Article

Optimizing the Sodium Hydroxide Conversion Using Regression Analysis in CSTR

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Abstract: The current study deals with the maximization of NaOH conversion using step-wise regression analysis in a CSTR. The dependence of temperature, volume, agitation rate, and feed rate on reactor performance is examined as well as interaction outcome of the operating parameters. The concentration of the reactants was fixed at 0.1 M. The steady state conversion with respect to NaOH is analyzed to find the process performance. Step-wise regression analysis is used to remove an insignificant factors. The agitation rate (X2) and feed rate (X3) proved to have an insignificant influence on the reaction conversion at a significant level (α) of 5%. Consequently, the temperature (X1) and reaction volume (X4) were found to have significant effect on the reaction conversion using step-wise regression. The temperature and volume dependence on steady state NaOH conversion were described by a polynomial model of 2nd and 3rd order. A maximal steady state conversion equal to 63.15% was obtained. No improvement was found in reaction conversion with 3rd order polynomial, so the second order polynomial is considered as the optimum reaction conversion modal. It may be recommended that 2nd order regression polynomial model adequately represents the experimental data very well.

Keywords: regression analysis; polynomial; optimization; CSTR; model

1. Introduction

Stepwise regression is a prevailing technique to forecast unidentified predicted variables from predictor variables. The independent variables are also known as predictors. The predicted variable is known as a dependent variable. The regression analysis is applied to study the impact of predictor variables on response (output). The main advantage of stepwise regression is the ability to manage large amounts of potential predictor variables and to select the best predictor variables from available options. The stepwise regression model is used to determine the most dominating factors among the variables. The central composite design (CCD) is useful in surface response methodology for developing a quadratic model to predict the response variable, whereas the Box-Behnken design (BBD) applies to obtain high order response surfaces using little needed runs than normal factorial design. The statistical designs are comprehensively applied to advance process identification and optimization. Good quality preferred products are produced frugally applying well acknowledged statistical design of experiment in chemical and other allied industries [1,2].

The computational attempts are essential to study the model behavior, and the cost can be optimized by evolving an effectual procedure. A factorial design was applied for process enhancement to minimalize process time [3]. A batch reactor performance was studied using statistical tools for methodical procedure [4]. It has been stated that developments in the design of experiments have made it conceivable to use the technique for wider applications [5], process enhancement of hydrolysis of ethyl acetate [6], and batch reactor process development [7]. The selection of experimental design decisively relies on
the objective of experimentation [2,8]. The response surface methodology (RSM) and screen designs are the most broadly used designs for optimizing the processes. The aim of selection design is to remove the irrelevant variables and then relating RSM to determine the optimal values of the substantial variables. The surface response methods (CCD and BBD) were applied to compare the efficiency and analyze the prime interacting parameters of anaerobic sludge reactor [9]. It was suggested that RSM can be applied for optimizing the wastewater treatment process.

The polynomial regression model has been optimized by multiple regressions. Sodium acetate produced in the current work is profitable and valuable carboxylic salt. The literature [10–16] have specifically focused on reaction mechanics for the hydrolysis of CH₃COOC₂H₅. An experimental study on saponification of ethyl acetate applying different reactive systems was performed, and the influence of reactor-type on reactor performance was examined [17]. It was suggested that a high-pressure drop was exhibited by microreactors. It was perceived that hydrolysis of CH₃COOC₂H₅ continues by direct attack of the ion of CH₃COOC₂H₅ on the carbon atom [18]. The fast hydrolysis process exploits dichloromethane/methanol in 1:9 ratio as a solvent with small concentration of sodium hydroxide [19]. A simulation study for an ethyl acetate hydrolysis was examined in detail [20]. A factorial design was applied to study the optimization of ethyl acetate [21,22].

The process intensification of applying a design approach with the aims of reducing the formation of unwanted products and increasing conversion was described [23]. A parametric study for ethyl acetate saponification was performed using a batch reactor and the impact of temperature, volume, rate of agitation, and initial reagents concentration was examined [24]. It was suggested that increased initial concentration resulted in reduced sodium hydroxide (NaOH) conversion. The batch reactor performance was studied using regression analysis for saponification of ethyl acetate [25] and maximal conversion of 0.995 realized under optimum conditions reactant concentration and agitation rate. An ethyl acetate saponification reaction in a tubular reactor was studied using factorial design [26], and the reaction order was determined as nearly equal to 2.

In the current work, the multiple regression was applied for examining the CH₃COOC₂H₅ hydrolysis using continuous stirred tank reactor (CSTR). The investigation was unambiguously dedicated to the process of upgrading reactants conversion to ethanol and sodium acetate. The novelty of the current work is that it optimizes the formation of sodium acetate and ethanol, applying step-wise regression applying polynomial models. The methodology comprises of two steps: experiments designed to remove the insignificant factors and RSM. The overall performance of CSTR was analyzed in terms of conversion with respect to sodium hydroxide (Xₙ). All the potential factors were supposed to have an influence on system performance. The authors chose to study the key and interaction influence of temperature X₁ (°C), stirrer speed X₃ (rpm), feed rate X₅ (mL/min), and reactor volume X₆ (L). The experimental outcomes [27] were chosen as a base to perform and carry out the optimization analysis. After the trial for screening of the first phase, feed rate and stirrer speed were established as important predicting variables and optimal outputs of these factors were calculated through a 2nd order polynomial.

2. Material and Method
2.1. Reaction Kinetics

The ethyl acetate hydrolysis is exemplified as Equation (1) [6]:

\[
\text{NaOH} + \text{CH}_3\text{COOC}_2\text{H}_5 \rightarrow \text{CH}_3\text{COONa} + \text{C}_2\text{H}_5\text{OH}
\]  

The rate law is given as Equation (2) [6]:

\[
-v_{\text{NaOH}} = k_{\text{NaOH}} C_{\text{EtAc}}
\]

The acetate ions are generated and hydroxyl ions are used as reaction progresses. A drop in conductivity observed by conductivity sensor is seen as the reaction continues,
owing to the datum that ions (acetate) are less conductive than hydroxide ions. The variation in conductivity values is essential to observe the advancement of hydrolysis. A correlation between the ionic conductivity of the mix and hydroxyl ion concentration is acquired and depicted through Equation (3) [6]. Studies [10–12,18] have explicitly concentrated reaction mechanics and kinetics of hydrolysis of ethyl acetate.

\[
\frac{C - c_{\infty}}{C_0 - c_{\infty}} = \frac{C_{NaOH} - C_{NaOH_{\infty}}}{C_{NaOH_{0}} - C_{NaOH_{\infty}}}
\]

B.C.: \( C_{NaOH_{\infty}} \rightarrow 0, \) as \( t \rightarrow \infty \)

The above equation is written as:

\[
\frac{C_{NaOH}}{C_{NaOH_{0}}} = 1 - X_{NaOH} = \frac{C - c_{\infty}}{C_0 - c_{\infty}}
\] 

2.2. Experiment

The data acquired [27] for hydrolysis of ethyl acetate was used for present optimization analysis by multiple regression. The NaOH and ethylacetate (\( CH_3COOC_2H_5 \)) of analytical grade were applied to perform the experiments. The standard solutions of required molarity were made by double distilled water generated in the laboratory. Only NaOH and \( CH_3COONa \) added conductance to the reaction mixture. The conductivity measurement method for hydrolysis of \( CH_3COOC_2H_5 \) was explained elaborately [28].

2.3. Experimental Strategy

An analysis was performed in two steps i.e., first screening and after that optimization. The stirrer rate, volume, temperature, and reagents molarity are the factors considered for multiple design. The insignificant factors were removed from analysis in the first phase. The values of predicting variables carefully chosen for experimental design are presented (Table 1). The momentous predicting variables were selected to augment the reaction conditions to predict the highest steady-state conversion of NaOH in the second stage. The 2nd order polynomial model was chosen for regression analysis using optimum factors. The operating limits of independent variables chosen are depicted (Table 1).

### Table 1. Operation variables.

|                        | N | Range | Minimum | Maximum | Mean |
|------------------------|---|-------|---------|---------|------|
| Temperature, \( X_1 \) (°C) | 16 | 15    | 25      | 40      | 30.63|
| Agitation rate, \( X_2 \) (rpm)   | 16 | 120   | 70      | 190     | 130.00|
| Feed flow rate, \( X_3 \) (mL/min) | 16 | 30    | 50      | 80      | 61.25|
| Reactor volume, \( X_4 \) (L)    | 16 | 0.75  | 1.00    | 1.75    | 1.4688|

3. Results and Discussion

3.1. Experimental Findings

The outcomes of the experimental study [25] were considered for the design of experiments and these outcomes are listed as:

a. The steady state conversion with respect to sodium hydroxide raised with increased temperature.

b. The higher reactor volume leads to reduced \( X_a \).

c. An increased stirrer rate from 70 to 150 rpm results in declined steady state NaOH conversion.

d. The increased reactants flow is attributed to a decrease in reaction conversion.

An analysis was applied to determine the correlation amongst the predictors (\( X_1, X_2, X_3 \) and \( X_4 \)), and steady state conversion (\( X_4 \)) is presented (Table 2). The moderate positive correlation \((r = 0.446)\) is revealed between conversion and temperature. The correlation coefficient between stirrer speed and steady-state conversion is 0.099, specifying that 1% variations in stirrer rate lead to 9.9% variation in
NaOH conversion. Low negative correlation ($r = -0.076$) was observed between feed rate and conversion, which states that conversion increases with reduced feed flows. Moderate positive correlation ($r = 0.599$) was observed between volume and conversion, which states that 100% increase in volume results in 59.9% surge in reaction conversion. It is realized that correlation is substantial at 0.05 significance level.

An attempt was made to develop a regression equation model to optimize reaction conversion. The $X_A$ was assumed as a process response variable (dependent variable) and $X_1$, $X_2$, $X_3$, and $X_4$ are the predictors. A SPSS tool was used to enter all the dependent and independent variable entered into model. Equation (5) was established to augment the steady state conversion of NaOH.

$$X_A = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_4 X_4 + e \quad (5)$$

$X_A$: predicted variable; $X_1$, $X_2$, $X_3$, and $X_4$ are the predictors. All beta terms are the coefficient of predictors and $\beta_0$ is an intercept of the developed model, which is a constant parameter. The symbol e signifies an error term of the developed model.

| Table 2. Correlation analysis with four predictors. |
|-----------------------------------------------|
| $X_A$       | $X_1$ | $X_2$ | $X_3$ | $X_4$ |
|-------------|-------|-------|-------|-------|
| Pearson Correlation | 1.000 | 0.446 | 0.099 | -0.076 | 0.599* |
| Sig. (2-tailed) | 0.083 | 0.715 | 0.780 | 0.014 |
| N            | 16    | 16    | 16    | 16    |
| Pearson Correlation | 0.446 | 1.000 | -0.043 | 0.043 |
| Sig. (2-tailed) | 0.083 | 0.873 | 0.873 | 0.873 |
| N            | 16    | 16    | 16    | 16    |
| Pearson Correlation | 0.999 | 0.000 | 0.000 | 0.000 |
| Sig. (2-tailed) | 0.715 | 1.000 | 1.000 | 1.000 |
| N            | 16    | 16    | 16    | 16    |
| Pearson Correlation | -0.076 | -0.043 | 0.000 | 1.000 |
| Sig. (2-tailed) | 0.780 | 0.043 | 0.043 | 0.043 |
| N            | 16    | 16    | 16    | 16    |
| Pearson Correlation | 0.599* | 0.043 | 0.043 | 0.043 |
| Sig. (2-tailed) | 0.014 | 1.000 | 1.000 | 1.000 |
| N            | 16    | 16    | 16    | 16    |

*Significant correlation at 0.05 level. $X_A$ = Conversion, $X_1$ = Temperature, $X_2$ = Agitation Rate, $X_3$ = Feed Rate, $X_4$ = Volume.

As illustrated in Table 3 Model Summary, moderate positive correlation was observed between the dependent and independent variable. R square demonstrates that the goodness of fit is moderate, whereas adjusted $R^2$ explains an extraordinary influence of the extraneous variable. F value, as depicted in model summary Table 3, explains that the influence of independent variables varies considerably at 0.05 significance level. The p-value less than 0.05 demonstrates that influence of independent variables varies significantly with 95% confidence interval as explained in Table 4. The tabulated F value is lower equated with the calculated one and recommends that independent variables variances of contribution are statistically substantial. It can also be concluded from the calculated sig. value presented in Table 5 (0.048) that the researcher is not fully confident about the variation of contribution of all predictors.
Table 3. Summary of developed model with four predictors.

| Model | R | R square | Adjusted R square | Std. error of the estimate | R square change | F change | df1 | df2 | Sig. F change | Durbin-Watson |
|-------|---|----------|------------------|--------------------------|----------------|----------|-----|-----|---------------|---------------|
| 1     | 0.743 | 0.553 | 0.390 | 0.0608951 | 0.553 | 3.397 | 4   | 11  | 0.048 | 0.304 |

1. Independent variables: X1, X2, X3, X4
2. Predicted variable: XA

Table 4. ANOVA with four predictors.

| Model | Sum of squares | df | Mean square | F   | Sig. |
|-------|----------------|----|-------------|-----|------|
| 1     | Regression     | 0.050 | 4 | 0.013 | 3.397 | 0.048 |
|       | Residual       | 0.041 | 11 | 0.004 | | |
|       | Total          | 0.091 | 15 | | | |

1. Predictors: XA
2. Independent variables: X1, X2, X3, X4

Table 5. Determination of coefficients with four predictors.

| Model | Unstandardized coefficients | Standardized coefficients | t | Sig. | 95.0% Confidence interval for B |
|-------|----------------------------|---------------------------|----|------|-------------------------------|
|       | B | Std. Error | | Beta | | Lower Bound | Upper Bound |
| 1     | (Constant) | -0.240 | 0.277 | -0.867 | 0.405 | -0.851 | 0.370 |
|       | X1 | 0.011 | 0.005 | 0.417 | 2.065 | 0.043 | 0.001 | 0.022 |
|       | X4 | 0.295 | 0.102 | 0.585 | 2.894 | 0.015 | 0.070 | 0.519 |
|       | X3 | -0.001 | 0.003 | -0.083 | -0.411 | 0.689 | -0.007 | 0.005 |
|       | X2 | 0.000 | 0.001 | 0.099 | 0.492 | 0.632 | -0.001 | 0.002 |

Table 5 illustrates the impact of all independent variables on steady-state conversion of NaOH. The calculated p value is statistically significant for X1 and X4, while insignificant for X2 and X3. Furthermore, the p value demonstrates that X3 is highly insignificant but X2 is low insignificantly as compared to X3. Equation (6) was formulated using the coefficients shown in Table 5. As stated in Equation (6), the impact of agitation rate and feed flow rate is insignificant amongst all predictors in calculating steady state reaction conversion XA.

\[
X_A = -0.240 + 0.011X_1 + 0.001X_2 - 0.001X_3 + 0.295X_4 + e
\]  

(6)

The investigation of residual described through Table 6 characterizes the highest and lowest steady state NaOH conversion expected by the model of Equation (7). As observed, maximal XA equals 0.6024, minimal XA = 0.3501, which specifies that the conversion predicted by the model is not very good and may be further amended for predicting higher reaction conversion. Relying on a highly insignificant predictor as depicted in Table 5, X3 (Feed flow rate) was removed in the first step for further analysis.

Table 6. Statistics- residuals with four predictors.

|                  | Minimal | Maximal | Mean | Std. deviation | N |
|------------------|---------|---------|------|----------------|---|
| Predicted value  | 0.350127 | 0.602460 | 0.493438 | 0.0579618 | 16 |
| Residual         | -0.1370079 | 0.0531587 | 0.0E-7 | 0.0521474 | 16 |
| Std. predicted value | -2.472 | 1.881 | 0.000 | 1.000 | 16 |
| Std. residual    | -2.250 | 0.873 | 0.000 | 0.856 | 16 |
The regression Equation (7) was formulated to augment the steady-state NaOH conversion.

\[ X_A = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_4 X_4 + e \]  
(7)

The coefficient of correlation with only three predictors declined slightly as compared to the four predictors, which is shown in Table 7. Fitness of model and contribution of extraneous variable also declined slightly with three predictors. F value is still significant with three predictors as stated in Table 8, which explain that the impact of three independent variables varies considerably.

Table 7. Summary of developed model with three predictors.

| Model | R   | R square | Adjusted R square | Std. error of the estimate | R square change | F change | df1 | df2 | Sig. F change | Durbin-Watson |
|-------|-----|----------|-------------------|---------------------------|----------------|----------|-----|-----|---------------|---------------|
| 1     | 0.739 | 0.546     | 0.432             | 0.0587488                 | 0.546          | 4.806    | 3   | 12  | 0.020         | 0.267         |

1. Independent variables: X₁, X₂, X₄

2. Depicted variable: X₄

Table 8. ANOVA with three predictors.

| Model | Sum of squares | df | Mean square | F       | Sig. |
|-------|----------------|----|-------------|---------|------|
| 1     | Regression     | 3  | 0.017       | 4.806   | 0.020|
|       | Residual       | 12 | 0.003       |         |      |
|       | Total          | 15 |             |         |      |

1. Dependent variable: X₄

2. Independent variables: X₁, X₂, X₄

The coefficient of predictors are summarized in Table 9. p value is statistically significant for X₁ and X₄ while insignificant for X₂. Equation (8) is formulated using the coefficient presented in Table 9. As reported in Equation (8), the stirrer rate’s contribution is marginal amongst all three predictors in forecasting the conversion with respect to sodium hydroxide (response variable).

\[ X_A = -0.305 + 0.011X_1 + 0.001X_2 + 0.293X_4 + e \]  
(8)

Table 10 signifies the highest and lowest steady state conversion for NaOH applying residual analysis predicted by model Equation (8). As stated in the Table, the highest NaOH conversion is 0.6019, which signifies that predicted conversion is not very good. The conversion can be further enhanced by the deletion or addition of some independent variables. There is not much difference on maximum and minimum conversion observed with four predictors and three predictors as indicated through Tables 6 and 10, respectively.

Table 9. Coefficients of predictors with three predictors.

| Model | Unstandardized coefficients | Standardized coefficients | t     | Sig. | 95.0% Confidence interval for B |
|-------|-----------------------------|---------------------------|-------|------|--------------------------------|
|       | B   | Std. Error | Beta |     | Lower Bound | Upper Bound |
| (Constant) | -0.305 | 0.221 |     | -1.377 | 0.194 | -0.787 | 0.177 |
| X₁    | 0.011 | 0.005 | 0.421 | 2.162 | 0.042 | 0.000 | 0.021 |
| X₄    | 0.293 | 0.098 | 0.581 | 2.983 | 0.011 | 0.079 | 0.506 |
| X₂    | 0.001 | 0.001 | 0.099 | 0.510 | 0.619 | -0.001 | 0.002 |
Table 10. Residuals statistics with three predictors.

|                  | Minimum   | Maximum   | Mean     | Std. Deviation | N  |
|------------------|-----------|-----------|----------|----------------|----|
| Predicted value  | 0.349652  | 0.601985  | 0.493438 | 0.0576003      | 16 |
| Residual         | -0.1351061| 0.0550606 | 0E-7     | 0.0525465      | 16 |
| Std. predicted value | -2.496  | 1.884     | 0.000    | 1.000          | 16 |
| Std. residual    | -2.300    | 0.937     | 0.000    | 0.894          | 16 |

On the basis of a highly insignificant predictor as depicted in Table 9, $X_2$ (stirrer rate) was removed in the second step for further analysis to optimize the reaction conversion. Regression Equation (9) was formulated to augment the NaOH conversion with only two predictors.

$$X_A = \beta_0 + \beta_1X_1 + \beta_4X_4 + e$$  \hspace{1cm} (9)

A moderate positive correlation is observed between dependent and independent variable as shown in Table 11. R square demonstrates that the goodness of fit is moderate while adjusted $R^2$ explains that extraneous variable has moderate influence on response variable. F value, as illustrated in ANNOVA Table 12, explains that the impact of independent variables varies considerably at a significance level of 0.05.

Table 11. Model summary with two predictors.

| Model | R   | R Square | Adjusted R Square | Std. Error of the Estimate | Change Statistics | Durbin-Watson |
|-------|-----|----------|-------------------|---------------------------|-------------------|---------------|
|       | 1   | 0.732 a  | 0.536             | 0.0570525                 | 7.507             | 0.296         |
|       | 2   | 0.732 a  | 0.536             | 0.0570525                 | 7.507             | 0.296         |

1. Independent variables: $X_1$, $X_4$
2. Dependent Variable: $X_A$

Table 12. ANOVA with two predictors.

| Model | Sum of squares | df  | Mean square | F     | Sig. |
|-------|----------------|-----|-------------|-------|------|
| 1     | Regression     | 0.049 | 2 | 0.024 | 7.507 | 0.007 |
|       | Residual       | 0.042 | 13 | 0.003 |       |      |
|       | Total          | 0.091 | 15 |       |       |      |

1. Dependent variable: $X_A$
2. Independent variables: $X_i$, $X_4$

The impact of all independent variables on steady-state NaOH conversion is shown in Table 13. The value of $p$ is statistically significant for $X_i$ and $X_4$. Equation (10) is formulated applying coefficients shown in Table 13. As observed in Equation (10), the effect of volume and temperature is positive and statistically significant in forecasting the NaOH conversion.

$$X_A = -0.261 + 0.011X_1 + 0.293X_4 + e$$  \hspace{1cm} (10)

Table 14 characterizes the maximal and minimal NaOH conversion as predicted by model Equation (10) applying residual analysis. The highest steady-state NaOH conversion is 0.6019. Whereas, the lowest conversion with respect to NaOH is equal to 0.3496. The findings of response variables with three predictors and with two predictors are almost the same.
Table 13. Coefficients with two predictors.

| Model | Unstandardized coefficients | Standardized coefficients | t | Sig. | 95.0% Confidence interval for B |
|-------|-----------------------------|---------------------------|---|------|--------------------------------|
|       | B | Std. error | Beta |      | Lower bound | Upper bound |
| 1     | Constant | −0.261 | 0.198 | −1.317 | 0.211 | −0.689 | 0.167 |
|       | $X_1$ | 0.011 | 0.005 | 0.421 | 2.226 | 0.044 | 0.000 | 0.021 |
|       | $X_4$ | 0.293 | 0.095 | 0.581 | 3.072 | 0.009 | 0.087 | 0.498 |

Table 14. Statistics-residuals with two predictors.

|                          | Minimum | Maximum | Mean   | Std. Deviation | N  |
|--------------------------|---------|---------|--------|---------------|----|
| Predicted value          | 0.349652| 0.601985| 0.493438| 0.0570784     | 16 |
| Residual                 | −0.1351061| 0.0560455| 0E-7 | 0.0531130     | 16 |
| Std. Predicted value     | −2.519 | 1.902  | 0.000 | 1.000         | 16 |
| Std. residual            | −2.368 | 0.982  | 0.000 | 0.931         | 16 |

1. Dependent variable: $X_A$

The two predictors have a positive impact and are statistically significant, hence an attempt was made to check the model based on a 2nd order polynomial to optimize the reaction conversion. To optimize the product formation, a 2nd order regression model among important (significant) variables was developed to examine the influence of significant factors (temperature, $X_1$ and volume, $X_4$) on predicted response (conversion, $X_A$) as follows (Equation (11)):

$$X_A = \beta_0 + \beta_1 X_1 + \beta_2 X_4 + \beta_3 X_1 X_4 + \beta_4 X_1^2 + \beta_5 X_4^2 + e$$ (11)

where, $\beta_{16}$ and $\beta_{19}$ are the coefficient of predictors, while $X_1^2$ and $X_4^2$ are squares of their corresponding predictors. $X_1 X_4$ are interactions between two significant independent variables. The regression analysis was examined to obtain optimum NaOH conversion utilizing 2nd order polynomial as shown (Table 15). As observed, there is robust optimistic correlation among the significant variables and steady-state NaOH transformation to products. The $R^2$ (observed) showed that the model in Equation (11) is appropriate about 80%, while adjusted $R^2$ clarifies that the influence of external factors is nearly equal to 18%, which is considerably smaller and equated to the preceding results. The sig. F variation that is equal to 0.001 is considerably < 0.05. Also, this value is substantial with an adopted 5% level of significance. The 2nd order model of polynomial having two independent variables clarifies that F-value is smaller. The significance level is < 0.05, which signifies that the impact of each independent variable varies considerably with 5% significance level as represented in Table 16.

Table 15. 2nd order polynomial model summary with two predict.

| Model | R   | R square | Adjusted R square | Std. error of the estimate | Change Statistics | Durbin-Watson |
|-------|-----|----------|-------------------|---------------------------|-------------------|---------------|
|       |     |          |                    |                           | R square change   | F change | df1 | df2 | Sig. F change |                |
| 1     | 0.890 | 0.793 | 0.718 | 0.0414313 | 0.793 | 10.530 | 4 | 11 | 0.001 | 0.914 |

1. Predictors: $X_1$, $X_4$, $X_1^2$, $X_4^2$, $X_A$
2. Dependent Variable: $X_A$

Table 16. ANOVA for 2nd order polynomial with two predictors.

| Model | Sum of squares | df | Mean square | F | Sig. |
|-------|---------------|----|-------------|---|------|
| 1     | Regression    | 0.072 | 4 | 0.018 | 10.530 | 0.001 |
|       | Residual      | 0.019 | 11 | 0.002 |
|       | Total         | 0.091 | 15 |

a. Dependent Variable: $X_A$

b. Predictors: (Constant), $X_1$, $X_4$, $X_1^2$, $X_4^2$, $X_A$
The coefficient in Table 17 is utilized to formulate a relationship applying values of β to obtain the best solution for the hydrolysis of CH₃COOC₂H₅ by NaOH. It can be established from Equation (9) that 100% rise of temperature may augment the conversion of NaOH by 2.10% while keeping all other predictors constant. The maximal and minimal NaOH conversion under steady-state condition equal to 63.15% and 28.03% with an average conversion rate of 49.34% was obtained using residual analysis as presented in Table 18. The 63.15% NaOH conversion obtained using Equation (9) is the maximum amongst four models. An increase in reaction NaOH conversion to product capacity was observed using second or der polynomial (Xₐ = 0.6315) as compared to a lower value of (Xₐ = 0.60198) using the same predictors with 1st order polynomials.

\[
X_a = -2.189 + 0.021X_1 + 3.480X_4 + 0.001X_1^2 - 0.862X_4^2 - 0.029X_1X_4 + \text{error} \quad (12)
\]

**Table 17. Coefficients for 2nd order polynomial with two predictors.**

| Model | Unstandardized coefficients | Standardized coefficients | t | Sig. |
|-------|-----------------------------|---------------------------|---|-----|
|       | B                           | Std. Error                | Beta |     |     | Lower bound | Upper bound |
| (Constant) | -2.189                     | 0.706                     | -3.101 | 0.010 | -3.742 | -0.635 |
| X₁    | 0.021                       | 0.006                     | 1.235 | 3.125 | 0.037  | .258       | 0.485       |
| X₄    | 3.480                       | 1.050                     | 6.909 | 3.316 | 0.007  | 1.170      | 5.791       |
| X₁²   | 0.001                       | 0.001                     | 2.142 | 1.247 | 0.238  | -0.001     | 0.002       |
| X₄²   | -0.862                      | 0.260                     | -4.616 | -3.311 | 0.007  | -1.435     | -0.289      |
| X₁X₄  | -0.029                      | 0.029                     | -2.523 | -1.017 | 0.331  | -0.093     | 0.034       |

1. Dependent Variable: Xₐ

**Table 18. Statistics of residuals for 2nd order polynomial with two predictors.**

| Minimum | Maximum | Mean | Std. deviation | N |
|---------|---------|------|----------------|---|
| Predicted value | 0.280386 | 0.631567 | 0.493437 | 0.0694271 | 16 |
| Residual | -0.0895859 | 0.0484141 | 0E-7 | 0.0354797 | 16 |
| Std. predicted value | -3.069 | 1.990 | 0.000 | 1.000 | 16 |
| Std. residual | -2.162 | 1.169 | 0.000 | 0.856 | 16 |

1. Predicted variable: Xₐ

The regression standardized residual to determine the steady-state conversion of NaOH mechanism is depicted in Figure 1a. An equivalent variance is witnessed for the experiment as revealed. No substantial difference between an observed and expected values of predicted response (Xₐ) in terms of conversion of NaOH was found. Figure 1b exemplifies a plot (scatter) of NaOH conversion and validates that the values (experimental data) of reaction conversion lie on the straight line, and this shows that the random error is at a minimal and acceptable level. The curve also signifies outlier number for determining the best conversion. As depicted in the graph, the optimum level of reaction conversion is 0.63 (63%).
To further optimize the reaction conversion, a 3rd order model of polynomial with major factors was used to examine the effect of significant factors ($X_1, X_4$) on response (Conversion, $X_A$) as shown in Equation (13):

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_4 + \beta_3 X_1^2 + \beta_4 X_4^2 + \beta_5 X_1 X_4 + \beta_6 X_1^3 + \beta_7 X_4^3 + \beta_8 + e$$

(13)

$\beta_1$ to $\beta_8$: coefficient of predictors; $X_1^2$ and $X_4^2$ are third order terms of respective predictors. To check any significant impact of 3rd order polynomial on reaction conversion, regression analysis was examined as shown in Table 19. There is a robust positive relationship among the independent variable, NaOH conversion, and the polynomials of predictors. The coefficient of correlation is exactly the same in 2nd order and 3rd order polynomials. The outputs of $R$, $R^2$, and adjusted $R^2$ are accurately the same in 2nd and 3rd order polynomials. The 3rd order polynomial of two independent variables of smaller F value with significance level < 0.05 show that the impact of each independent variable varies significantly at 5% significance level as shown in Table 20. Even in the significance level, no changes were observed with the change in order of polynomial. A lower $p$ value states that the chances of an error in the findings is minimal in decimal percentage. This means that the confidence level is at its maximum capacity i.e., 99.9%.

Table 19. 3rd order polynomial model summary with two predictors.

| Model | R    | R square | Adjusted R square | Std. Error of the estimate | Change Statistics | Durbin-Watson |
|-------|------|----------|-------------------|---------------------------|-------------------|--------------|
|       |      |          |                   |                           | R Square change   | F change | df1 | df2 | Sig. F change |              |
|       | 0.890 | 0.793    | 0.718             | 0.0414313                 | 0.793            | 10.530    | 4   | 11  | 0.001         | 0.914        |

1. Predictors: $X_4X_1$, $X_4^2$, $X_1^2$, $X_1$

2. Predicted variable: $X_A$

Table 20. ANOVA for 3rd order polynomial with two predictors.

| Model | Sum of squares | df | Mean square | F       | Sig. |
|-------|----------------|----|-------------|---------|------|
| 1     | Regression     | 0.072 | 4  | 0.018 | 10.530 | 0.001 |
|       | Residual       | 0.019 | 11 | 0.002 | | |
|       | Total          | 0.091 | 15 | | | |

1. Predicted response: $X_A$

2. Predictors, $X_4X_1$, $X_4^2$, $X_1^2$, $X_1$
The coefficient in Table 21 is utilized to formulate the 3<sup>rd</sup> order polynomial equation of regression to get the best solution for the hydrolysis of CH₃COOC₂H₅ to synthesize C₆H₅OH and CH₃COONa as reaction products. As stated in Equation (14), the influence of extraneous variable declined in the 3<sup>rd</sup> degree polynomial as compared to the 2<sup>nd</sup> degree polynomial, which explained that the model with the 3<sup>rd</sup> degree polynomial improved with respect to reaction conversion. The coefficient of X₄ in Equation (11) depicts the inverse relationship between the temperature and reaction conversion, which contradicts the underlying theory. The contribution of volume with the 2<sup>nd</sup> order term is also negative and has a significant impact on reaction conversion. Significance values in Table 21 state that only the volume with 2<sup>nd</sup> order and 3<sup>rd</sup> order terms was significant with 95% confidence level. Whereas, the temperature with the 1<sup>st</sup> order term and the interaction of volume with the 1<sup>st</sup> order with 2<sup>nd</sup> order term were insignificant. As depicted in residual Table 22, the maximal and minimal steady-state conversion of NaOH equal to 63.15% and 28.03% with an average conversion rate of 49.34% were obtained as depicted in residual Table 22. Further, no change was observed in the reaction conversion with 3<sup>rd</sup> order polynomial. In addition to this, the 3<sup>rd</sup> degree polynomial contradicts the underlying theories. The analysis of results clearly states that there is no change in reaction conversion with change in order of polynomial. The results also state that the NaOH conversion is maximal with 2<sup>nd</sup> order polynomial based on significant factors.

\[ X_A = -0.139 - 0.044X_1 - 0.862X_2^2 + 0.705X_3 + 0.001X_4X_2^2 + e \]  

(14)

### Table 21. Coefficients of 3<sup>rd</sup> order polynomial with two predictors.

| Model | Unstandardized coefficients | Standardized coefficients | t | Sig. | 95.0% Confidence interval for B |
|-------|-----------------------------|----------------------------|---|------|-------------------------------|
|       | B   | Std. Error | Beta |       | Lower bound | Upper bound |
| (Constant) | -0.139 | 1.429 | -0.097 | 0.924 | -3.285 | 3.007 |
| 1     | X₁  | -0.044 | 0.043 | -1.746 | -1.017 | 0.331 | -0.139 | 0.051 |
| 1     | X₂  | -0.862 | 0.260 | -4.616 | -3.311 | 0.007 | -1.435 | -0.289 |
| 1     | X₃  | 0.705 | 0.281 | 4.197 | 2.503 | 0.029 | 0.085 | 1.324 |
| 1     | X₄X₂² | 0.001 | 0.000 | 2.393 | 1.247 | 0.238 | 0.000 | 0.001 |

1. Dependent Variable: X₄

### Table 22. Residuals Statistics for 3<sup>rd</sup> order polynomial model with two predictors.

|                  | Minimum | Maximum | Mean | Std. deviation | N  |
|------------------|---------|---------|------|----------------|----|
| Predicted value  | 0.280386 | 0.631567 | 0.493438 | 0.0694271 | 16 |
| Residual         | -0.0895859 | 0.0484141 | 0 | 0.0354797 | 16 |
| Std. predicted value | -3.069 | 1.990 | 0.000 | 1.000 | 16 |
| Std. residual    | -2.162 | 1.169 | 0.000 | 0.856 | 16 |

1. Depicted response: X₄

### 4. Results and Conclusions

The present study deals with the maximization of NaOH conversion using step-wise regression analysis in a CSTR. The dependency of temperature, volume, agitation rate, and feed rate on reactor performance was examined as well as the interaction outcome of the operating parameters. The agitation rate (X₄) and feed rate (X₅) proved to have an insignificant influence on the reaction conversion at a significant level of 5%. Consequently, the temperature (X₁) and reaction volume (X₄) were found to have a significant influence on the reaction conversion using step-wise regression. The temperature and volume dependence on steady state NaOH conversion were described by a polynomial model of 2nd
and 3rd order. A maximal steady-state conversion equal to 63.15% was achieved. No further improvement was found in the reaction conversion with 3rd order polynomial, so the 2nd order polynomial was considered as the optimum reaction conversion model. It may be stated that 2nd order regression polynomial model is adequate to represent the experimental data very well.

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