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ESTIMATION OF THE ACTIVATION ENERGY AS A FUNCTION OF THE EXTENT OF CONVERSION IN A THERMITE SYSTEM BY VARIOUS ISOCONVERSIONAL METHODS

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Abstract. *Solid energetic mixtures like thermites usually burn through a heterogeneous reaction where the burning reactants present more than one phase. So, the chemical reaction happens at the interface between reactants, where the particles interact with the neighboring particles. This process is complex and can be affected by the formation of products, crystalline defects, and experimental variables, following, in general, more than one-step mechanisms. Therefore, the estimated activation energy of this kind of reaction is generally an apparent value and can vary throughout the overall process. It is necessary to apply a proper kinetic method to obtain this variation. Isoconversional methods are more suitable for this application because they allow the activation energy estimation at several instants of the reaction. Also, they do not require the fitting of a reaction model. So, in this present study, various isoconversional methods are applied to obtain the activation energy as a function of the extent of conversion in the* Al-Ti-Fe_2O_3 *system. The data analyzed was retrieved from literature in the differential thermal analysis of this thermite system at three different heating rates. The differential method of Friedman and the flexible integral methods of Vyazovkin and Popescu, evaluated in small segments, captured substantial variations in activation energy as the extent of conversion varies. On the other hand, the rigid integral methods of Ozawa-Flynn-Wall, Kissinger-Akahira-Sunose, and Starink predicted nearly constant activation energies throughout the reaction process. Finally, among all methods analyzed, we indicate the isoconversional method of Popescu for the kinetics analysis of this kind of reaction because of its simplicity of application, accuracy, and proper estimation of the variations in activation energy during the combustion process.*

Keywords: *thermite, isoconversional method, model-free method, activation energy, heterogeneous reaction*

1. INTRODUCTION

A thermite reaction refers to a class of reactions that involves a more active pure metal reacting with a metal or non-metal oxide, resulting in a more stable oxide (Mei *et al.*, 1999). It is a highly exothermic reduction-oxidation reaction with a production of great amounts of heat at high temperatures and a self-sustained behavior. The main application of thermite has been at rail welding process (Lonsdale, 1999), but it also has applications in synthesis and processing of materials (Wang *et al.*, 1993), melting of municipal solid waste incinerator fly ash (Wang *et al.*, 2009), well abandonment (Mortensen, 2016), welding in space (Alvarez *et al.*, 2012), fabrication of lunar physical assets (Faierson *et al.*, 2010), as additive in solid fuel (Deng *et al.*, 2006), and pyrotechnic applications (Carter Jr and Carter, 2003). With the development of nanotechnology, nano-thermite has emerged and has been widely investigated for applications such as propulsive systems (Gangopadhyay *et al.*, 2011), joining (Zanjani, 2013), and well abandonment (Hearn *et al.*, 2018).

Thermite is part of an energetic material group other than propellants and explosives. Kubota (2007) explains that propellants cause high exhaust speeds, while explosives generate a shock wave. On the other hand, we have the pyrolyzing energetic materials, as thermites, which do not generate propulsive or destructive forces but are highly exothermic. When the reducing agent of thermite is aluminum, it is known as an aluminothermic reaction. Aluminum is the most commonly used fuel in this type of reaction because of its high affinity for oxygen, easy handling, high boiling temperature, and abundance (Zanjani, 2013)(Souza *et al.*, 2022). Although aluminothermic reactions are usually defined merely by a condensed-phase reaction, it is much more complex than that. It has many processes included, such as phase changes, decompositions, surface tension effects, fluidized particle movements, droplet collisions and transport of heat and species (Weiser *et al.*, 2010). All these mechanisms make thermite reactions very difficult to be modeled and characterized, which induced a few simplified numerical studies such as the ones carried out by Brito *et al.* (2005), Baijot *et al.* (2015), Souza

and de Lemos (2019, 2021).

Therefore, a detailed kinetics study is necessary to further increase the accuracy of these numerical models. As an initial contribution, the present study aims to compare the ability of various methods in accounting for variations in the effective activation energy in a thermite system. For this, the experimental data obtained by Rafiei *et al.* (2014), from differential thermal analysis (DTA) in the first stage of the Al-Ti-Fe_2O_3 reaction system, was analyzed through various isoconversional methods. Rafiei et al. had already applied the Kissinger method to estimate the activation energy as a function of the extent of conversion. However, this is a rigid integral method indicated for systems with low variability of the activation energy. Therefore, in addition to rigid-integral methods, more adequate methods as the Friedman differential method and the Vyazovkin flexible-integral method were applied in the present study to evaluate their ability in estimating the effective activation energy as a function of the extent of conversion.

2. THERMODYNAMICS OF THE REACTION

Rafiei *et al.* (2014) conducted thermal analysis of powder $3Al$-Ti-Fe_2O_3 system at three heating rates: $\beta = 5$, 15, and $20K/min$. They identified that the overall thermite reaction in this system occurs in two consecutive stages. In the first stage, Al_2O_3 was formed and they attributed this stage to the oxide-reduction reaction between aluminum and iron oxide, in the form of

$$2Al + Fe_2O_3 = 2Fe + Al_2O_3 \tag{1}$$

In the second stage, $(Fe, Ti)_3Al$ phases were formed, indicating the following mechanism

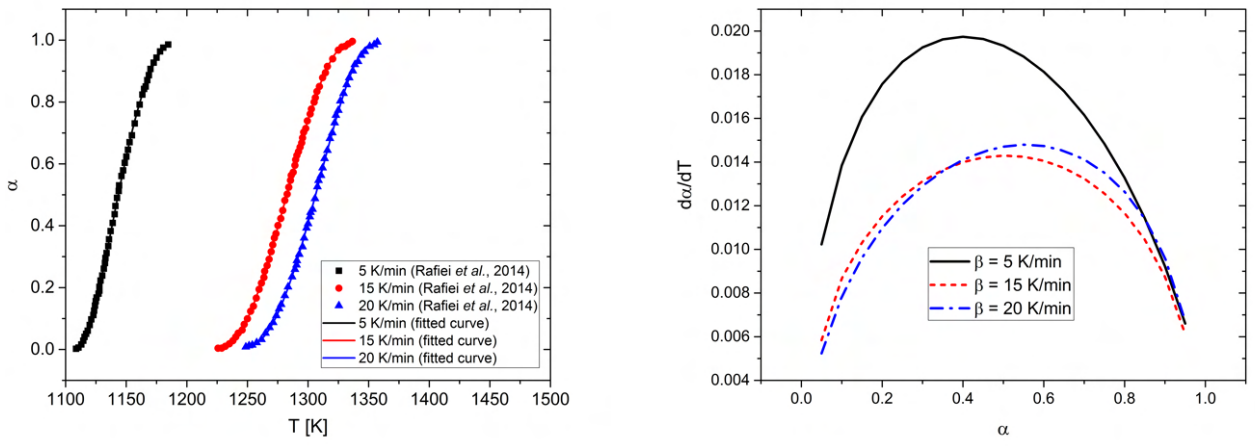
$$2Fe + Al + Ti = (Fe, Ti)_3Al \tag{2}$$

Kinetic analysis of solid-state reactions like this thermite system may be carried out by considering the following relationship

$$\frac{d\alpha}{dt} = Ae^{-\frac{E}{RT}} f(\alpha) \tag{3}$$

where α is the extent of conversion, t is time, A is the pre-exponential factor, E is the activation energy, R is the ideal gas constant, T is temperature, and $f(\alpha)$ is the reaction model.

Figure 1(a) shows α vs T data obtained by Rafiei *et al.* (2014) for the first reaction stage in the $3Al$-Ti-Fe_2O_3 system. Then, fitting curves of these data, at each heating rate, allowed to obtain the derivative of α with respect to temperature, as shown in Figure 1(b). Observed that these sigmoid-shaped curves describe auto-catalytic heterogeneous kinetics, where there is an acceleration of the reaction rate up to a maximum followed by deceleration. Once the data is retrieved at various heating rates, the isoconversional methods can be applied to estimate the effective activation energy as a function of α, as presented in the next section.



(a) (b)

Figure 1: (a) Data (symbols) of the extent of conversion, α, as a function of temperature from Rafiei *et al.* (2014), and corresponding fitting curves (lines); and (b) derivative of fitted α data with respect to temperature.

3. ISOCONVERSIONAL METHODS

3.1 Friedmann Method

One of the first isoconversional method for treatment of nonisothermal kinetics appeared in 1964 by Friedmann (Friedman, 1964), and is described by the relation

$$ln\left(\frac{d\alpha}{dt}\right)_{\alpha,i} = ln[f(\alpha)A_\alpha] - \frac{E_\alpha}{RT_{\alpha,i}} \tag{4}$$

which is a rearrangement of Equation 3 evaluated at each extent of conversion α and heating rate β_i identified by the index i. This method was proposed initially to study the decomposition of complex polymeric materials, which led Friedman to identify variations in the activation energy for decomposition of cured phenolic resin. This method is differential and does not require any assumption. However, it may amplify experimental errors in estimating the differential form of α with respect to time for experimental data of the integral type such as mass loss in TGA (Thermogravimetric Analysis). This differential form can also be calculated from temperature data as:

$$\left(\frac{d\alpha}{dt}\right)_{\alpha,i} = \beta_i \left(\frac{d\alpha}{dT}\right)_{\alpha,i} \tag{5}$$

Then, from the data shown in Figure 1, at each extent of conversion α, the left-side of equation 4 can be plotted against reciprocal temperature $T_{\alpha,i}$. Finally, the effective activation energy, at each extent of conversion, is estimated from the angular coefficient of each linear regression. This way, the Friemann method, as any isoconversional method, allows to obtain such dependence of the effective activation energy on the extent of conversion.

3.2 Ozawa-Flynn-Wall Method

The Ozawa-Flynn-Wall method was also first proposed in the 1960's to study decomposition of complex polymeric materials in which a variable activation energy was noticed (Ozawa, 1965, 1970, 1992; Flynn and Wall, 1966a,b). This method is described by the relation

$$ln(\beta_i) = Const. - 1.052\left(\frac{E_\alpha}{RT\alpha,i}\right) \tag{6}$$

This method is derived from the following integral form of Equation 3, assuming a constant heating rate β and $T = T_0 + \beta t$:

$$g(\alpha) = \frac{A}{\beta}\int_{T_0}^{T_\alpha} e^{E_\alpha/RT_\alpha} dT = \frac{A}{\beta} I(E_\alpha, T) \tag{7}$$

where $g(\alpha) = \int_0^\alpha$, and T_0 it the initial temperature at $t = t_0$. The Ozawa-Flynn-Wall method approximates this temperature integral shown in Equation 7 with an approximation function developed by Doyle (1962) to derive the final Equation 6. Therefore, this method employs integration as part of deriving the final equation, and thus it cannot be modified, which characterizes rigid-integral methods (Vyazovkin, 2015).

3.3 Kissinger-Akahira-Sunose Method

The Kissinger-Akahira-Sunose method (Kissinger, 1956, 1957; Akahira and Sunose, 1971) is described by the relation

$$ln\left(\frac{\beta_i}{T^2_{\alpha,i}}\right) = Const. - \left(\frac{E_\alpha}{RT\alpha,i}\right) \tag{8}$$

Like the previous method, this method is also rigid-integral, but it employs a more accurate integral approximation function presented by Murray and White (1955).

3.4 Starink Method

The Starink method (Starink, 2003) is also rigid-integral and is described by the relation

$$ln\left(\frac{\beta_i}{T^{1.92}_{\alpha,i}}\right) = Const. - 1.0008\left(\frac{E_\alpha}{RT\alpha,i}\right) \tag{9}$$

According to Starink (2003), the integral function approximation employed to derive Equation 7 is even more accurate than the previous ones.

3.5 Vyazovkin Method

The rigid-integral methods shown are simple to apply, however, they are accurate only for single-step mechanisms. If there is a significant dependence of the activation energy on the extent of conversion, these methods may not present accurate estimations since they assume constant activation energy from T_0 to T_α. To overcome the limits of traditional rigid-integral methods, some modern methods that compute the temperature integral as a part of estimating the activation energy were developed. They are also known as flexible-integral methods. The first one presented here is the non-linear Vyazovkin method (Vyazovkin, 1996, 2001), described by the relation

$$\phi(E_\alpha) = \sum_{i=1}^{n} \sum_{j \neq i}^{n} \frac{I(E_\alpha, T_{\alpha,i})\beta_j}{I(E_\alpha, T_{\alpha,j})\beta_i} \tag{10}$$

which estimates the activation energy from the minimization of the function ϕ. In this way, the temperature integrals in the form of Equation 7 can be evaluated from T_0 to T_α, or, in a more accurate way, it can be evaluated in small intervals of $\Delta\alpha$, from T_α to $T_{\alpha+\Delta\alpha}$.

3.6 Popescu Method

The Popescu method (Popescu, 1996) is also flexible-integral, and it applies the following mathematical theorem to solve the temperature integral:

$$\int_{T_\alpha}^{T_{\alpha+\Delta\alpha}} e^{-E_\alpha/RT_\alpha} dT = (T_{\alpha+\Delta\alpha} - T_\alpha) e^{-E_\alpha/RT_c} \tag{11}$$

where T_c is the corresponding temperature at which the exponential function presents a mean value in the interval $T_\alpha - T_{\alpha+\Delta\alpha}$. However, there is no simple way to determine T_c. As so, it is more practical to approximate it as $T_c = (T_{\alpha+\Delta\alpha} - T_\alpha)/2$, which is more accurate at smaller sizes of $\Delta\alpha$.

The Popescu method is then described by the relation

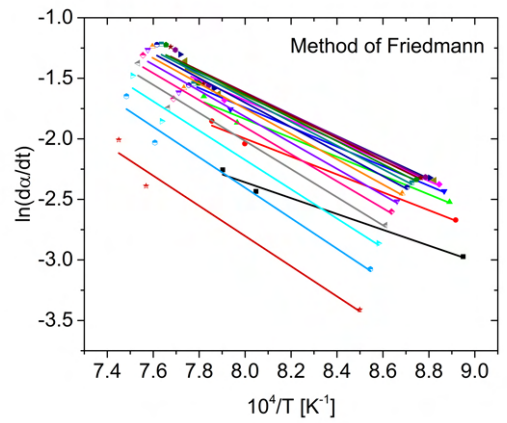
$$ln \frac{\beta_i}{T_{\alpha+\Delta\alpha,i} - T_{\alpha,i}} = Const. - \frac{E_\alpha}{RT_{c,i}} \tag{12}$$

which enables the activation energy evaluation as a function of the extent of conversion. This is done by plotting the left-side of this equation against reciprocal temperature T_c at each α and β_i, and calculating the the angular coefficient of linear regressions of the plots at each α, as the previous linear methods.

4. Results

Figure 2 shows the linearization plots for the differential method of Friedmann, rigid-integral methods of Ozawa-Flynn-Wall, Kissinger-Akahira-Sunose, and Starink, and the flexible-integral method of Popescu applied to Rafiel et al. (2014) data, shown in Figure 1. At each extent of conversion, α, the activation energy is estimated from a linear regression. Observe that the linear plots for rigid-integral methods presents an overall linear pattern as the extent of conversion varies. On the other hand, the differential method and the flexible-integral method studied presented plots that resembles a parabolic shape as the extent of conversion varies. Different from these linear methods presented in Figure 2, the non-linear method of Vyazovkin estimates the activation energy from a minimization process, as explained before. Therefore, Figure 3 shows the minimization plots for the flexible-integral method of Vyazovkin evaluated at $\alpha = 0.5$ and with segmented (integral from T_α to $T_{\alpha+\Delta\alpha}$) and non-segmented (integral from T_0 to T_α) temperature integrals. Observe that the more accurate segmented approach estimated lower activation energy than the non-segmented approach.

The mean activation energy was estimated for each method applied, in the overall extent of conversion range from $\alpha = 0.5$ to $\alpha = 0.95$. The rigid-integral methods estimated a mean value of 98.4 ± 1.3, 83.2 ± 1.4, and 84.0 ± 1.4 kJ/mol for Ozawa-Flynn-Wall, Kissinger-Akahira-Sunose, and Starink methods, respectively. The Friedmann method estimated a mean activation energy of 80.7 ± 14.6 kJ/mol. Finally, the flexible-integral method of Vyazovkin and the method of Popescu, applied to segmented intervals of $\Delta\alpha = 0.001$, estimated the same mean value of 80.7 ± 14.1 kJ/mol, while the method of Vyazovkin with a non-segmented evaluation estimated a mean value of 84.9 ± 1.4. Observe that the differential method and the flexible-integral methods evaluated in segmented intervals identified a much larger standard deviation from the mean value of the effective activation energy in comparison to the traditional rigid-integral methods and non-segmented Vyazovkin method. This observation enhances the superior capacity of the differential and flexible-integral methods to capture the variation of the effective activation energy throughout the reaction process, while the rigid-integral methods estimates a nearly constant value.



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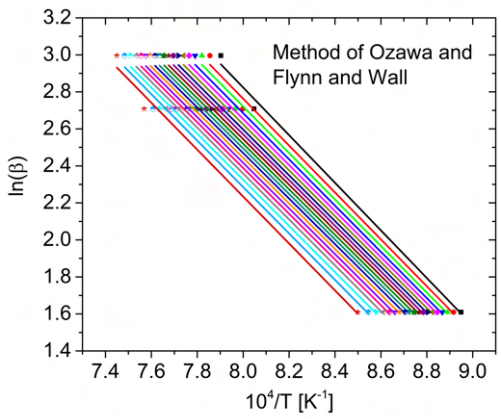

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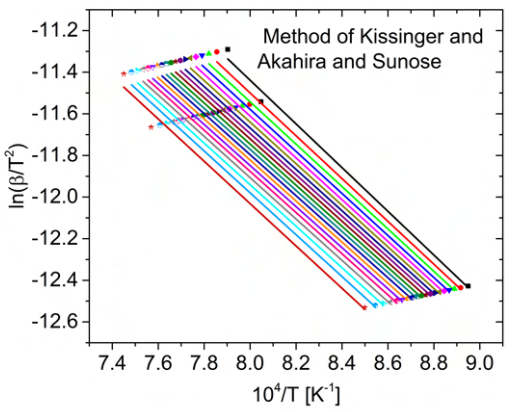

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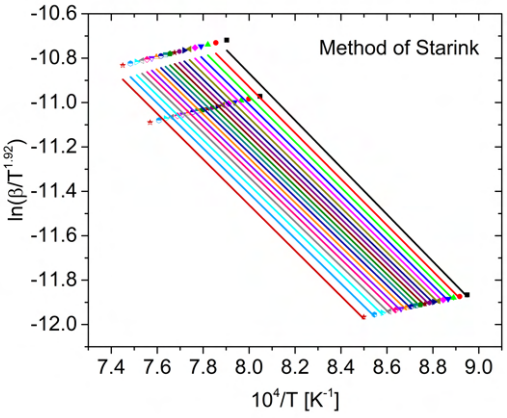

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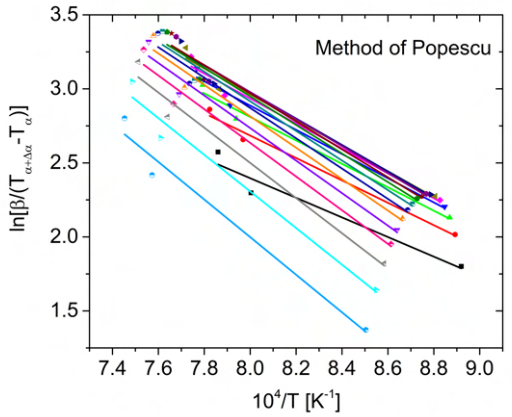

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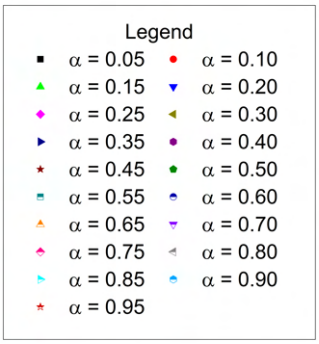

(f)

Figure 2: Linearization plots for the isoconversional method of (a) Friedmann, (b) Ozawa-Flynn-Wall, (c) Kissinger-Akahira-Sunose, (d) Starink, and (e) Popescu

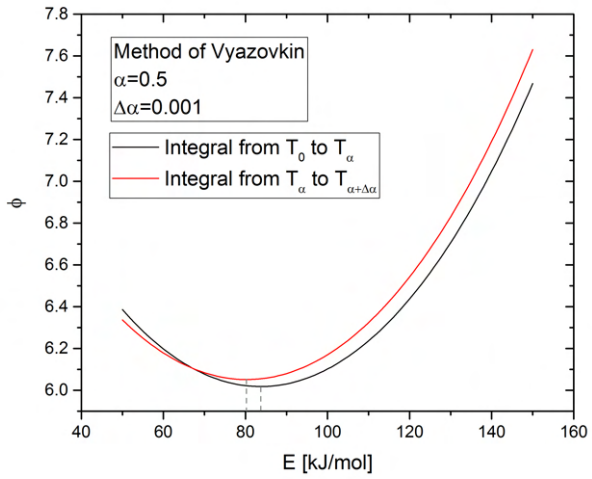

Figure 3: Minimization plots for the isoconversional method of Vyazovkin at $\alpha = 0.5$.

Figure 4 shows the results for activation energy as a function of the extent of conversion, α, for all methods considered in this study. The Ozawa-Flynn-Wall method overestimated the activation energy in most of the α range when compared to the other methods applied. The Kissinger-Akahira-Sunose method, Starink method, and the non-segmented method of Vyazovkin estimated a lower but also nearly constant activation energy. This low variability observed in the effective activation energy estimated from these methods are due to the evaluation of the integral term, shown in Equation 7, from T_0 to T_α, assuming constant activation energy in this temperature range. This methodology induces an increasing systematic error as α increases. This led Rafiei *et al.* (2014) to assume a one-step mechanism with only one activation energy of 82.3 kJ/mol estimated by the Kissinger method. On the other hand, the differential method of Friedmann and the flexible-integral methods of Popescu and Vyazovkin (segmented) captured a significant variation of the activation energy, increasing from $E = 55kJ/mol$ at $\alpha = 0.05$ to $E = 105kJ/mol$ at $\alpha = 0.9$, followed by a small reduction to $E = 102kJ/mol$ at $\alpha = 0.95$, which indicates a multi-step mechanism. These last two integral methods captured these variations due to the possibility of evaluating the activation energy in small segments of $\Delta\alpha$ in which the activation energy is assumed constant, and the Friedmann method is differential which does not require integral estimations.

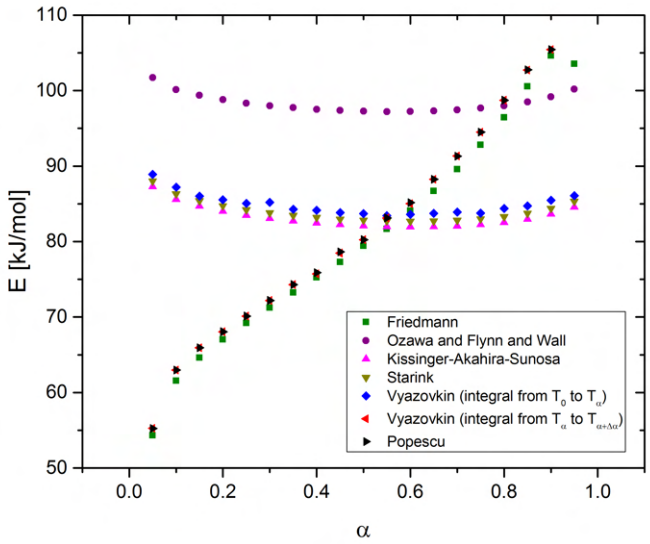

Figure 4: Activation energy varying with the extent of conversion according to different isoconversional methods.

Therefore, the assumption of one-step mechanism must be validated with appropriate methods indicated to evaluate the variations in effective activation energy. In this present study, the Popescu method is recommended due to the simplicity of application and precision. Also, the Popescu linear method presented the exact same results as the advanced non-linear isoconversional method of Vyazovkin evaluated in small intervals of $\Delta\alpha$, and it can be applied to data of the integral type

without amplification of experimental error in estimating differential forms.

5. CONCLUSIONS

This research presents an in-depth investigation of the activation energy variations through the first stage of the Al-Ti-Fe_2O_3 thermite reaction. Modern methods such as the Vyazovkin method (segmented) and the Popescu method showed a good ability to identify high variations of the activation energy, which led to the conclusion that this reaction stage follows a multi-step mechanism. Moreover, although the Vyazovkin method (segmented) is non-linear and the Popescu method is linear, they both presented identical results, which in turn are very similar to those obtained using the traditional Friedmann method. On the other hand, the traditional Ozawa-Flynn-Wall, Kissinger-Akahira-Sunose, and Starink methods were not able to capture the high variation in activation energy and estimated near-constant values, which can induce the improper assumption of a one-step mechanism.

6. ACKNOWLEDGEMENTS

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