Prediction and Optimization of Sulphur Trioxide Yield from Calcination of Aluminium Sulfate Using Central Composite Design

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Abstract. Sulphur trioxides are common toxic gaseous pollutants which can be produced from alternative routes via calcination of aluminum sulfate derived from kaolin clay. Its demand increases geometrically, thus the need to optimize the yield of SO₃ from the calcination of alum is essential. The rate of alum decomposition was monitored by the formation of SO₃ via thermogravimetric analysis and X-ray fluorescence analysis. This study aimed to evaluate the effect of calcination temperature and curing time on the SO₂ conversion and yields using Face Central Composite Design and optimize the process conditions to evaluate the maximum yield of SO₃ using response surface methodology and its effects and interactions were investigated between 800–900 °C at 60-180 minutes. Results indicated that experimental data satisfied second order polynomial regression model for SO₃ conversion and SO₂ yield from TG analysis while XRF analysis satisfied first order model respectively. An increase in SO₂ conversion and yields was observed as the calcination temperature and time were increased both independently and simultaneously. The calcination temperature was found to have a stronger influence compared to the calcination time. Validation indicated agreement between experimental and predicted values with a regression value of 97.8 %, 97.77 % and 97.67 % for SO₃ conversion, SO₂ yield via TG and XRF analyses respectively. Based on the ANOVA, the SO₃ yield via XRF produced the best model with R²pred of 91.98% while SO₂ yield via TG analysis and SO₂ conversion had R²pred of 79.99% and 78.01% respectively. Optimization of the production of SO₃ was carried out and the optimal condition for SO₂ conversion, SO₂ yield via TG and XRF analyses were 90.11 %, 91.67 % and 75.81 % respectively at an optimal calcination temperature of 877.43 °C and time of 155.04 minutes respectively.

Keywords: Calcination temperature and time; Conversion; Face central composite design; Sulphur trioxide; Yield.
its demerits [4]. The thermal decomposition of aluminium sulfate results in the yield of sulphur trioxide which can be influenced by the calcination temperature, time and particle size of the aluminium sulfate in which the particle size was considered to be constant.

Optimization is an essential technique employed in improving the existing condition of a process [6] such as sulphur trioxide (SO₃) production and can be achieved through the use of Response Surface Methodology (RSM). The optimization involves either variation of a given parameter per unit time while the other parameter is held constant using RSM. Its techniques can be employed to establish functional relationships between responses of interest and some inputs [7] and based on their relationships, the dependent variables can be used to predict responses that can be compared with the experimental values [8]. The use of RSM cannot be overemphasized as it assists in the evaluation of several parameters simultaneously with their interactions by limiting the number of an experiment to be conducted, as well as optimize parameter processes and estimation of interactions [9, 10]. Central Composite Design (CCD) is amongst one of the several techniques of RSM employed to design experimental procedures which have the advantage of screening a wide range of parameters as well as evaluating single variable/ cumulative effect of the variables to response [11]. It can also determine the number of the experiment to be able to evaluate for optimization of variables and responses [12] and has been found to widely used for the optimization techniques for calcination processes to produce significantly better models compared to other models [13].

An understanding of the interaction of the factors is essential in evaluating their relationship because their interactions are difficult to be determined using the one-factor-at-a-time approach [14]. The three stages in implementing response surface techniques include the design of experiment i.e. Box- Behnken or Central Composite Design (CCD), development of a model equation through statistical and regression analysis and finally optimization of parameters via model equation [15]. RSM has found applications in numerous experimental designs ranging from palm oil transesterification [16], extraction processes [8], drilling process [17], biodiesel production [18], prediction of blended cement properties [19, 20, 21] and decomposition as well as other areas of engineering.

The aim of this paper is to investigate the effect of aluminum sulfate calcination temperature and time on the production of SO₃ through response surface methodology using central composite design (CCD) and interactions studied. The comparison of the SO₃ yields via TG and XRF techniques and SO₃ conversion to ascertain which produces the best yield. It also involves optimization of the process conditions for the production of SO₃ from the decomposition of aluminium sulfate derived from kaolin.

**EXPERIMENTAL DESIGN**

The summary of the design for responses; Sulphur trioxide conversion and yield estimation for XRF and TG values with calcination temperature and time as factors. The following parameters were chosen as independent variables: calcination temperature (800 °C, 850 °C, 900 °C), while the calcination time (60 min, 120 min, 180 min). Face central composite factorial design (3 level 2 factors) with 9 runs (1 block) (design expert 6.0) where -1 denotes low value of the independent variable (800 °C, 60 min), 0 used for the medium value (850 °C, 120 min) and the high value (900 °C, 180 min) were employed to investigate the effect of the above factors on the responses. A model was fitted to the response surface generated by the experiment.

$$Y_i = f(\text{Calcination temperature, Calcination time})$$

Design-Expert 6.0.8 software was employed to analyze the best fit data and to estimate the optimal value of the factors considered. RSM was used to determine the optimal process parameters to obtain maximum SO₃ content. CCD at 3 levels, 2 factors was selected as independent variables and the interaction of variables were estimated. 9 runs were carried out to fit the general model of equation (1) and to obtain economically optimum conditions for the SO₃ removal efficiency.

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_i^2 x_i^2 + \sum_{i=1}^{k} \beta_{ij} x_i x_j ,$$

Where $Y$ is the SO₃ yield, $\beta_0$ is the coefficient constant, $\beta_i$ is the linear coefficient, $\beta_{ii}$ quadratic coef-
ficient effect, $\beta_{ij}$ is the interaction coefficient effect and $X_i X_j$ is the coded values of variable $i$ and $j$ respectively. $Y_1$, $Y_2$, $Y_3$ denotes $SO_3$ conversion, $SO_3$ yield via TG and XRF analyses respectively. $X_1$ is the calcination temperature and $X_2$ is calcination time.

Table 1 indicates the experimental results for the determination of the $SO_3$ content via Thermogravimetric (TG) analysis and X-ray Fluorescence (XRF) analysis obtained from the calcination of alum derived kaolin to investigate its effect of calcination temperature and time on the $SO_3$ formation. The statistical analysis of the results was carried out by ANOVA to evaluate the model and its parameters were tabulated in Table 2.

The statistical significance was achieved by the F-test of the experimental result obtained. The model terms were selected or rejected based on the probability value with 95% confidence level. Then, the response surface contour plots are generated to visualize the individual and the interactive effects of the variables.

Table 1 - Experimental Design and Results

| Run | Temp °C, $X_1$ | Time min, $X_2$ | Conversion %, $Y_1$ | $SO_3$ TGA %, $Y_2$ | $SO_3$ XRF %, $Y_3$ |
|-----|---------------|----------------|-------------------|------------------|------------------|
| 1   | 800           | 60             | 8.30              | 7.55             | 6.33             |
| 2   | 800           | 120            | 12.60             | 12.97            | 8.63             |
| 3   | 800           | 180            | 16.97             | 17.46            | 11.59            |
| 4   | 850           | 60             | 48.55             | 49.95            | 25.62            |
| 5   | 850           | 120            | 68.29             | 70.25            | 45.91            |
| 6   | 850           | 180            | 80.16             | 82.47            | 57.28            |
| 7   | 900           | 60             | 97.40             | 94.44            | 93.75            |
| 8   | 900           | 120            | 97.40             | 97.26            | 95.49            |
| 9   | 900           | 180            | 97.40             | 97.36            | 97.23            |

Face central composite design was employed and the factors required include calcination temperature ($X_1$) and time ($X_2$) with the responses; $SO_3$ conversion ($Y_1$) and $SO_3$ yield from TG ($Y_2$) and XRF ($Y_3$) analyses. The factors and the response variables were investigated and the effect of the various factors on the responses were determined using design expert 6.0.8. Results indicated that a quadratic equation was obtained for $SO_3$ conversion and $SO_3$ yield from TG analysis whereas $SO_3$ yield from XRF analysis satisfied linear model:

$$Y_1 = -4037.45 + 8.67X_1 + 0.86X_2 - 0.0045X_1^2 - 0.000563X_2^2 - 0.0072X_1X_2$$  (3)

$$Y_2 = -4663.90 + 10.172X_1 + 0.79X_2 - 0.0055X_1^2 - 0.00057X_2^2 - 0.0058X_1X_2$$  (4)

$$Y_3 = -701.79 + 0.86X_1 + 0.11X_2$$  (5)

The Equations (3) to (5) represent quantitative effect of the factor variables; calcination temperature and time ($X_1$, $X_2$) and their interactions on the response; $SO_3$ conversion and $SO_3$ yield from TG and XRF values ($Y_1$, $Y_2$, $Y_3$). The values of $X_1$ and $X_2$ were substituted in the equation to obtain the theoretical value of $Y_1$, $Y_2$ and $Y_3$ respectively. Based on the experimental design and factor combination, linear model was found to be significant for $SO_3$ via XRF analysis amongst other responses which were significant for quadratic models.

Table 2 indicates the analysis of variance (ANOVA) for $SO_3$ conversion, $SO_3$ yield from TG analysis and $SO_3$ yield from XRF analysis, all gave F value for lack of fit was 2.34, 2.33 and 1.53 respectively which also confirms that the models are significant due to the fact that it has an insignificant lack of fit. Table 2 also indicates the model F values for $SO_3$ conversion, $SO_3$ yield for TG and $SO_3$ yield for XRF are 62.54, 69.16 and 125.09 respectively, thus the models are significant implying that there is 0.01% possibility that the noise will be large.

Tables 3-5 indicate that the Predicted $R^2$ value for the three responses were in logical conformity with the adjusted $R^2$ value for determination of the 3 responses. The several models produced adequate precision ratios indicating a desirable signal which was greater than 4 [22].
Table 2 – ANOVA for Response Surface Quadratic Model Analysis of Variance for Conversion and Percentage SO$_3$ Yield for XRF & TG analyses with Central Composite Design CCD

| Source          | Sum of Squares | DF | Mean Square | F Value | Prob > F |
|-----------------|----------------|----|-------------|---------|----------|
| Model Y$_1$     | 11558.43       | 5  | 2311.69     | 62.54   | < 0.0001 |
| X$_1$           | 10780.62       | 1  | 10780.62    | 291.65  | < 0.0001 |
| X$_2$           | 270.41         | 1  | 270.41      | 7.32    | 0.0304   |
| X$_1^2$         | 357.8          | 1  | 357.8       | 9.68    | 0.0171   |
| X$_2^2$         | 11.35          | 1  | 11.35       | 0.31    | 0.5968   |
| X$_1X_2$        | 18.79          | 1  | 18.79       | 0.51    | 0.4989   |
| Residual        | 258.75         | 7  | 36.96       |         |          |
| Lack of Fit     | 258.75         | 3  | 86.25       | 2.34    | 0.8240   |
| Model Y$_2$     | 11567.17       | 5  | 2313.43     | 69.16   | < 0.0001 |
| X$_1$           | 10506.86       | 1  | 10506.86    | 314.08  | < 0.0001 |
| X$_2$           | 342.77         | 1  | 342.77      | 10.25   | 0.015    |
| X$_1^2$         | 512.73         | 1  | 512.73      | 15.33   | 0.0058   |
| X$_2^2$         | 17.68          | 1  | 17.68       | 0.53    | 0.4908   |
| X$_1X_2$        | 12.22          | 1  | 12.22       | 0.37    | 0.5647   |
| Residual        | 234.17         | 7  | 33.45       |         |          |
| Lack of Fit     | 234.17         | 3  | 78.06       | 2.33    | 0.8240   |
| Model Y$_3$     | 11531.76       | 2  | 5765.88     | 125.09  | < 0.0001 |
| X$_1$           | 11259.73       | 1  | 11259.73    | 244.29  | < 0.0001 |
| X$_2$           | 272.03         | 1  | 272.03      | 5.09    | 0.0355   |
| Residual        | 460.93         | 10 | 46.09       |         |          |
| Lack of Fit     | 460.93         | 6  | 76.82       | 1.53    | 0.1176   |

Table 3 – Model Summary Statistics/ Sequential Model Sum of Squares for CCD for SO$_3$ Conversion

| Source       | Linear | 2FI  | Quadratic | Cubic |
|--------------|--------|------|-----------|-------|
| Sum of Squares | 11051.04 | 18.79 | 488.60    | 247.98 |
| DF           | 2      | 1    | 2         | 2     |
| Mean square  | 5525.52 | 18.79 | 244.3     | 123.99 |
| F value      | 72.12  | 0.23 | 6.61      | 57.54  |
| Prob > F     | < 0.0001 | 0.6406 | 0.0244    | < 0.0004 |
| Std. Dev.    | 8.75   | 9.11 | 6.08      | 1.47   |
| R$^2$        | 0.9352 | 0.9368 | 0.9781    | 0.9908 |
| Adj. R$^2$   | 0.9222 | 0.9157 | 0.9625    | 0.9978 |
| Pred. R$^2$  | 0.87173 | 0.752 | 0.7801    | 0.8941 |
| PRESS        | 1516.96 | 2930.38 | 2598.21   | 1251.95 |
| Suggested    |        |      | Suggested |       |
| Aliased      |        |      |           |       |

Authors [23] and [24] reported that a fitted model is said to be acceptable when the R$^2$ is not less than 80% and greater than 75% respectively. In this study, the predicted values for developed models had a good correlation with the experimental results as shown in Table 3 indicated R$^2$ values for 97.81%, 98.02% and 96.16% respectively while R$^2_{adj}$ value for SO$_3$ conversion, SO$_3$ yield via TG and XRF analyses were 96.25%, 96.60% and 95.39% respectively, indicating appropriateness of the developed model in predicting the SO$_3$ conversion, SO$_3$ yield via TG and XRF analyses for the two factors with R$^2$ and R$^2_{adj}$ value close to unity. Authors [25] and [26] stated that a better empirical model fit was obtained with the experimental data when the R$^2$ value is close to unity and observed that a relatively high R$^2$ value does not imply that the model is adequate, thus, [25] suggested that a R$^2_{adj}$ of above 90% is most appropriate to evaluate the model adequacy for the three responses which were closer to unity. Thus, indicating a good fit of the model to experimental results.

The analysis of variance showed the significant effect of the independent variables on the responses and determine the responses which were significantly affected by the various interactions. The following model terms X$_1$, X$_2$, X$_1^2$ were
considered significant while the model terms greater than 0.10 were considered not significant for experimental SO$_3$ conversion and SO$_3$ yield via TG analysis whereas, SO$_3$ yield via XRF analysis showed that only the linear model terms $X_1$, $X_2$ were considered significant. The calcination temperature, ($X_1$) obtained a F value of 291.65, 314.08 and 244.29, while for the calcination time ($X_2$) produced a F value of 7.32, 10.25 and 5.09 for the experimental SO$_3$ conversion, SO$_3$ yield for TG and XRF analyses respectively. The high F values are a strong indication that the effect of the calcination temperature is far more significant compared to the calcination time for all the models. The quadratic term of the temperature obtained a F values of 9.68 and 15.33 respectively with $p$ values falling within $p<0.05$ or $p<0.10$ respectively. The quadratic term of the calcination time as well as the product of the calcination temperature and time obtained low F values, thus indicating that their effect is insignificant for the first two responses. It could be concluded that both factors $X_1$ and $X_2$ significantly affected the responses.

| Source                  | Linear | 2FI | Quadratic | Cubic |
|-------------------------|--------|-----|-----------|-------|
| Sum of Squares          | 10849.63 | 12.22 | 705.32 | 227.42 |
| DF                      | 2      | 1   | 2         | 2     |
| Mean square             | 5424.82 | 12.22 | 352.66 | 113.71 |
| F value                 | 57     | 0.12 | 10.54    | 84.28 |
| Prob> F                 | <0.0001 | 0.7401 | 0.0077 | 0.0001 |
| Std. Dev.               | 9.76   | 10.22 | 5.78     | 1.16  |
| R$^2$                   | 0.9194 | 0.9204 | 0.9802 | 0.9994 |
| Adj. R$^2$              | 0.9032 | 0.8939 | 0.966   | 0.9986 |
| Pred. R$^2$             | 0.8403 | 0.6755 | 0.7999 | 0.9336 |
| PRESS                   | 1884.27 | 3829.56 | 2361.51 | 783.86 |

From the experimental results, statistical testing was carried out employing Fishers test for ANOVA and the statistical significance of the second-order model indicated that the regression is statistically significant ($P<0.0001$) for the first two responses while the third response statistical data satisfied linear model; however, the lack of fit is not statistically significant at 99% confidence level, thus the residual variance for the models were insignificant [27, 28]. The analysis of variance indicated significant effect of the independent variables on the responses.

| Source                  | Linear | 2FI | Quadratic | Cubic |
|-------------------------|--------|-----|-----------|-------|
| Sum of Squares          | 11531.76 | 0.79 | 197.1     | 248.28 |
| DF                      | 2      | 1   | 2         | 2     |
| Mean square             | 5765.88 | 0.79 | 98.55     | 124.14 |
| F value                 | 125.09 | 0.015 | 2.62     | 42.08 |
| Prob> F                 | <0.0001 | 0.9037 | 0.1412 | <0.0007 |
| Std. Dev.               | 6.79   | 7.15 | 6.13      | 1.72  |
| R$^2$                   | 0.9616 | 0.9616 | 0.9781 | 0.9988 |
| Adj. R$^2$              | 0.9539 | 0.9488 | 0.9624 | 0.997  |
| Pred. R$^2$             | 0.9198 | 0.8478 | 0.7808 | 0.8571 |
| PRESS                   | 962.12 | 1830.36 | 2628.47 | 1714.22 |

Normal Probability and Predicted vs Actual Plots. Figures 1 (b), 2 (b) and 3 (b) also indicated that there is a strong relationship between the predicted and actual values for SO$_3$ conversion, SO$_3$ yield for TG and XRF values respectively based on the results obtained.
Figure 1 – (a) Normal Plot of residuals indicating significance of the model developed for SO$_3$ conversion and (b) Predicted vs Actual plot of the model developed for SO$_3$ conversion

Figure 2 – (a) Normal Plot of residuals indicating significance of the model developed for SO$_3$ yield with TG analysis and (b) Predicted vs Actual plot of the model developed for SO$_3$ yield with TG analysis

Figure 3 – (a) Normal Plot of residuals indicating significance of the model developed for SO$_3$ yield with XRF and (b) Predicted vs Actual plot of the model developed for SO$_3$ yield with XRF

It could be inferred that the predicted model obtained from the Design Expert software was significantly adequate in predicting SO$_3$ conversion and SO$_3$ yield for TG and XRF values respectively. Tables 6–8 illustrate the predicted values, actual values and residual errors of SO$_3$ conversion and SO$_3$ yield via TG and XRF analyses respectively.

| Temp °C | Time min | Actual value % | Predicted Value % | Residual % |
|---------|----------|----------------|-------------------|------------|
| 800     | 60       | 8.3            | 3.07              | 5.23       |
| 800     | 120      | 12.6           | 13.97             | -1.37      |
| 800     | 180      | 16.97          | 20.83             | -3.86      |
| 850     | 60       | 48.55          | 59                | -10.45     |
| 850     | 120      | 68.29          | 67.74             | 0.55       |
| 850     | 180      | 80.16          | 72.43             | 7.73       |
| 900     | 60       | 97.4           | 92.18             | 5.22       |
| 900     | 120      | 97.4           | 98.75             | -1.35      |

Table 6 – Diagnostisc Case Statistics for SO$_3$ Conversion
Contour and 3D Plots. The correlation between the responses and the factors were further explained via contour and response surface plots. The diagnostic plots represented by Figures 4–6 employed to estimate the adequacy of the regression model which shows the response plots (3D) and the contour plots for the effect of factors $X_1$ (calcination temperature), $X_2$ (calcination time) on the first response $Y_1$ ($SO_3$ conversion), second response $Y_2$ ($SO_3$ yield with TG analysis) and third response $Y_3$ ($SO_3$ yield with XRF analysis) respectively. The response surface curves illustrate the interaction between the factors and determination of the optimal level of the factors for maximum response. The non-parabolic nature of contours implies no significant interaction between both factors [29] as observed in Figure 6.

The calcination temperature and time both caused an increase in the $SO_3$ conversion and yield % when their values were increased from lower level to higher level as observed from the 3D surface plots. The plotted response surface curves were employed to elucidated the interaction of the factors and to determine the optimal level of each factor for a maximum response. From the predictive model, an increase in the calcination temperature from 800–900 °C at constant time of 60, 120 and 180 minutes led to a significant increase in the $SO_3$ conversion respectively as illustrated in Figure 7.

Table 7 – Diagnostic Case Statistics for $SO_3$ Yield via TG Analysis

| Temp °C | Time min | Actual value % | Predicted Value % | Residual % |
|---------|----------|----------------|-------------------|------------|
| 800     | 60       | 7.55           | 2.51              | 5.04       |
| 800     | 120      | 12.97          | 14.35             | -1.38      |
| 800     | 180      | 17.46          | 21.12             | -3.66      |
| 850     | 60       | 49.95          | 59.73             | -9.78      |
| 850     | 120      | 70.25          | 69.82             | 0.43       |
| 850     | 180      | 82.47          | 74.85             | 7.62       |
| 900     | 60       | 94.44          | 89.7              | 4.74       |
| 900     | 120      | 97.26          | 98.04             | -0.78      |

Table 8 – Diagnostic Case Statistics for $SO_3$ Yield via XRF Analysis

| Temp °C | Time min | Actual value % | Predicted Value % | Residual % |
|---------|----------|----------------|-------------------|------------|
| 800     | 60       | 6.33           | -1.94             | 8.27       |
| 800     | 120      | 8.63           | 4.79              | 3.84       |
| 800     | 180      | 11.59          | 11.53             | 0.064      |
| 850     | 60       | 25.62          | 41.38             | -15.76     |
| 850     | 120      | 45.91          | 48.11             | -2.2       |
| 850     | 180      | 57.28          | 54.85             | 2.43       |
| 900     | 60       | 93.75          | 84.7              | 9.05       |
| 900     | 120      | 95.49          | 91.43             | 4.06       |

Similar trends of an increase in the $SO_3$ yield from TG and XRF analyses were observed as the calcination temperature was increased at constant times of 60, 120 and 180 minutes illustrated in Figures 5–6 respectively. A significant increase in the $SO_3$ yield via TG and XRF analyses was experienced as both factors were gradually increased. Similarly, an increase in the $SO_3$ conversion was experienced as the calcination time was gradually increased from 60 to 180 min at constant calcination temperature of 800, 850 and 900 °C.
Figures 8 and 9 illustrate the effect of calcination time on the SO$_3$ yield via TG and XRF analysis at various constant calcination temperature. From the predictive model for the determination of the SO$_3$ via TG analysis, it could be observed that the SO$_3$ yield increased as the calcination time progressed from 60-180 minutes while the calcination temperature was held constant at 800, 825, 850, 875 and 900 °C respectively. The SO$_3$ yield via TG analysis increased from 24.32–43.54%, 49.93–65.67% as the calcination time progressed from 60–180 minutes at constant calcination temperature of 850 and 900 °C respectively. This increase in SO$_3$ yield could be attributed to the increase in the duration of calcination stemming from the increase in kinetic energy gained by the molecules to overcome the activation energy resulting in increased SO$_3$ yield.

Similar trend of an increase in the SO$_3$ yield via XRF analysis as the calcination time progressed at constant calcination temperature of 800, 825, 850, 875 and 900 °C respectively. The SO$_3$ yields via XRF analysis were found to be higher compared to those obtained from TG analysis. The values of SO$_3$ yield via XRF were also significantly close to SO$_3$ conversion values at various calcination temperatures and time compared to those of SO$_3$ yield via TG analysis. This could be attributed to the accuracy of the analyses of the SO$_3$ yield. The increase in yield of SO$_3$ from the decomposition of alum derived from kaolin clay could be attributed to the increase in amount of kinetic energy required to propagated the decomposition reaction as the temperature was increased or the calcination time progressed [29].
Figure 7 – Response surface plot (3D surface and Contour) indicating the optimal conditions ($X_1$: Calcination temperature, $X_2$: calcination time) for $SO_3$ conversion

Figure 8 – Effect of calcination time on the $SO_3$ yield via TG analysis at various calcination temperatures

Figure 9 – Effect of calcination time on the $SO_3$ yield via XRF at various calcination temperatures
It could be observed in Figure 10 and 11, that as the calcination temperature was gradually increased from 800–900 °C, there was a steady increase in the SO₃ yield for both XRF and TG analyses respectively. On the other hand, the predictive model for the determination of the SO₃ yield via XRF analysis, it could be seen that as the calcination time was held constant at 180 minutes and the calcination temperature was increased from 800–900 °C, the SO₃ yield via XRF increased from 6.01–92.01 %. Similar trend of an increase in the SO₃ yield via XRF was observed for other calcination time at 60, 90, 120 and 150 minutes respectively.

**Optimization.** Optimization of the production of SO₃ was conducted and the optimal conditions for optimal SO₃ conversion of 90.11 %, SO₃ yield via TG analysis of 91.67 % and SO₃ yield via XRF of 75.81 % at an optimal calcination temperature of 877.43 °C and time of 155.04 minutes. Figures 12–13 indicated similar trend of an increase in the SO₃ conversion and SO₃ yield obtained via TG and XRF analyses as the calcination temperature and time of the aluminum sulfate was simultaneously increased as illustrated by the response surface plots.
CONCLUSION
An increase in the calcination temperature and time between 800–900 °C and 60–180 minutes led to an increase in the SO₃ conversion, SO₃ yield via XRF and TG analyses respectively. Based on experimental results, an empirical relationship between the response and factors was obtained and found SO₃ conversion and SO₃ yield via TG analysis best suited with quadratic models whereas SO₃ yield via XRF satisfied a linear model. The SO₃ yields and conversion were established by the response surface and contour plots of the model-predicted responses. The SO₃ conversion and SO₃ yields via TG and XRF analyses of 90.11 %, 91.67 % and 75.81 % were obtained under optimal value of process parameters for calcination temperature of 877.43 °C and time of 155.04 minutes respectively. Analysis of variance for SO₃ conversion and SO₃ yields via TG and XRF analyses indicated a high coefficient of determination value for SO₃ conversion and yields (R² = 97.8%, R² adj = 97.06%) (97.77%, R² adj = 97.03) and (R² = 97.67 R² adj = 97.06) respectively. Thus, a satisfactory agreement of the second-order regression and first order model with the experimental data for TG and XRF analyses respectively. The calcination temperature provided the most significant effect on the SO₃ yields and conversion compared with calcination time. It was also observed from the ANOVA that SO₃ yield via XRF gave the best model with (R² pred = 91.98%) compared to SO₃ yield via TG analysis (R² pred = 79.99 %) and SO₃ conversion (R² pred = 78.01 %) respectively.
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CONFLICT OF INTEREST

The authors declared that they have no conflict of interest.

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