

# Kinetic assessment of the calcination reaction for bauxite sample containing boehmite mineral

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## ABSTRACT

The kinetic assessments for calcination reaction (dehydration reaction) of the bauxite sample was studied in this article under non-isothermal condition. After crushing and grinding the bauxite sample, the chemical analysis of the sample was obtained using x-ray fluorescence (XRF) method. The X-ray diffraction (XRD) patterns identified boehmite, hematite, and silica (silicon oxide) as the primary phases in the bauxite sample. The calcination reaction of the bauxite sample was evaluated using simultaneous thermal analysis (STA). The results of STA analysis indicated that the calcination reaction occurred at approximately 537 °C, with the final mass loss reaching about 13%. When the bauxite sample was heated to 600 °C in an air atmosphere for one hour, the boehmite phase decomposed, but the aluminum oxide phase did not fully crystallize. Kinetic evaluations were conducted using thermogravimetric analysis (TGA) data (STA analysis) under non-isothermal conditions, employing the Coats-Redfern method. The kinetic assessments revealed that the activation energy for the calcination reaction of the bauxite sample containing the mineral boehmite is approximately 198 kJ/mol, and the reaction follows the Crank equation of the diffusion models.

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## 1. Introduction

Bauxite is one of the most significant sources of aluminum in nature, primarily comprising the minerals gibbsite, boehmite, diaspore, or a mixture of these. Additionally, other compounds, including iron oxides (in the form of hematite or goethite), silica (silicon oxide, SiO<sub>2</sub>), titanium oxides (anatase), and other impurities such as kaolin (Kaolinite) and various oxides, can be found with bauxite [1–4]. Boehmite and diaspore, with the chemical formula Al<sub>2</sub>O<sub>3</sub>·H<sub>2</sub>O or AlO(OH), are among the hydrated aluminum oxides (single water molecule), differing in crystal structure and Mohs hardness. In contrast, gibbsite, with the chemical formula Al<sub>2</sub>O<sub>3</sub>·3H<sub>2</sub>O or Al(OH)<sub>3</sub>, contains three water molecules [1–4]. Both boehmite and diaspore have orthorhombic crystal structure, however the crystal structure of boehmite is similar to lepidocrocite mineral FeO(OH) [5]. The Mohs hardness of boehmite mineral is in the range of 3-3.5 and lower than diaspore mineral [6].

Previous studies on the kinetics of the calcination reaction and dehydration of aluminum hydroxide or minerals such as diaspore, boehmite, and gibbsite have presented the activation energy of the calcination reaction, the mechanism, and the governing kinetic equations [7–11]. However, variations in activation energy data have been observed in different studies depending on the type and properties of the material used (bauxite sample and its chemical composition), the conditions of the thermal analysis (TGA), or the method used for kinetic evaluation. The results and activation data about the dehydration kinetics of the nanocrystalline boehmite under non-isothermal conditions by Alphonse and Courty indicated the transformation of nanocrystalline boehmite into alumina can be accurately modeled by a 4-reaction mechanism involving: The loss of physisorbed water, the loss of chemisorbed water, the conversion of boehmite into transition alumina and the dehydration of transition alumina [12].

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Among the numerous methods for evaluating kinetic models and calculating the activation energy of a reaction under non-isothermal conditions, the Coats-Redfern method is one of the most common approaches. Using this method, activation energy ( $E_a$ ) can be determined from the slope of the line and the pre-exponential constant (A) from the intercept using equation (1) and plotting the left-hand side of the equation against the reciprocal of temperature ( $\frac{1}{T}$ ) in Kelvin. In equation (1), the terms R,  $\beta$ , and  $\bar{T}$  represent the universal gas constant, heating rate, and mean experimental temperature, respectively [13–16].

$$\ln \left[ \frac{g(\alpha)}{T^2} \right] = \ln \left[ \frac{AR}{\beta E_a} \left( 1 - \frac{2R\bar{T}}{E_a} \right) \right] - \frac{E_a}{RT} \quad (1)$$

Various equations are used for the kinetics of solid-state reactions, and Table 1 presents the integral form ( $g(\alpha)$ , integral reaction model) for some of them. By determining the reaction progress values ( $\alpha$ ) (extent of reaction) using thermal analysis data (TGA) and applying these data to the  $g(\alpha)$  functions listed in Table 1, graphs of  $\ln \left[ \frac{g(\alpha)}{T^2} \right]$  versus  $\left( \frac{1}{T} \right)$  can be plotted for the different mathematical equations listed in Table 1. Then, the mathematical equation that best fits the experimental data can be considered the optimal model by evaluating the results. It is worth mentioning that the theoretical values of the pre-exponential constant for the solid-state reactions should be in the range of  $10^6$ - $10^{18} \text{ s}^{-1}$  [13–16]. Considering these points, the related kinetic model can be selected from the equations in Table 1 for the reaction in question, and the kinetic data can be calculated.

Given the bauxite resources in Kohgiluyeh and Boyer-Ahmad province, there is limited information on the kinetics of the calcination reactions (dehydration reaction) of bauxite samples from this region. This paper examines the calcination reaction of the bauxite sample using STA analysis under non-isothermal conditions and the XRD patterns of the heated samples. The associated kinetic evaluation of the calcination reaction was conducted using the Coats-Redfern method, and the activation energy for the calcination reaction was determined in this article.

Table 1. Some equations of integral reaction model ( $g(\alpha)$ ) for solid-state rate reactions [13].

Reaction model	$f(\alpha)$	$g(\alpha)$
<b>Nucleation models</b>		
Power law	$4\alpha^{3/4}$	$\alpha^{1/4}$
Power law	$3\alpha^{2/3}$	$\alpha^{1/3}$
Power law	$2\alpha^{1/2}$	$\alpha^{1/2}$
Avrami-Erofeev	$4(1 - \alpha)[- \ln(1 - \alpha)]^{3/4}$	$[- \ln(1 - \alpha)]^{1/4}$
Avrami-Erofeev	$3(1 - \alpha)[- \ln(1 - \alpha)]^{2/3}$	$[- \ln(1 - \alpha)]^{1/3}$
Avrami-Erofeev	$2(1 - \alpha)[- \ln(1 - \alpha)]^{1/2}$	$[- \ln(1 - \alpha)]^{1/2}$
<b>Diffusion models</b>		
One dimensional diffusion	$1/2 \alpha^{-1}$	$\alpha^2$
Diffusion control (Jander)	$2(1 - \alpha)^{2/3}[1 - (1 - \alpha)^{1/3}] - 1$	$[1 - (1 - \alpha)^{1/3}]^2$
Diffusion control (Crank)	$3/2 [(1 - \alpha)^{-1/3} - 1]^{-1}$	$1 - 2/3 \alpha - (1 - \alpha)^{2/3}$
<b>Reaction order and geometrical contraction models</b>		
Mempel (first order)	$1 - \alpha$	$- \ln(1 - \alpha)$
Second order	$(1 - \alpha)^2$	$(1 - \alpha)^{-1} - 1$
Contracting cylinder	$2(1 - \alpha)^{1/2}$	$1 - (1 - \alpha)^{1/2}$
Contracting sphere	$3(1 - \alpha)^{2/3}$	$1 - (1 - \alpha)^{1/3}$

## 2. Materials and Methods

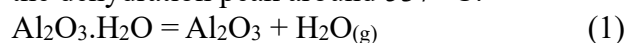
A bauxite sample was obtained from the Industry, Mine, and Trade Organization of Kohgiluyeh and Boyer-Ahmad province. After the crushing and grinding process, the powder sample was sieved using a 30-mesh sieve to separate the coarse particles of the bauxite. The chemical analysis of the bauxite sample was conducted using XRF and other chemical analysis methods (such as the ICP) at the Iran Mineral Processing Research Center-IMPRC (Tehran, Iran). The details of the chemical analysis for the bauxite sample were indicated in the previous research [17]. The XRF analysis results reported the sample's aluminum oxide content at approximately 58.15%, iron oxide (hematite phase) at about 15.4%, silica (silicon oxide) at 8.5%, titanium oxide at 2.62%, and the loss on ignition (L.O.I) at around 13.84%. The

predominant mineral in the bauxite sample was identified as boehmite using XRD analysis. The total oxides content (CaO, MgO, Na<sub>2</sub>O, K<sub>2</sub>O, P<sub>2</sub>O<sub>5</sub>) in the bauxite sample was less than 1.5% based on XRF analysis results.

Thermal analysis on the bauxite sample was conducted using an STA device (simultaneous thermal analysis, PerkinElmer STA6000) in an air atmosphere with a heating rate of 20 °C per minute in the Central Laboratory of Yasouj University. XRD evaluations were performed using a Rigaku Ultima IV device with copper radiation. Isothermal heating of the bauxite sample at 600 °C in an air atmosphere, with a holding time of one hour, was performed in a tube furnace (LENTON, LFT, 16 180) in the Metallography Laboratory of Yasouj University. Thermodynamic calculations were carried out using HSC software [18], and kinetic evaluations and activation energy calculations were performed using Excel, applying the equations listed in Table 1 after transferring the thermal analysis (STA) data.

### 3. Results and Discussion

The thermal analysis results on the bauxite sample, after grinding and passing through a 30-mesh sieve (test sample), are conducted and the results of TGA analysis and DTGA (differential TGA) presented in Figure 1. The graphs of STA analysis for bauxite sample have been presented before [17], however, the details of TGA and DTGA graphs are shown in Figure 1. According to Figure 1a., an endothermic peak is observed at around 537 °C in the DTGA curve of the bauxite sample, which, considering the mass loss in the TGA curve (Figure 1b.), can be associated with the chemical reaction (1). This endothermic peak at around 537 °C (Figure 1.a) was obtained from the results of STA analysis [17]. Reaction (1) represents the calcination of hydrated aluminum oxide. Given the presence of the boehmite phase in the bauxite sample, the results in Figure 1 demonstrate the calcination and dehydration reactions of the bauxite sample. According to Figure 1, the calcination reaction (reaction 1) begins at approximately 400 °C, with the dehydration peak around 537 °C.



Additionally, a small mass loss is observed at temperatures below 200 °C in the TGA curve, which, based on the little endothermic peak in the

dashed area in Figure 1a, is attributed to moisture evaporation (a physical process).

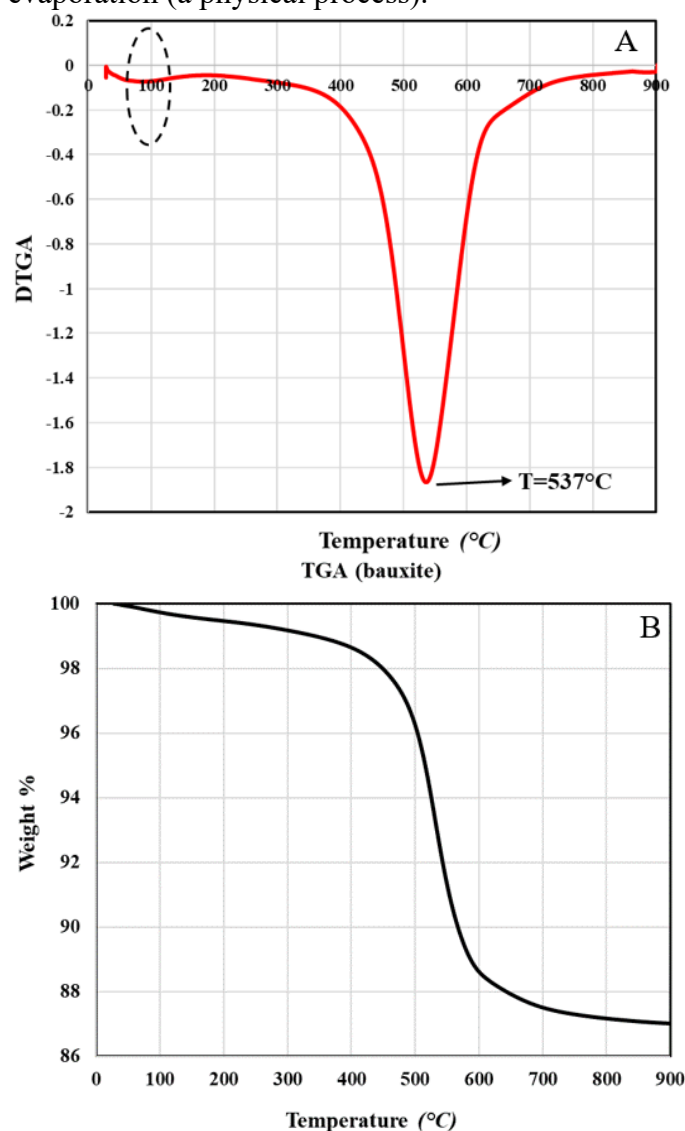


Figure 1. a) DTGA (differential TGA) graph, b) TGA graph from the STA analysis of bauxite sample.

According to thermodynamic calculations, reaction (1) is endothermic and feasible at temperatures above 220 °C [18], which aligns with the results in Figure 1. However, kinetic considerations must be considered for the reaction to occur. According to Figure 1, the final mass loss percentage in the bauxite sample after heating to 900 °C is approximately 13%, which corresponds well with the L.O.I reported in the XRF analysis. Mass balance calculations indicate that the mass loss percentage related to reaction (1) is 15%, but the mass loss observed in the bauxite sample, according to Figure 1, is less than the theoretical mass loss of reaction (1). Based on the XRF analysis, the aluminum oxide content in the bauxite sample is approximately 58.15%. If the

bauxite sample consisted of a 100% boehmite phase, the aluminum oxide content would be 85%. Therefore, the observed mass loss for reaction (1), as shown in Figure 1, is less than the theoretical value of 15%.

The XRD pattern of the bauxite sample (test sample) along with the solid product after heating at 600 °C for one hour is shown in Figure 2. Using phase identification software (HighScore Plus, version 5.2), the phases boehmite (JCPDS-021-1307), hematite (JCPDS-084-0311), and silicon oxide (SiO<sub>2</sub>) (JCPDS-80-1288) were identified in the bauxite sample, which is expected given the XRF analysis of the bauxite sample.

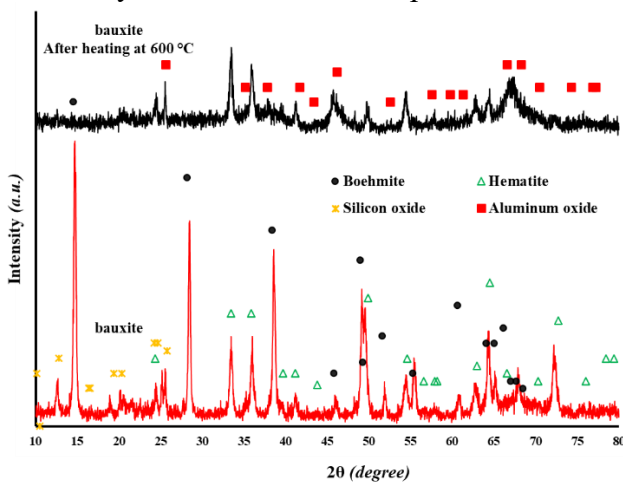


Figure 2. XRD patterns of the bauxite sample and bauxite after heating at 600 °C (1 h, air atmosphere). Hematite (Fe<sub>2</sub>O<sub>3</sub>), Silicon oxide (SiO<sub>2</sub>), Aluminum oxide (Al<sub>2</sub>O<sub>3</sub>)

According to Figure 2, the presence of peaks at angles around 14.5, 28, 38, and 49° indicates that the predominant mineral in the bauxite sample is boehmite with the chemical formula Al<sub>2</sub>O<sub>3</sub>·H<sub>2</sub>O. Upon heating the bauxite sample to 600 °C, the indications of the boehmite phase disappear, but the peaks for the hematite phase remain (i.e., at ~33.5 and 35.8°). Peaks at angles such as 25.5, 35, 37.7, 43, 52.5, and 57.5°, which correspond to the aluminum oxide phase (JCPDS-046-1212), indicate that despite the decomposition of the boehmite phase upon heating to 600 °C, the kinetic conditions for the crystallization of the aluminum oxide phase (the solid product of reaction 1) are not met at this temperature.

Figure 3 shows the changes in (α) as a function of temperature. Additionally, in Figure 3, the changes related to the 400-600 °C temperature range are plotted separately. For calculating the extent of

reaction (α) values in the graphs of Figure 3, the data from Figure 1b and equation (2) were used.

$$\alpha = \frac{(W_0 - W_t)}{(W_0 - W_f)} \quad (2)$$

The terms  $W_0$ ,  $W_f$  and  $W_t$  in equation (2) correspond to the initial mass, final mass, and instantaneous mass (mass at time, t), respectively, which are extracted from the TGA analysis data shown in Figure 1.

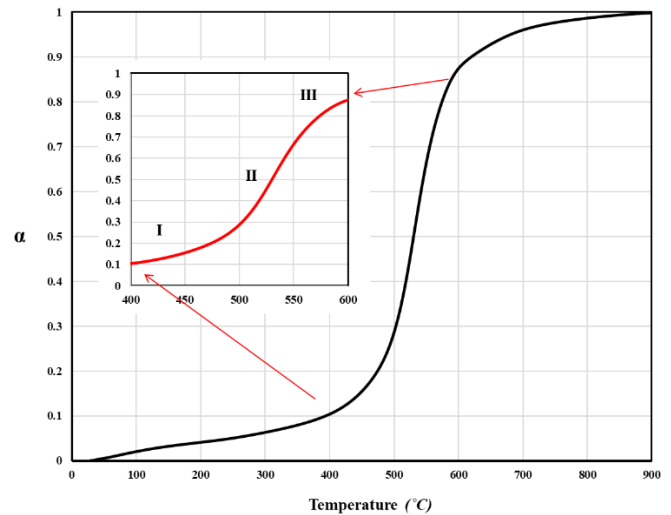


Figure 3. The change of extent of reaction (α) versus temperature and the change of (α) within 400-600 °C using the data of Figure 1 b.

According to Figure 1b., the temperature range of 400-600 °C pertains to reaction occurrence (1). As illustrated in Figure 3, three distinct regions with different slopes can be observed within this temperature range. It is important to note that every reaction requires an induction period for initiation. Based on Figure 3, it appears that after a certain time and reaching a temperature of approximately 500 °C, the rate of reaction (1) accelerates with increasing temperature. However, after 550 °C, the slope of the curve changes, and the reaction rate decreases with further temperature increases. These stages are all depicted in Figure 3. Kinetic reaction (1) evaluations were performed in the 500-550 °C temperature range, as indicated in Figures 1 and 3. All equations (integral reaction model) related to the kinetics of solid-state reactions were examined using Table 1. Then, using equation (1), the graphs of  $\ln\left[\frac{g(\alpha)}{T^2}\right]$  versus  $\frac{1000}{T(K)}$  were plotted for the various equations listed in Table 1. The equation whose line exhibited the highest correlation ( $R^2$  approaching 1, i.e.,  $R^2 \rightarrow 1$ ) with the experimental



data and whose pre-exponential constant (A) fell within the range of  $10^6$ - $10^{18}$   $s^{-1}$  could be considered the governing model in this method [13, 16]. Upon evaluating the related equations, these two conditions were satisfied for the Crank equation (equation 9 in Table 1), and the corresponding graph in the temperature range of 500-550 °C is shown in Figure 4. These two conditions were not simultaneously met for other equations listed in Table 1, such as equations 10 and 13.

Using the linear equation in Figure 4 and referring to equation (1), the slope of this equation indicates the activation energy, and the intercept represents the pre-exponential constant (A). The calculations for the kinetic equation shown in Figure 4 indicate an activation energy of 198 kJ/mol and a pre-exponential constant of approximately  $1.5 \times 10^{12}$   $s^{-1}$ . Based on these results, the formation of the solid product of the reaction influences the reaction (1) and the mechanism of boehmite calcination. As reaction (1) progresses and the boehmite phase decomposes, the formation of a solid phase (aluminum oxide) in the products affects the kinetics of reaction (1). It is worth noting that Figure 2 shows that despite the disappearance of boehmite phase indications due to heating at 600 °C (reaction 1), the complete crystallization of the aluminum oxide phase has not occurred and requires a higher temperature.

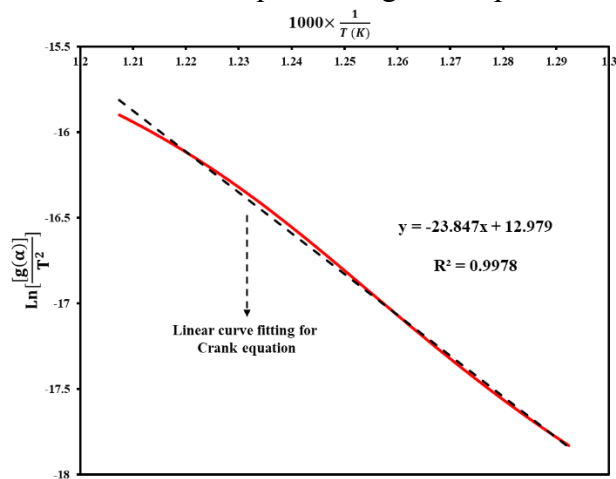


Figure 4. The graph of  $\ln\left[\frac{g(\alpha)}{T^2}\right]$  versus  $\left(1000 \times \frac{1}{T(k)}\right)$  for the Crank model of reaction (1) using the data of Figure 3.

Other studies have provided activation data for the dehydration reaction of bauxite samples (calcination reaction). The activation energy for a diasporic bauxite sample is approximately 175 kJ/mol [9], while for a bauxite sample that is a mixture of boehmite and diaspore, the activation

energy is reported to be in the range of 185-190 kJ/mol [10]. In another study, the average activation energy for the dehydration reaction of boehmite samples was determined to be in the range of 194-198 kJ/mol based on computational methods and the evaluation model used [19].

The summary of the fitted equation models for calcination (dehydration) reaction of the bauxite sample containing boehmite mineral are presented in Table 2. The results of Table 2 indicate that the computed amounts of pre-exponential constants using equation (1) for Mempel (first order) and Contracting sphere models are not in the range of  $10^6$ - $10^{18}$   $s^{-1}$ . The results of Table 2 show also these conditions are satisfied for other diffusion models such as Jander's equation. However, it should be noted that Jander's equation is applicable only to the hypothetical situations, and an improved models such as Crank equation can take into account in the actual situations [20].

Table 2. The summary of the fitted equation models for calcination (dehydration) reaction of the bauxite based on the reaction models in Table 1 and data of Figure 3.

Reaction model	The fitted equation	R <sup>2</sup>
Power law and Avrami-Erofeev models in Table1	Not fitted with experimental data	-
One dimensional diffusion	$y = -20.159 \times 10^3 + 10.295$	<b>0.995</b>
Diffusion control (Jander)	$y = -25.954 \times 10^3 + 15.757$	<b>0.998</b>
Diffusion control (Crank)	$y = -23.847 \times 10^3 + 12.979$	<b>0.998</b>
Mempel (first order)	$y = -13.828 \times 10^3 + 3.4685$	<b>0.998</b>
Contracting sphere	$y = -12.136 \times 10^3 x + 0.1408$	<b>0.998</b>

The previous results indicated the mechanism of boehmite transformation to  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> in the dehydration process involves the elimination of water formed by protons and hydroxyl groups and the migration of Al cations. It is reported that the diffusion of Al cations from octahedral to tetrahedral sites is a necessity for the formation of spinel  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> and it has been found to be a rate-determining step in the dehydration process of the boehmite [19]. It is also indicated that the dehydration process of boehmite is a topotactic reaction [19,21,22] and the activation energy of the dehydration process depends on the reaction extent [19]. The results of Xu et. al., [19] showed the dehydration process of boehmite is chemical

controlled with a diffusion complication as the reaction extent increases. Other results about dehydration processes of  $\text{Al}(\text{OH})_3$  and  $\text{AlOOH}$  indicated that the rate process is limited by the crystal-chemical transformation at the reagent-solid reaction product boundary surfaces [7].

The calcination reaction (dehydration) of the bauxite sample is a decomposition reaction and the solid products (i.e.,  $\text{Al}_2\text{O}_3$ ) of the reaction plays a significant role when the reaction proceeds. The results in Figure 4 and Table 2 indicate that the activation energy obtained using the Coats-Redfern method for the calcination reaction (dehydration reaction) of the bauxite sample containing the mineral boehmite falls within the range reported in other studies. By considering the fitted equation models in Table 2, The results of this article indicate the calcination reaction of the bauxite sample obeys the diffusion model and the Crank equation is the best fitted model for the experimental data.

#### 4. Conclusions

In The results showed that the predominant mineral in the bauxite sample used is boehmite, with other phases including hematite ( $\text{Fe}_2\text{O}_3$ ) and silicon oxide ( $\text{SiO}_2$ ). According to the results of STA analysis, the endothermic peak related to the calcination reaction (dehydration reaction) of boehmite was identified at approximately 537 °C. Despite the decomposition of the boehmite phase upon heating the bauxite sample to 600 °C in an air atmosphere for one hour, the kinetic conditions for the crystallization of the aluminum oxide phase were not achieved. Based on the kinetic evaluations and the method used in this paper, it appears that the kinetics of the boehmite calcination reaction in the bauxite sample is influenced by the formation of the solid product (aluminum oxide), with an activation energy of approximately 198 kJ/mol, which falls within the range of results from other studies. However, the type of bauxite sample, computational methods, and kinetic models can lead to variations in the results and activation energy data.

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#### Authors declaration

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