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Data Article

# Optimization of the calcination of brucite for the production of magnesia using response surface methodology

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ABSTRACT

An environmentally friendly pathway and an alternative raw material for the synthesis of MgO from brucite (produced from dolomite) using pentagonal experimental design under response surface methodology was investigated. The variables considered for the calcination include temperature between 510 and 890 °C and time between 13 and 40 min. Analysis of variance (ANOVA) shows that the developed model in this study is significant with a P-value of 0.0328, a high correlation coefficient ($R^2$) value of 0.9988, and an adjusted $R^2$ of 0.9957. According to XRD analysis, the periclase/magnesia (MgO) mineral phase has the most prominent peaks, while XRF analysis recorded the highest MgO of 72.72% at the optimum process conditions of 700 °C for 25 min. The MgO produced is categorized as reactive, it can therefore be used for waste water treatment, production of fertilizer, manufacturing of chemicals. Dolomite is a suitable alternative for the production of MgO.

## 1. Rationale

There are several dolomite deposit at different locations across the globe [1,2]. The outcome of the combined geological formation of calcium carbonate and magnesium carbonate together give rise to the mineral called dolomite [3]. Dolomite formation also comes along side with other impurities such as silica, iron etc. The methods used in beneficiating dolomites which includes silicothermic process and molten salt electrolysis for recovery of valuables require high energy usage and are expensive. Pyrohydrolysis process which is also a known method of producing MgO from dolomite is associated with the release of gaseous HCl and corrosive effect which makes the method not environmentally friendly [4]. The one of the current major source of industrial MgO is from seawater; the occurrence of boron in seawater takes the form of a non-dissociated orthoborate acid ($H_3BO_3$) and borate ions ($H_2BO_3^{2-}$ and $BO_3^{3-}$). This boron is adsorbed onto the produced MgO thereby serving as impurity with negative impact on it strength [5,6]. It then becomes necessary to seek an alternative MgO synthesis that is more ecofriendly and also at reduced production cost.

MgO is one of the valuable metal oxides in the metallurgical industry, this is due to it different areas of application. The high melting temperature of about (2800 °C) makes it suitable for use as refectory material [7]. The growing industrialization is not without the release of toxic waste water which has continue to constitute serious damage to the environmental and the human population. MgO

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**Table 1**
Boundary conditions for pentagonal experimental design.

| Design variables | Units | Type | Minimum | Maximum | Coded Low | Coded High | Mean | Std. Dev. |
|---|---|---|---|---|---|---|---|---|
| Time | Minutes | Numeric | 13 | 40.00 | -1 ↔ 10.00 | +1 ↔ 40.00 | 25.00 | 8.96 |
| Temperature | °C | Numeric | 510 | 890.00 | -1 ↔ 500.00 | +1 ↔ 900.00 | 700.00 | 119.53 |

**Table 2**
Pentagonal design for the calcination of $Mg(OH)_2$.

| Run | A:Time (Minutes) | B:Temperature (°C) | Calcination Yield (%) | | Residual | | |
|---|---|---|---|---|---|---|---|
| | | | Actual | Predicted | | | |
| 1 | 30 | 890 | 61.3 | 61.3 | 0 | $R^2$ | 0.99 |
| 2 | 13 | 582. | 59.5 | 59.5 | 0 | Adjusted $R^2$ | 0.95 |
| 3 | 25 | 700 | 61.5 | 61.30 | 0.1967 | | |
| 4 | 25 | 700 | 61.21 | 61.30 | -0.0933 | | |
| 5 | 40 | 700 | 60.3 | 60.30 | 0 | | |
| 6 | 30 | 510 | 60.5 | 60.50 | 0 | | |
| 7 | 13 | 818 | 62.0 | 62.00 | 0 | | |
| 8 | 25 | 700 | 61.2 | 61.30 | -0.1033 | | |
| Sum | | | | | 7E-6 | | |

is one of the economic materials that can be used for the treatment of this industrial waste water effluents. It can also be used for the scrubbing of toxic gases such as sulphur dioxide from industrial waste gas stream [7,8]. Other areas of application include treatment of constipation, heartburn, magnesium deficiency and indigestion [9,10].

Process optimization is very useful in reducing the cost and the cumbersome experimental runs needed to arrive at the optimum point [11–15]. Despite the wide application of MgO, the information on the optimization process of MgO from $Mg(OH)_2$ is not properly documented. This study is therefore aimed at establishing an optimized eco-friendly process conditions using response surface methodology in Design Expert 11.0 for the production of MgO from $Mg(OH)_2$ obtained from dolomite.

## 2. Experiment section

### 2.1. Procedure

In an earlier study by the same author, Are et al. [16], the dissolution of dolomite in nitric acid was established. $Mg(OH)_2$ was precipitated out of 50 mL of pregnant solution using 34% calcium oxide slurry within time of 8 min. During the precipitation process, the co-precipitation of calcium hydroxide was minimized by citric acid (0.1 M). The Pentagonal experimental design in design expert 11.0 was used for the calcination study due to the low number of experimental runs required for optimum points predictions compared to other optimization methods, effect of temperature and time were taken into consideration. Calcination time was studied between 10 and 40 min and temperature 400 to 800 °C. Eight experimental runs was generated by the pentagonal design, for each of the runs the muffle furnace used was preheated to calcination temperature, after which 0.004 kg of the magnesium hydroxide was measured into the crucible and charged into the muffle furnace for each of the runs. The calcination performance was determined by calculating a parameter called calcination efficiency (%Y), which was used as the response function in the RSM. The whole procedure was repeated for the other different experimental conditions in Table 1.

$$\%Y = \frac{M1 - M2\ X\ 100\ \%}{M1} \tag{1}$$

Where $M_1$ is the initial mass of magnesium hydroxide charged into the furnace and $M_2$ is the mass of magnesium hydroxide after the calcination time.

## 3. Data, value and validation

### 3.1. Regression model and statistical analysis

The pentagonal design in design expert 11.0 yielded eight (8) experimental runs. The responses (calcination yield) from the experimental results were simulated in order to obtain the regression and graphical analysis. The choice of quadratic model was based on the highest value of coefficient of regression ($R^2$) of 0.99, adjusted R-Squared of 0.95 as seen in (Table 2). Hence, the second order polynomial regression model best fits the relationship between the independent (temperature and time) and dependent (calcination yield) variable, Eq. (2) describes the relationship as follows:

$$Y = \beta_o + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{12} X_1 X_{2+} \beta_{12} X_1 X_3 + \beta_{23} X_2 X_3 + \beta_{11} X_1^2 + \beta_{22} X_2^2 + \beta_{33} X_3^2 \tag{2}$$

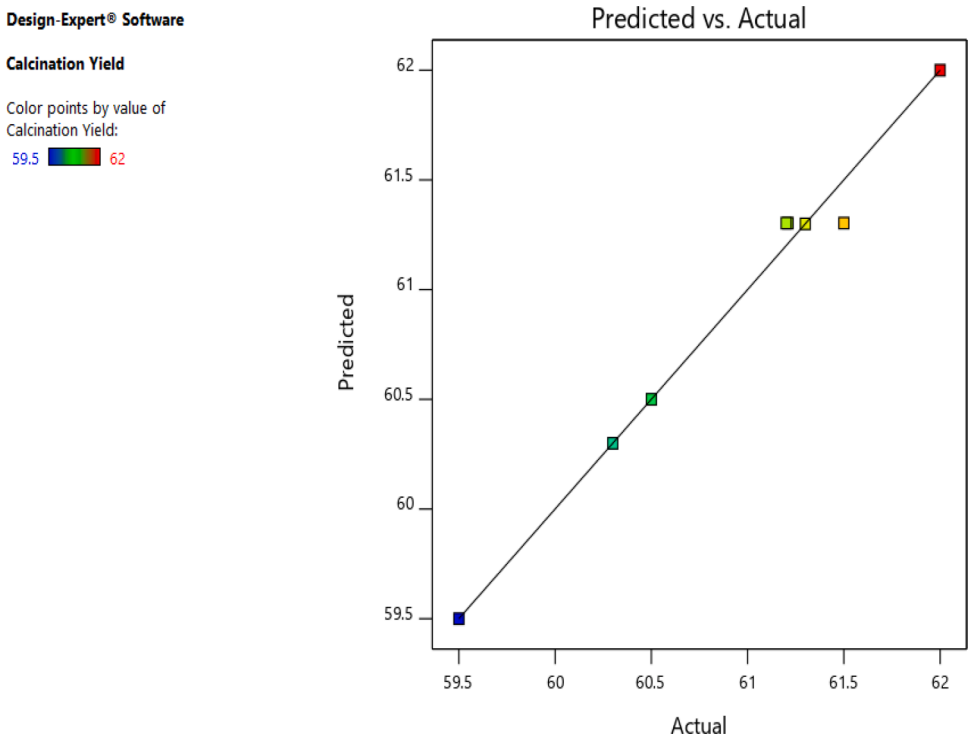


**Fig. 1.** Plot of the predicted against the actual calcination yield.

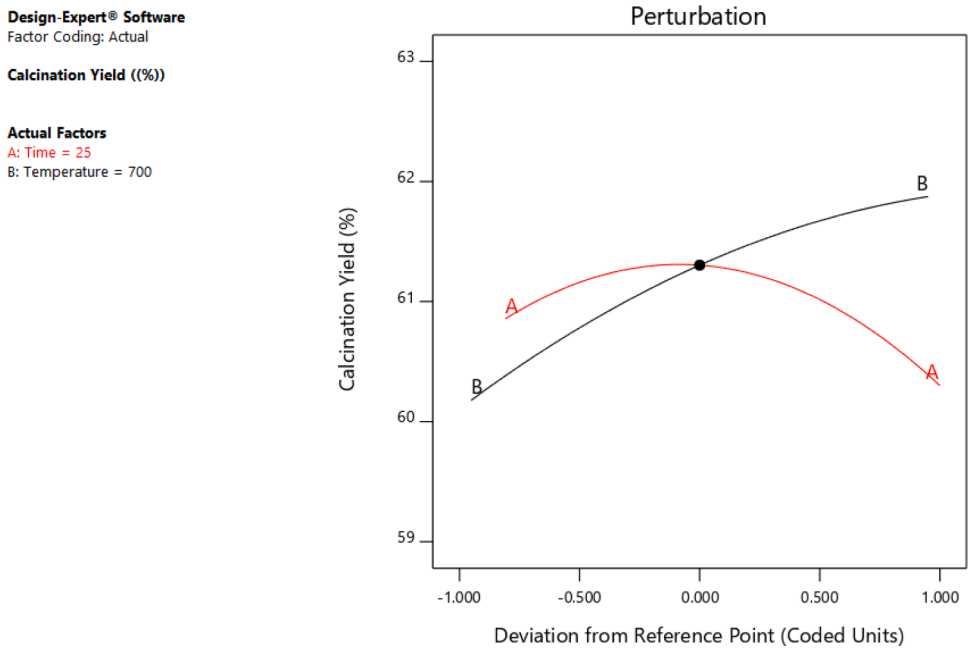


**Fig. 2.** Effect of time and temperature on the calcination yield.

Where Y is the predicted response, $\beta_o$ is the model constant, $X_1$, $X_2$, and $X_3$ are independent variables, $\beta_1$, $\beta_2$, $\beta_3$ are linear coefficients, $\beta_{11}$, $\beta_{12}$ and $\beta_{13}$ are quadratic coefficients (Rahman et al., 2011).The results in Table 2 shows that the developed quadratic model in Eqs. (3) and (4) was able to predict the calcination yield at the various experimental conditions. This model can be used to navigate the design space. The quadratic model in term of the coded factor for predicting the calcination yield in terms of coded parameters was

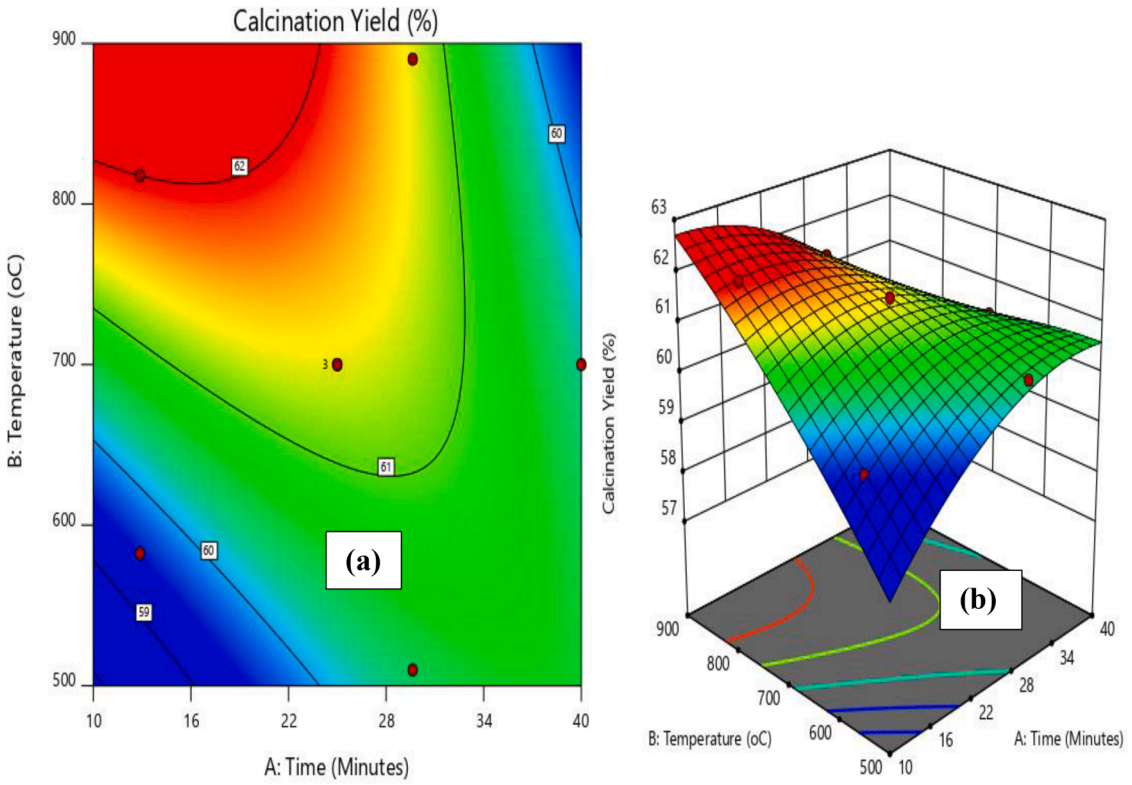


**Fig. 3.** (a) Contour plot, (b) three dimensional plot of Interactive effect of Time and Temperature on the calcination Yield from

developed from the pentagonal experimental design Eq. (3), while Eq. (4) is in terms of the actual factors.

$$\text{Calcination Yield}\,(\%) = +61.30 - 0.1430A + 0.8919B - 1.53AB - 0.8604A^2 - 0.3063B^2 \tag{3}$$

$$\begin{aligned}\text{Calcination Yield}\,(\%) = {} & +43.38060 + 0.537556\,\text{Time} + 0.027890\,\text{Temperature} \\ & -0.000508\,\text{Time} * \text{Temperature} - 0.003824\text{Time}^2 - 7.65723\text{E} - 06\text{Temperature}^2\end{aligned} \tag{4}$$

The predicted calcination yield values are very much close to the experimental results with residual value of 0.000007. The plot of the predicted values against the experimental results of the calcination yield is shown in Fig. 1.

The Model F-value of 29.80 implies the model is significant. There is only a 3.28% chance that an F-value this large could occur due to noise. *P*-values less than 0.0500 indicate model terms are significant. In this case B, AB, $A^2$ are significant model terms. Adequate Precision measures the signal to noise ratio. A ratio greater than 4 is desirable. Ratio of 16.942 obtained indicates an adequate signal. The model efficiency was further confirmed with high regression coefficient ($R^2$) value of of 0.9988 and adjusted $R^2$ of 0.9957.

### *3.2. Single effect of time and temperature on the calcination yield*

The individual effect of the process variables of time and temperature on the calcination yield is presented in Fig. 2. As time increases from 10 to 25 min, the calcination yield also increases slightly from 60.27 to 61.3%, while further increase in calcination time to 40 min resulted in decrease in the calcination yield back to 60.3%, this implies that the exposure time of $Mg(OH)_2$ to calcination is insignificant and this is in agreement with ANOVA that effect of calcination time has *P*-value of 0.3158. Increases in temperature from 500 to 652.8 °C shows a slight increase in the calcination yield from 60.11 to 61.9%, this implies that the effect of temperature is significant on the calcination yield and this in agreement with ANOVA that effect of calcination temperature has *P*-value of 0.0143.

### *3.3. Interactive effect of time and temperature on the calcination yield*

The contour plot of the combined effect of temperature and time on the calcination yield is shown in Fig. 3 (a), while the three dimensional plot is shown in Fig. 3 (b). It can be clearly seen that as temperature increases from 500 to 900 °C and time increase from 10 to 40 min, the calcination yield increases from 59 to 62%. The smallest blue region represent calcination yield between 59 and 60%, this implies that only small fractions of the calcined samples falls within this region. The green region has the widest area which means most of the calcined samples fall within calcination yield of 61%. The highest calcination yield of 62% falls within the red region with an average area, this means a good number of the calcined samples have calcination yield within this region.

**Table 3**
ANOVA for quadratic model of calcination yield.

| Source | Sum of Squares | df | Mean Square | *F*-value | p-value | | | |
|---|---|---|---|---|---|---|---|---|
| Model | 4.33 | 5 | 0.8652 | 29.80 | 0.0328 | Significant | | |
| A-Time | 0.0511 | 1 | 0.0511 | 1.76 | 0.3158 | Not Significant | $R^2$ | 0.9988 |
| B-Temperature | 1.99 | 1 | 1.99 | 68.51 | 0.0143 | Significant | Adjusted $R^2$ | 0.9957 |
| AB | 1.45 | 1 | 1.45 | 50.10 | 0.0194 | Significant | | |
| $A^2$ | 0.7931 | 1 | 0.7931 | 27.32 | 0.0347 | Significant | | |
| $B^2$ | 0.1005 | 1 | 0.1005 | 3.46 | 0.2039 | Not Significant | | |
| Pure Error | 0.0581 | 2 | 0.0290 | | | | | |
| Cor Total | 4.38 | 7 | | | | | | |

**Table 4**
Optimization constraints for the calcination yield.

| Name | Goal | Lower Limit | Upper Limit |
|---|---|---|---|
| A:Time | Minimize | 10 | 40 |
| B:Temperature | is target = 700 | 500 | 900 |
| Calcination Yield | is in range | 59.5 | 62 |

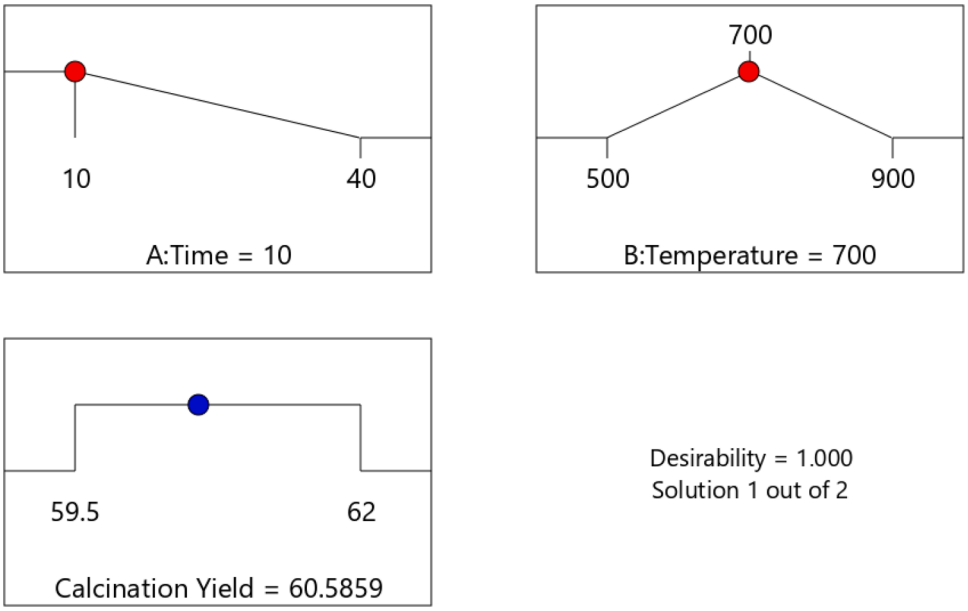


**Fig. 4.** Optimum predicted point for the calcination of yield.

The result of the ANOVA in Table 3 shows that effect of time is insignificant on the calcination yield, therefore the need to minimise its effect, while temperature is slightly significant on the calcination yield and its effect was then set at the center point to predict the optimim point Table 4. Optimum prediction via numerical optimization method indicates that calcination yield of 60.59% can be achieved at temperature of 700 °C and and time of 10 min (Fig. 4) calcination yield of 60.51% was validated at the predicted process conditions.

### *3.4. Characterization on synthesized MgO*

The XRD Result of Syntheses MgO is presented in Fig. 5. Two main prominent periclase (MgO) mineral diffraction peaks with JCPDS 45-0946 were identified at 42.90 °, 62.4 °, 72.8 ° and 74.47 ° in the synthesized MgO. Calcite ($CaCO_3$) mineral diffraction (JCPDS 5-0586) was identified at diffraction angles of at 29.33 °, other smaller calcite diffraction angles at 36.94 °, 47.36 ° and 57.44 ° are in traces with smaller intensity. This shows that calcite mineral as impurity is present in smaller amount.

The selected samples of the synthesized MgO were subjected to XRF analysis to determine the oxide composition and the result is presented in Table 5. At temperature of 700 °C and time of 25 min, the highest amount of MgO (72.72%) was recorded, followed by 69.78% MgO at 818 oC and 13 min and at 69.2% MgO was achieved at 582 °C and13 min. This implies that the process conditions at

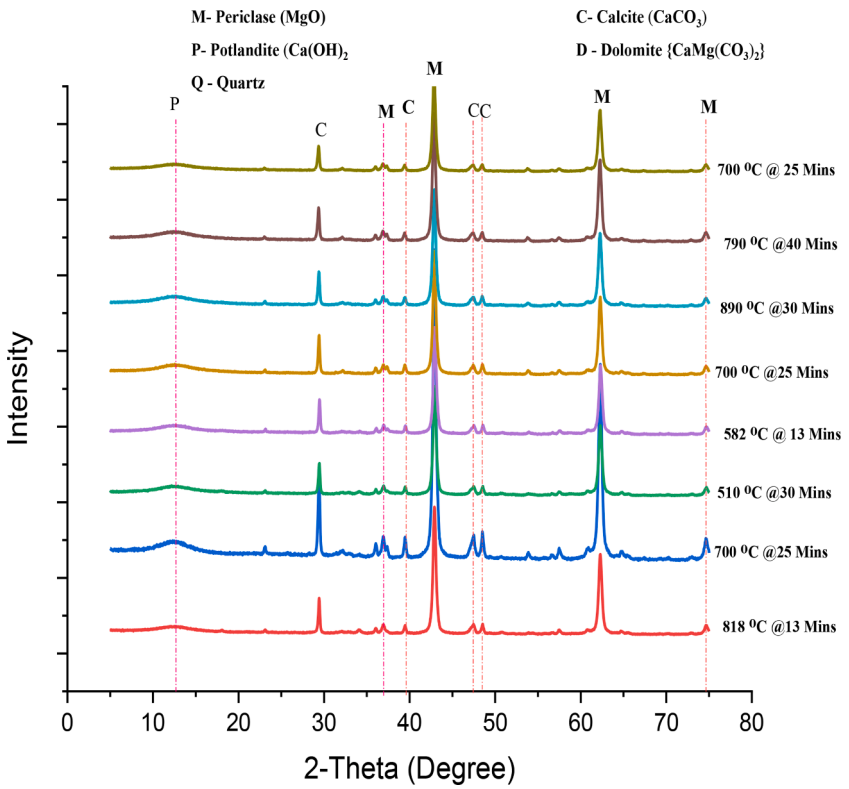


**Fig. 5.** XRD result of synthesized MgO.

**Table 5**
XRF result of the synthesized MgO.

| Sample | $Fe_2O_3$ | MnO | $Cr_2O_3$ | $V_2O_5$ | $TiO_2$ | CaO | $K_2O$ | $P_2O_5$ | $SiO_2$ | $Al_2O_3$ | MgO | $Na_2O$ | LOI | Sum |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| N1 | 0.33 | 0.02 | 0.00 | <0.01 | 0.06 | 14.66 | 0.01 | 0.50 | 1.10 | 0.38 | **69.78** | <0.01 | 12.41 | 99.26 |
| N4 | 0.34 | 0.02 | 0.01 | <0.01 | 0.07 | 13.89 | 0.01 | 0.46 | 1.08 | 0.41 | **69.20** | 0.04 | 14.37 | 99.87 |
| N7 | 0.35 | 0.02 | 0.01 | <0.01 | 0.06 | 14.64 | 0.02 | 0.48 | 1.05 | 0.49 | **72.72** | 0.06 | 10.06 | 99.95 |

N1: 818@ 13 Min, N4: 582 @ 13 Min, N7: 700 °C @25 Min.

700 °C and 25 min is better for producing of MgO from $Mg(OH)_2$, this might be due to the prolong calcination time. The MgO produced in this study is reactive, this is in agreement with the report by Fraga [6] that calcination temperature below 900 °C will produce MgO that has a fast reaction with dilute acid due to high surface area above 25 $m^2/g$. Therefore the reactive MgO can be used in agriculture, pharmaceuticals, manufacturing of chemicals, environmental [7–10]. The MgO purity of 72.72% in this study is close to the 76.4% obtained at 1700 °C and 1 h sintering time from magnesite by [6]. However, it is lower than the 88% MgO synthesized by Mubarok and Kurniawan [17] from dolomite. The outcome in this study is an alternative eco-friendly process route for the production of MgO as opposed to the other MgO production processes associated with the releases of poisonous hydrogen chloride gas. Fraga, [2020] reported that MgO from minerals (mines) sources is free of boron. Hence, the synthesized MgO in this study is considered to be boron free.

## 4. Conclusion

An eco-friendly optimization of the synthesis of MgO from $Mg(OH)_2$ (precipitated from dissolved dolomite in nitric acid solution) was carried out to examine the effect of the process parameters on the calcination process. The pentagonal design was used as the response surface, with 8 experimental runs. The quadratic model with a regression coefficient of 0.9536 best fits the prediction of the calcination yield. The analysis of variance shows the model is significant with a *p*-value of 0.00328. The result also shows that the effect of temperature was significant, while time seemed not to have a significant effect. 60.59% of the calcination yield was predicted at optimum conditions of temperature 700 °C and time of 10 min, while 60.51% was validated at optimum conditions of temperature and time with desirability of 1. The XRD analysis confirms the presence of MgO, while XRF analysis confirms the amount of the MgO present in the synthesized samples. The synthesized reactive MgO can be used for treatment of industrial toxic waste water, production

of magnesium metal, agriculture, manufacturing of chemicals, and other pharmaceutical applications. Hence, dolomite is a good alternative source of MgO.

## Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

## Data availability

No data was used for the research described in the article.

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