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Heating Rate Behaviour Pyrolysis of Empty Fruit Bunches : Kissinger Kinetic Analysis

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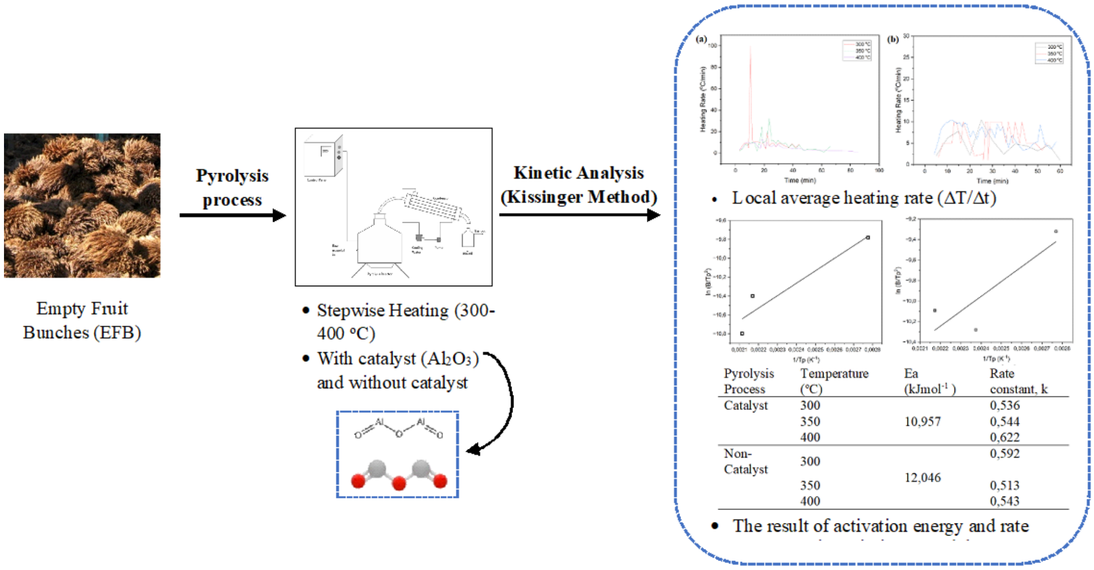
GRAPHICAL ABSTRACT



Pyrolysis Process	Temperature (°C)	Ea (kJmol^{-1})	Rate constant, k
Catalyst	300	10,957	0,536
	350		0,544
	400		0,622
Non-Catalyst	300	12,046	0,592
	350		0,513
	400		0,543

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ABSTRACT

Biomass waste such as Empty Fruit Bunches (EFB), a byproduct of palm oil production in Indonesia, represents a promising renewable energy resource. Pyrolysis, a thermochemical conversion process, transforms this waste into valuable products and energy sources. This study uses the Kissinger kinetic model to investigate how a stepwise heating rate analysis, under both catalytic and non-catalytic conditions, influences the pyrolysis behaviour of EFB. Pyrolysis experiments were conducted in a batch reactor at 300 °C, 350 °C, and 400 °C, with and without adding a catalyst (aluminium white) at a 1:2 catalyst-to-feedstock ratio. Heating rates were calculated at 10 °C intervals, and the activation energy (Ea) was determined using the Kissinger kinetic model. The results indicate that, with the presence of a catalyst, the heating rate increases with temperature, while without a catalyst, the rate tends to decrease. Moreover, the catalyst substantially reduces the activation energy, from 12.046 kJ/mol (non-catalytic) to 10.957 kJ/mol (catalytic), indicating its effectiveness in enhancing thermal decomposition and facilitating pyrolysis with lower energy requirements.

1. INTRODUCTION

Agricultural and forestry activities generate large quantities of residues, which are increasingly recognized as valuable resources for biomass-based energy production. Agricultural wastes such as corn straw, wheat straw, and sugarcane bagasse, once considered disposal problems, are now widely utilized as feedstocks for renewable bioenergy, providing both environmental benefits and new economic opportunities for farmers [1]. Similarly, residues from the forest products industry, such as wood chips, sawdust, and bark, are used in biopower and biofuel production, enhancing the economic sustainability of the logging and timber industries [2].

One underutilized biomass resource is Empty Fruit Bunches (EFB), a solid palm oil industry waste abundantly available in Indonesia. EFB constitutes approximately 20–23% of processed fresh fruit bunches, resulting in millions of tons of waste annually [3]. Due to its high cellulose, hemicellulose, and lignin content, combined with low ash and sulfur levels, EFB holds significant potential as a feedstock for thermochemical and biochemical conversion processes [4, 5]. This potential enables the production of diverse energy carriers, underscoring the important role that agricultural and forestry residues, such as EFB, can play in the bioenergy sector.

As one of the most promising thermochemical conversion pathways for biomass, pyrolysis has attracted significant attention in recent decades as a solution for producing renewable energy and value-added chemicals. Biomass conversion through pyrolysis offers excellent potential for generating various products that can be used as alternative fuels and chemical precursors. Among the operational parameters, the heating rate significantly influences the yield and quality of pyrolysis products. In this complex process, the heating rate plays a crucial role in determining the mechanism of biomass decomposition, the product yields (bio-oil, biochar, and gas), and the physicochemical characteristics of the resulting products [6,7]. Variations in the heating rate can alter the decomposition reaction pathways, ultimately affecting the proportions of liquid, solid, and gas products and the composition of each fraction [8]. At slower heating rates, the mass reduction is minor, resulting in a greater amount of char being produced. The final temperature also influences the extent of mass reduction; higher final temperatures during the pyrolysis process lead to greater mass loss, reducing the quantity of char produced [9].

Furthermore, the efficiency and optimal yield of the pyrolysis process highly depend on the control of operational parameters. The heating rate not only influences heat transfer into the biomass particles but is also intrinsically linked to the kinetics of the thermal decomposition reactions [10]. Variations in heating rates can activate different reaction pathways, thereby altering the activation energy and frequency factors of the pyrolysis reaction stages [11].

Thermal analysis reveals that activation energy is a critical determinant of reactivity and a significant modulator of reaction kinetics. Consequently, the reactivity of biomass is often evaluated based on its activation energy [12]. Prior research has extensively explored the kinetic properties of various biomass sources. For example, Mian et al. (2024) reported a comprehensive kinetic analysis of pine particle pyrolysis, with activation energies ranging from 91.8 to 175.8 kJ/mol, consistent with findings by Zakrzewski (2003) [13,14]. Furthermore, Gao et al. (2017) conducted a comparative analysis of rice straw to elucidate the impact of various additives on activation energy and decomposition kinetics under both oxidative and inert atmospheres, identifying an activation energy of 74 kJ/mol for the biomass [15]. In a study examining the influence of heating rate, Reina et al. (1998) reported activation energies of 130.1, 136.2, and 128.0 kJ/mol across heating rates ranging from 5 to 100 °C/min [16].

Unlike previous studies that focused on isothermal or constant-rate pyrolysis, this work introduces a stepwise heating rate analysis under both catalytic and non-catalytic conditions to investigate the pyrolysis behavior of EFB using the Kissinger kinetic model. The novelty lies in utilizing localized heating rate data to evaluate reaction kinetics, providing a more detailed insight into the dynamic thermal behavior of EFB during pyrolysis.

2. EXPERIMENTAL METHODS

2.1 Material

The feedstock used in this study was empty fruit bunches (EFB) obtained from a palm oil plantation in Lampung Province, Indonesia. The EFB was initially dried and then chopped to a particle size of approximately 1 mm. Subsequently, it was dried in an oven at 105°C for 1 hour to reduce the moisture content. The catalyst was aluminum oxide, used at a ratio of 1:2 relative to the EFB feedstock.

2.2 Method

The pyrolysis process was conducted in a batch reactor equipped with an electric heater, an internal thermocouple, and a temperature controller to maintain the desired temperature [17]. The system also included a condenser set and a collection flask to recover the pyrolysis vapor condensate. The experimental setup is illustrated in Figure 1. Experiments were performed at temperatures of 300°C, 350°C, and 400°C, both with and without the addition of a catalyst. The pyrolysis process employed a stepwise heating method with local heating rate analysis. The reactor was heated incrementally from ambient to the predetermined target temperatures, with temperature readings recorded at each 10°C interval. The time required to reach the subsequent interval was recorded at each temperature increment. These temperature and time data were then used to calculate each interval's local heating rate ($\Delta T/\Delta t$). This approach enabled dynamic analysis of heating rate variations throughout the heating process, avoiding the constant rate assumption and providing a more accurate understanding of the system's actual heating dynamics. The pyrolysis process produced liquid, gas, and biochar products. The yield of each pyrolysis product was determined by calculating the ratio of the mass of the respective product (liquid, gas, or biochar) to the initial mass of the feedstock.

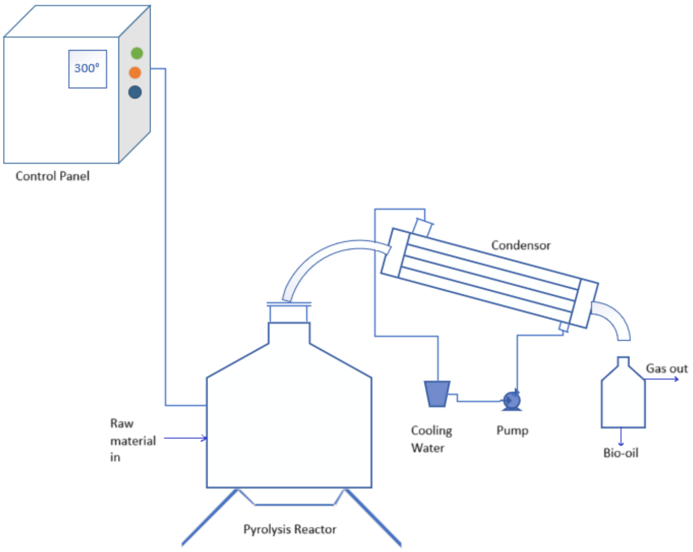

Figure 1. Scheme of EFB pyrolysis process using batch reactor.

2.3 Kinetic Study

The stepwise heating method employed in this study falls into the category of non-isothermal heating. The stepwise heating method used in this study is a form of non-isothermal heating, where the temperature is not held constant but gradually increases over time. This approach enables monitoring changes in material properties or reaction kinetics across a continuously varying temperature range. A typical application of non-isothermal data is the Kissinger method, which is employed to analyze activation energy [18].

Some researchers use a model-based approach to determine the kinetic parameters of pyrolysis [4]. Quantitative thermal decomposition kinetics models are classified as either model-free or model-based. The model-based approach employs the Arrhenius equation to analyze integral experiments at various heating rates. Examples of the model-free approach include the Kissinger-Akahira-Sunose (KAS) and Flynn-Wall-Ozawa (FWO) methods [19]. In this study, the Kissinger equation (Equation (1)) was used to perform kinetic analysis on the acquired data [20].

$$ln\left[\frac{\beta}{{T_p}^2}\right] = ln\left(\frac{A.R}{E}\right) - \frac{E}{RT_p} \tag{1}$$

where T_p (°C) is the peak decomposition temperature, R ($JK^{-1}mol^{-1}$) is the universal gas constant, and β (°$Cmin^{-1}$) is the heating rate. By plotting ln $\left(\frac{\beta}{{T_p}^2}\right)$ againts $\left(\frac{1}{T_p}\right)$, the Ea ($kJmol^{-1}$) were calculated using the values of the slope and intercept, respectively.

The rate constant (k) is defined by the relative rate constant that indicates the speed of a chemical reaction at a specific temperature with a time unit (min^{-1}) because it compares k without knowing the value of A (pre-exponential), making the value of k relative rather than absolute. The equation being approached is the Arrhenius equation, as shown in Eq. (2):

$$k = A.e^{-\left(\frac{Ea}{RT}\right)} \tag{2}$$

where A is the pre-exponential factor or frequency factor (min^{-1}); Eα is the apparent activation energy of the decomposition reaction (kJ mol^{-1}); R is the universal gas constant (8.314 × 103 kJ mol^{-1} K^{-1}); T is the temperature (K).

3. RESULTS AND DISCUSSIONS

3.1 Heating Rate Analysis

The heating rate was determined by calculating the ratio of the operating temperature to the time required to reach the target temperature (°C/min). This method enables localized analysis of heating behavior at each stage of the process. The results comparing calculated and actual heating rates, along with their linear regression correlations (R^2), are presented in Table 1.

TABLE I. Heating rate pyrolysis process EFB with catalyst and without catalyst.

Pyrolysis Process	Temperature (°C)	Calculated Heating Rate (°C/menit)	Actual Heating Rate (°C/menit)	R square
Catalyst	300	5,03	4,88	0,976
	350	6,44	6,85	0,974
	400	6,85	6,84	0,983
Non-Catalyst	300	8,24	8,12	0,993
	350	6,12	6,82	0,964
	400	4,64	4,66	0,927

The calculated heating rate differs from the actual heating rate because the latter is determined using linear regression, which provides more accurate estimates. The coefficient of determination (R^2) represents the proportion of variance in the dependent variable (temperature) explained by the independent variable (time) in the regression model. The R^2 values from the linear regression (Table 1) range from 0.927 to 0.993, indicating a strong correlation between time and temperature increase and confirming the heating consistency across the experiments. Furthermore, the criteria for simple regression testing are reflected in the p-value, which indicates whether the relationship between the independent and dependent variables is statistically significant. A p-value less than 0.05 indicates a significant effect, while a p-value greater than 0.05 indicates no significant effect [21]. All obtained p-values were below 0.05, signifying statistically significant relationships between the independent variable (time) and the dependent variable (temperature) under all conditions.

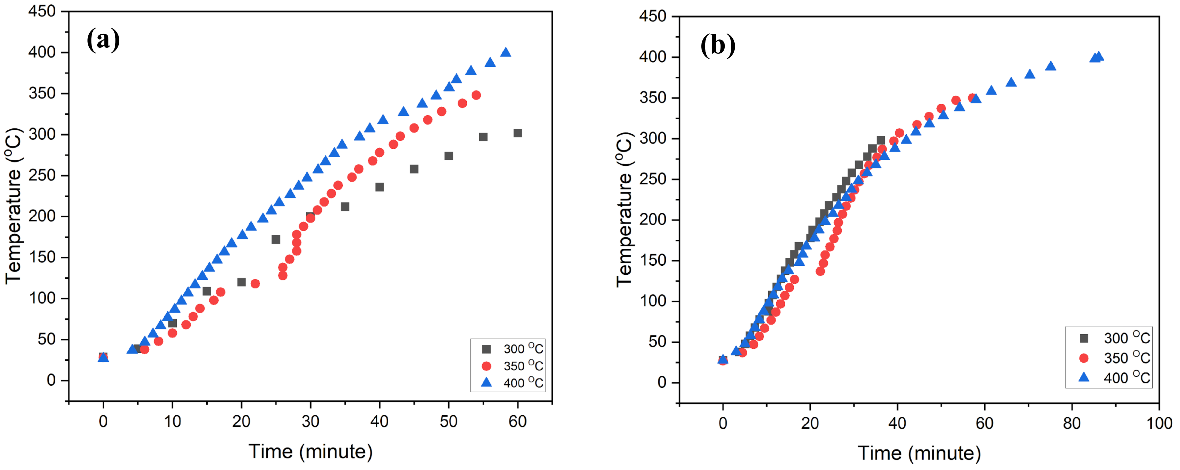

Figure 2. Heating rate profile EFB pyrolysis, (a) with catalyst and (b) without catalyst.

Figure 2 illustrates the heating rate profiles of EFB pyrolysis with (a) and without (b) a catalyst. Under catalytic conditions, the heating rate tends to increase with temperature, whereas in non-catalytic runs, the heating rate decreases as temperature rises. This suggests that the catalyst enhances heat transfer efficiency within the biomass, likely due to improved thermal conductivity and reaction kinetics. The heating profile also indicates that the presence of the catalyst contributes to a more stable and sustained heating rate, particularly at 350 °C and 400 °C. These findings are consistent with Adha's study on the pyrolysis of ulin wood powder, which used heating rates ranging from 10 to 25 °C/min. The study revealed that at lower heating rates, the biomass decomposes more slowly, allowing improved heat transfer into the biomass particles [22]. Conversely, higher heating rates generate more thermal energy, promoting effective heat transfer; however, they also result in increased heat losses between the surrounding environment and the biomass components [23]. Although experimental replicates were not performed, the consistency of the heating rate trends and the strong linear correlation ($R^2 > 0.97$) between temperature and time support the validity of the data obtained. Nevertheless, the lack of reported standard deviations remains a limitation and should be addressed in future studies to enhance statistical robustness.

Overall, the catalyst facilitates heat distribution and appears to influence the reaction environment, which may affect subsequent kinetic behavior. The key observations are:

- The heating rate increases with temperature under catalytic conditions.
- Without a catalyst, the heating rate tends to decrease at higher temperatures.
- Catalysts enhance thermal stability and promote more consistent heating behavior.

These findings suggest a strong relationship between thermal behavior and the presence of a catalyst, highlighting the need for kinetic evaluation to understand better the underlying reaction mechanisms, which are discussed in the following section.

3.2. Kinetic Study Analysis

Kinetic analysis was performed using the Kissinger method, suitable for non-isothermal conditions. In this study, the local heating rate ($\Delta T/\Delta t$), derived from stepwise temperature increments, was used to determine the peak decomposition temperature (Tp), corresponding to the maximum reaction rate at each temperature setting.

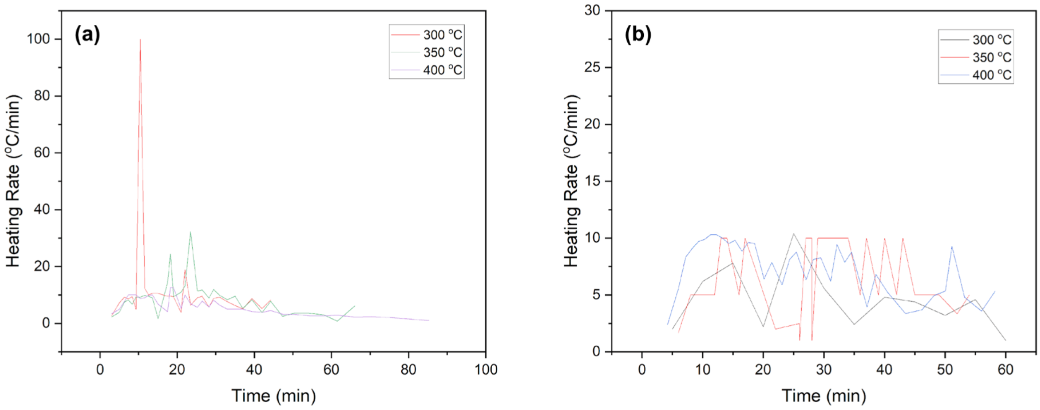

Figure 3. Local average heating rate ($\Delta T/\Delta t$) profile of EFB pyrolysis, (a) without catalyst and (b) with catalyst.

The graph in Figure 3(a) illustrates how the average heating rate changes over time during pyrolysis without a catalyst at 300 °C, 350 °C, and 400 °C. At the beginning of the process, there is a noticeable spike in the heating rate, particularly at 300 °C, indicating a very rapid initial heating of the sample. The temperatures of 350 °C and 400 °C also exhibit an initial peak, although less pronounced than at 300 °C. After this initial peak, the heating rate decreases rapidly for all three temperatures, likely due to the sample reaching the target temperature or a phase change in the material affecting heat transfer. At 400 °C, the heating rate stabilizes at a low value after approximately twenty minutes. Toward the end of the observation period, the heating rates for all three temperatures approach zero, indicating that the temperature change of the material has significantly slowed or has almost stopped.

Compared to Figure 3(a), the peak heating rate at the beginning of the process in Figure 4(b) is lower and broader, particularly at temperatures of 300 °C and 350 °C. The catalyst may influence heat transfer to the material during the early stages. After the initial phase, the heating rate in Figure 3(b) becomes more stable and, at several points (especially at 350 °C and 400 °C), appears higher than under conditions without a catalyst at the same time. This suggests that the catalyst facilitates heat transfer or heat-generating reactions during this period.

From the graphs, the maximum peak decomposition temperature (Tp) was determined by analyzing the local average heating rate ($\Delta T/\Delta t$) profile of the EFB pyrolysis process, both with and without a catalyst. The values, denoted by the symbol (β), are presented in Table 2. Subsequently, the Kissinger model (Eq. 1) was applied for kinetic analysis of the data by plotting ln $\left(\frac{\beta}{T_p{}^2}\right)$ againts $\left(\frac{1}{T_p}\right)$, as shown in Figure 4 for pyrolysis with catalyst (a) and without catalyst (b).

TABLE II. The peak decomposition temperature (Tp) value with local average heating rate (β) of EFB pyrolysis.

Pyrolysis Process	**β (°C/min)**	**Tp (°C)**
Catalyst	10,40	120
	10,00	68
	10,31	87
Non-Catalyst	100	88
	32,6	187
	12,66	148

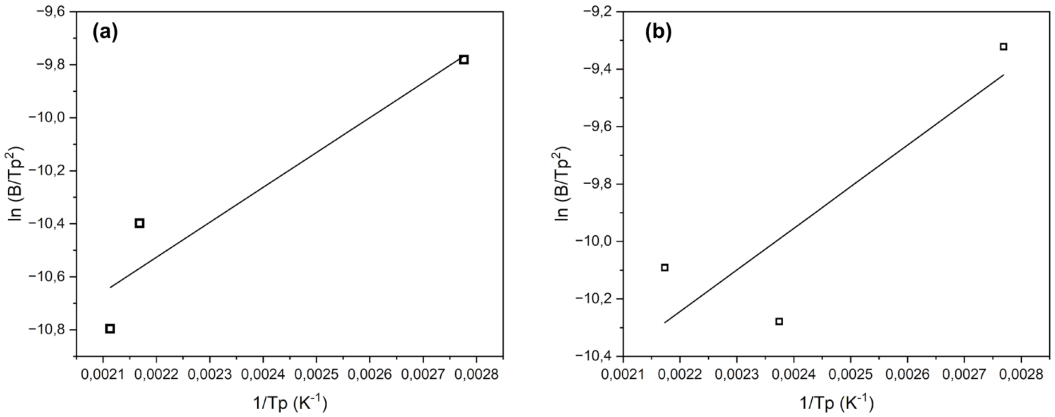

Figure 4. Ea, (a) with catalyst and (b) without catalyst.

The activation energy (Ea, kJ/mol) calculated using the Kissinger kinetic model is presented in Table 3. The activation energy for the process with the catalyst, shown in Figure 3, is 10.957 kJ/mol. A lower activation energy generally indicates that the reaction proceeds more easily in the presence of a catalyst, as less energy is required to reach the transition state. In contrast, the Ea without the catalyst is higher, at 12.046 kJ/mol. This demonstrates that, without a catalyst, more energy is needed to initiate the pyrolysis reaction. The reduction in activation energy is the primary role of the catalyst in accelerating the chemical reaction—in this case, the pyrolysis reaction. Several studies employing the Kissinger model with varying heating rates report different activation energy values depending on whether low [24] or high [18] heating rate categories are used. At low heating rates, the activation energy values tend to be lower, whereas at high heating rates, the obtained activation energy values are significantly higher. Furthermore, using a catalyst during the pyrolysis process reduces the measured activation energy compared to pyrolysis conducted without a catalyst [25].

The Ea values obtained are significantly lower than the typical biomass pyrolysis values reported in the literature, which commonly range from 50 to 200 kJ/mol. Discrepancy may be attributed to several factors:

- Stepwise heating, rather than continuous thermogravimetric analysis (TGA), lowers apparent energy barriers because the system cools slightly during each interval.
- The measured Tp values are based on local peaks in the heating rate, which may not accurately reflect the global maximum decomposition rate.
- The catalytic decomposition likely involves surface-mediated mechanisms, particularly affecting hemicellulose or low-temperature volatiles requiring lower energy input.

Despite these lower values, the trend remains consistent: the presence of the catalyst reduces the activation energy and stabilizes the thermal degradation pathway. This supports the hypothesis that the catalyst facilitates bond breaking and accelerates the reaction. A comprehensive assessment may require further validation using model-fitting or isoconversional methods (e.g., FWO, KAS).

TABLE III. The result of activation energy and rate constant using Kissinger models.

Pyrolysis Process	**Temperature (^{o}C)**	**Ea (kJmol^{-1})**	**Rate constant, k**
Catalyst	300	10,957	0,536
	350		0,544
	400		0,622
Non-Catalyst	300	12,046	0,592
	350		0,513
	400		0,543

The reaction rate constant (k) values with the catalyst vary at each temperature, as shown in Table 3. The reaction rate increases constantly with rising temperature, which aligns with chemical kinetics theory, which states that reaction rates generally increase at higher temperatures. In contrast, reaction rate constants without a catalyst exhibit less consistent changes with temperature than those with a catalyst. A comparison of k values reveals that at 300 °C, the reaction rate without a catalyst is slightly higher; however, at 400 °C, the reaction rate with the catalyst surpasses that without it. At 350 °C, the reaction rates are nearly identical. These observations suggest that the catalyst's effectiveness in enhancing the reaction rate may depend on the operating temperature. The resulting rate constant (k) is low at a rapid heating rate. This may be because heat transfer to the biomass occurs quickly, leaving insufficient time for the heat to break the molecular bonds in the biomass before it reaches a high temperature [26]. The key Interpretations:

- Ea values are lower than those reported in the literature, possibly due to differences in experimental design and catalyst effects.
- A catalyst reduces the activation energy and increases the reaction rate, especially at higher temperatures.
- The Kissinger model remains applicable in stepwise pyrolysis, although its accuracy is limited due to non-TGA experimental conditions.

These results underscore the importance of controlling the heating rate and the presence of catalysts in tuning the kinetics of EFB pyrolysis, indicating potential improvements in energy efficiency and process design.

4. CONCLUSIONS

This study demonstrated that applying a stepwise heating approach during the pyrolysis of Empty Fruit Bunches (EFB) enables detailed analysis of localized heating behavior and its effect on kinetic parameters. The presence of a catalyst enhanced the stability of the heating rate. It significantly reduced the activation energy from 12.046 kJ/mol (non-catalytic) to 10.957 kJ/mol (catalytic), improving thermal efficiency and reaction performance. Despite the relatively low activation energy values obtained—likely due to localized peak temperatures and non-TGA conditions—the overall trends remain consistent with the catalytic facilitation of decomposition. These findings underscore the importance of controlling the heating rate and catalyst application to optimize biomass pyrolysis processes. Future work should incorporate experimental replicates and broader kinetic modeling approaches (e.g., model-free methods) to enhance statistical validity and deepen the understanding of reaction mechanisms. Overall, this research offers a novel perspective on kinetic evaluation using stepwise heating data, which may serve as a valuable reference for further developing efficient pyrolysis systems for lignocellulosic biomass.

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