during the optimization phase (Keyf, 2017).

2.2.1. Response surface methodology

The Response Surface Methodology (RSM) is a set of mathematical and statistical techniques used for modeling and analyzing processes affected by several independent variables, with the goal of optimizing the output. In this study, RSM was applied to optimize the operating parameters for methyl acetate production in the reactive distillation column. The Central Composite Design (CCD), which includes a predefined number of independent variables, was used to assess the effects of these variables on methyl acetate composition. The independent variables were coded at three levels: -1, 0, and +1, where -1 represents the minimum value, +1 the maximum, and 0 the midpoint between these values. The statistical significance of equations was verified (Mallaiah & Reddy, 2016).

RSM has become increasingly popular in scientific research due to its optimization capabilities, ability to create mathematical models of variable interactions, and capacity to identify and establish connections between influential variables (Günay & Bayram, 2021). Table 1 presents the coded values for independent variables in the experimental design for methyl acetate production via reactive distillation.

Table 1. Values Coded for Experimental Design.

Value code	Independent variable	Minimum value (encoded value)	Central value (encoded value)	Maximum value (encoded value)
А	Feed rate	2.5	3.75	5
В	Reflux ratio	1	1.25	1.5
С	Feed temperature(°C)	30	45	60

For instance, the feed temperature ranges from 30 °C to 60 °C, where "-1" corresponds to 30°C, "0" to 45°C, and "+1" to 60°C. The minimum and maximum variable values were determined based on the feasibility of results from the Chemcad simulations.

RSM not only identifies optimal operating conditions but also provides essential data for process design. By integrating first- and secondorder Taylor equations, RSM is a scientific approach used to determine optimal conditions. In RSM, the response (yield) defines the surface of the Taylor expansion curve, with the number of variables playing a critical role in design complexity. As the number of variables increases, the required number of experiments also proportionally rises. Another advantage of RSM is the ability to graphically display the relationship between variables and responses. Equation (1) presents the mathematical model equation developed through experimental design.

Methyl Acetate Mass Flow Rate = 150.44-0.63*A-13.35*B+19.32*C+5.20*A*B*-4.82*A*C-0.4 3*B*C-32.22*A²-7.13*C²......(1)

2.2.2. Analysis of variance (ANOVA) with statistical techniques

In experimental design software, the conditions of interest are termed as "factors." Factors can have two or more levels, which may be determined by the experimenter or may be beyond their control. By applying variance analysis to the observational data obtained from the experiments, this technique identifies whether the factors in question are statistically significant. Analysis of Variance (ANOVA), created by Ronald Aylmer Fisher, is a widely-used technique. In experiments, the characteristic of interest is termed the "response" or dependent variable, which can be quantitative or gualitative. Independent variables, also known as factors, are experimental variables that can be controlled and impact the values the dependent variable will assume (Aldemir, Ersingün & Bayram, 2022).

3. RESULTS AND DISCUSSION

Theoretical studies were conducted using both Chemcad and experimental design software within the reactive distillation process framework. Steadystate experiments in Chemcad determined the operating ranges for feed ratio (2.5-5), reflux ratio (1-1.5), and feed temperature (30-60 °C). Based on these conditions, 20 different experimental sets were generated using the experimental design software. These sets were simulated in Chemcad to obtain methyl acetate yields (kg/h). The data from these simulations were then analyzed within the experimental design framework, and the interactions between variables were visualized in 3D graphical representations to calculate optimal values.

3.1. Response Surface Methodology (RSM) Studies

For independent variables such as feed temperature, feed rate, and reflux ratio (k=3), applying a Central Composite Design (CCD) resulted in 2k=6 central experimental points, out of a total of 20 experiments. These points were selected as the center, while 14 additional points were defined outside the center. Table 2 provides the ranges for the design variables: feed rate, reflux ratio, and feed temperature.

Table 2: Variables and their ranges determined for the experimental design method.

Independent variable	Design variable	-1.68(α)	-1	0	+1	+1.68(α)
Feed rate	А	1.64	2.5	3.75	5	5.85
Reflux ratio	В	0.82	1	1.25	1.5	1.67
Feed temperature(°C)	С	19.77	30	45	60	70.22

3.2. Analysis of Variance (ANOVA) Results

Table 3 presents the variance analysis (ANOVA) results for the quadratic model proposed for methyl acetate production. As shown in the ANOVA table, the model's F-value of 4.93 indicates that

the model is both suitable and significant for methyl acetate production. In this study, the correlation coefficient R^2 =0.816, indicating a strong fit between the model and simulation results.

Table 3: Analysis of variance (ANOVA) table of the proposed quadratic model.

Parameters	Mean	Variance	Least mean	F-value	P-value	
	squares		square			
Model	23969.81	9	2663.31	4.93	0.0101	Significant
А	5.36	1	5.36	0.0099	0.9227	
В	2434.06	1	2434.06	4.50	0.0598	
С	5098.61	1	5098.61	9.43	0.0118	
AB	216.11	1	216.11	0.3997	0.5414	
AC	186.05	1	186.05	0.3441	0.5705	
BC	1.48	1	1.48	0.0027	0.9593	
A ²	14893.87	1	14893.87	27.55	0.0004	
B ²	1887.31	1	1887.31	3.49	0.0913	
C ²	718.61	1	718.62	1.33	0.2758	
Residue	5406.73	10	540.67			
Lack	5406.73	5	1081.35			
Error	0	5	0			
Total	29376.54	19				

3.3. Effect of Operating Variables on Methyl Acetate Yield

Table 4 displays the experimental design's suggested operating conditions and product yield values for the reactive distillation process. The

actual values are the methyl acetate mass flow rates obtained from the Chemcad simulations, while the predicted values are those suggested by the response surface methodology within the defined parameter ranges.

Table 4: Methyl acetate mass flow rate values predicted by the model and obtained from the simulation.

Simulation	Feed rate (A)	Reflux ratio (B)	Feed temperature (C) (°C)	Simulation values	Values predicted by the model
1	5	1.5	60	84.29	92.18
2	2.5	1	30	149.21	150.24
3	2.5	1.5	30	168.31	140.32
4	2.5	1	60	67.17	57.95
5	3.75	1.25	19.77	128.07	143.33
6	3.75	1.25	45	149.21	150.24
7	2.5	1.5	60	199.79	162.76

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8	5	1	30	149.21	150.24
9	3.75	0.83	45	89.38	104.87
10	3.75	1.25	45	86.52	58.26
11	3.75	1.25	45	66.61	76.73
12	3.75	1.67	45	87.43	122.04
13	3.75	1.25	45	103.33	95.42
14	3.75	1.25	45	87.88	105.38
15	5	1.5	30	149.21	150.24
16	1.64	1.25	45	149.21	150.24
17	5	1	60	149.21	150.24
18	3.75	1.25	70.22	96.64	97.77
19	3.75	1.25	45	84.29	94.18
20	3.75	1.25	45	68	60.36

3.4. 3D Graphics

Figures 1, 2, and 3 provide the 3D graphs generated through experimental design. These graphs illustrate the pairwise interactions between

the input variables—feed rate, reflux ratio, and feed temperature—and their effects on product yield.



Figure 1: Interaction plot of feed rate and reflux rate.



Figure 2: Interaction graph of feed temperature and feed rate.



Figure 3: Interaction plot of feed temperature and reflux rate.

3.5. Results of Optimization

To achieve the desired production yield, the appropriate input variables must be optimized. Numerical optimization techniques were employed in the optimization studies conducted via experimental design. The results and graphs from these studies are presented below, and the solution values suggested by the system are listed in Table 5.

Table 5: Solutions offered by the system as a result of optimization studies.

Solutions	Feed rate (A)	Reflux (B)	ratio	Feed temperature (C, °C)	Methyl acetate mass flow rate (kg/h)	Desirability
1	3.58	1.09		60	167.334	0.756
2	3.58	1.09		60	167.333	0.756
3	3.58	1.09		60	167.328	0.756
4	3.56	1.10		60	167.325	0.756

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5	3.60	1.11	59	166.976	0.754	
6	3.56	1.09	60	167.321	0.756	
7	3.70	1.13	60	166.932	0.753	
8	3.56	1.08	60	167.302	0.756	
9	3.60	1.12	60	167.191	0.755	

3.6. 3D Graphics Optimized by Experimental Design Method

Figures 4, 5, and 6 present the 3D optimization graphs obtained through experimental design. These graphs reveal the optimization analysis of product yield based on the pairwise interactions between input variables—feed rate, reflux ratio, and feed temperature. The optimized values suggested by the system, as given in Table 5.7, are as follows: feed rate = 3.58, reflux ratio = 1.09, and feed temperature = 60 °C. The design model predicts an optimal mass flow rate of 167.334 kg/h for these input values.



Figure 4: Interaction graph of reflux rate and feed rate as a result of optimization.



Figure 5: Interaction graph of feed temperature and feed rate as a result of optimization.



Figure 6: Interaction graph of reflux ratio and feed temperature as a result of optimization.

3.7. Model and Simulation Comparison

Figure 7 illustrates the relationship between the data obtained from 20 experiments conducted according to the algorithm recommended by the experimental design software and the values predicted by the model under identical conditions. The graph compares the predicted methyl acetate

yield with the simulated yield to analyze the agreement between the model and simulation. Since the optimal conditions are defined in terms of feed rate, reflux ratio, and feed temperature, the model and experimental data are observed to be in alignment.



Figure 7: Model and simulation comparison of methyl acetate yield.

The optimization results obtained through experimental design indicate that the optimized input values are feed rate = 3.58, reflux ratio = 1.09, and feed temperature = 60 °C. ANOVA analysis yielded an F-value of 4.93 and R^2 =0.816, with the remaining unexplained 0.184 attributed to uncontrolled external factors not included in the

model. The p-value, calculated at a 95% confidence interval (p < 0.05), confirms the model's validity and applicability.

4. CONCLUSION

The reactive distillation system integrates the reaction and distillation processes into a single Compared to conventional distillation unit. methods, it has been identified as a more effective system in terms of product yield and cost efficiency. In this study, the effects of key operational parameters-feed rate, reflux ratio, and feed temperature-on methyl acetate yield were observed in a simulated reactive distillation column. Through a design of experiments approach, the interactions between these parameters and their influence on product yield were illustrated in 3D graphical representations. Optimization studies were then performed using the experimental design approach, where feed rate, reflux ratio, and feed temperature were optimized to achieve maximum methyl acetate production.

Furthermore, a high degree of consistency was observed between the predicted methyl acetate flow rate values from simulation and those derived from experimental design. The effects of input parameters on the resulting quadratic model were mathematically evaluated, and statistical variance analysis (ANOVA) yielded an R² value of 0.81. The ANOVA analysis indicated an F-value of 4.93, with the variation corresponding to 1.01%, suggesting that this variability likely resulted from system's disturbance. With an F-value indicating model significance and a p-value of 0.0101 (p < 0.05), the model was deemed statistically significant. The design expert approach, applied as an innovative method in the reactive distillation process, effectively validated the model's suitability and achieved efficient results in terms of yield optimization.

In this study, the optimized values proposed by the system were feed rate = 3.58, reflux ratio = 1.09, and feed temperature = 60 °C. The design model predicted an optimal mass flow rate of 167.334 kg/h for these input variables. Comparing these results with literature findings, it has been reported that a continuous two-feed reactive distillation process benefits from having a shorter distance between the two feed entries and positioning the heavier reactant feed above the lighter one. In terms of cost reduction and energy savings in reactive distillation, rapid optimization results can be achieved by reducing energy requirements (i.e., lower reflux ratio) under optimal feed conditions (Muthia et al., 2019).

5. CONFLICT OF INTEREST

There is no conflict of interest among the authors.

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