

THERMOGRAVIMETRIC ANALYSIS ON COMBUSTION BEHAVIOR OF MARINE MICROALGAE *Spirulina platensis* INDUCED BY MgCO₃ AND Al₂O₃ ADDITIVES

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(Received: August 2019 / Revised: October 2019 / Accepted: October 2019)

ABSTRACT

The impact of MgCO₃ and Al₂O₃ additives on the thermal behavior of *Spirulina platensis* (SP) biomass during combustion in a thermal analyzer was evaluated to understand their catalytic effect in the decomposition process. The samples were pure SP and a mixture of SP and additives at mass fractions of 3, 6, and 9 (wt,%). Each sample of around 8.5 mg was mounted in a thermobalance and subjected to a furnace on a heating program of 10°C/min. The 100 ml/min air atmosphere was kept continuously flowing during the combustion process from 30–1200°C. The thermal behavior of the sample was then characterized from the thermogravimetric (TG) and derivative thermogravimetric (DTG) curves, those were recorded by a computer during the experiment. The Horowitz–Metzger method was used to evaluate the impact of additives on the kinetic parameters of the samples. The results indicated that the presence of additives shifted the main decomposition stage toward a lesser temperature. The rate of mass loss (ML) in the main decomposition zone decreased in the 1st peak and increased in the 2nd, in accordance with the increase in the fraction of additives. This indicates that additives play different roles during the decomposition process. The mass mean activation energy (E_m) increased at the additive fraction of 3% for both MgCO₃ and Al₂O₃, as well as at 6% MgCO₃ compared to combustion with no additives. Conversely, the presence of greater additives promoted a shift in E_m toward smaller values. These results confirm that both additives significantly influenced the thermal behavior and kinetics of the SP combustion.

Keywords: Additives; Combustion; Microalgae; *Spirulina platensis*; Thermogravimetry

1. INTRODUCTION

Global energy demand is estimated to show a continuous increase of around a third by 2040 (*BP Energy Outlook*, 2018); conversely, there is a rapid depletion of fossil-based reserves (Sukarni et al., 2017). This situation has prompted the exploration of alternative energy sources, and biomass has received increasing interest (Purwanto et al., 2015; Ghatak & Mahanta, 2017; Wahyudi et al., 2018). Among the various biomass-based fuels, aquatic microorganisms have attracted attention due to their extensive spread across the globe, high efficiency during the photosynthetic process, ability to rapidly multiply their biomass, non-dependence on land, thus creating no interference

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Permalink/DOI: <https://dx.doi.org/10.14716/ijtech.v10i6.3611>

with food production, automatic reproduction process, high capacity for transforming CO₂ during the growth phase to become biomass, and their low sulfur and nitrogen content (Sukarni et al., 2014; Gai et al., 2015).

Various techniques for converting microalgae to energy have previously been performed, including biochemical conversions such as digestion (Passos et al., 2014) and fermentation (Hossain et al., 2015), as well as thermochemical conversions such as pyrolysis, gasification, liquefaction, and combustion (Sanchez-Silva et al., 2013; Sukarni et al., 2015, 2018a, 2018b; Viju et al., 2018). Combustion is the most important choice owing to the ease of transferring heat to energy using currently available technological devices. The combustion method accounts for over 97% of the world's bioenergy production (Demirbas, 2004).

Several studies on microalgae direct combustion have been performed previously. Combustion of *Nannochloropsis oculata* (Sukarni et al., 2015), *Nannochloropsis gaditana* (Sanchez-Silva et al., 2013), *Chlorella vulgaris* (Chen et al., 2011), and *Scenedesmus almeriensis* (López et al., 2013) revealed the diverse range of thermal behavior among the species. These differences in their thermal behavior were strongly thought to derive from variances in their organic matter and the mineral compounds that made up the compartment of each species. Differences in the major components of algal biomass, i.e., protein, carbohydrate, and lipid, affect thermal behavior because each component has a different thermal degradation. From the different literature, it was also understood that the mineral content of algal biomass highly determined its ash content (Chen et al., 2014) and that this would significantly affect its decomposition behavior due to its catalytic effect during thermal degradation (Gai et al., 2015). However, the catalytic effect of each mineral compound on microalgal combustion has yet to be reported by scholars.

This paper presents the catalytic effect of MgCO₃ and Al₂O₃ additives on the thermal behavior of *Spirulina platensis* (SP) biomass during oxidative-thermal degradation under thermal analyzer equipment. The characteristic temperatures were determined according to the thermogravimetric (TG) and derivative thermogravimetric (DTG) curves. The Horowitz–Metzger fitting method was applied to evaluate the kinetic parameters in terms of activation energies, frequency factors, and reaction orders.

2. METHODS

2.1. Materials Preparation

The method for preparing SP has been presented in previous literature (Sukarni et al., 2018a). The sample was obtained from BBPBAP, Jepara, Indonesia. The water content was removed from the biomass using an oven at a temperature setting of 100°C for a period of 24 h. The dry biomass chips were then crushed to a powder and bottled and stored in a dry condition.

Each of the dry, pulverized MgCO₃ and Al₂O₃ additives were then mixed on the SP sample using the mechanical mixing technique. For each SP sample weighing 10 mg, the additives were added in the amounts of 0.3, 0.6, and 0.9 mg, respectively. Next, they were decanted into small glass bottles and shaken to ensure homogeneous mixing. Following these methods ensured that the overall samples of SP, SP/MgCO₃ (with mass ratios of 3, 6, and 9%), and SP/Al₂O₃ (with mass ratios of 3, 6, and 9%) were ready for testing.

2.2. Thermogravimetric Experiment

A thermogravimetric analyzer (model: Mettler Toledo TG/DSC1) was used as the thermal testing apparatus. A sample of around 8.5 mg was placed uniformly in the crucible and then subjected to the furnace. The temperature was adjusted linearly, rising from approximately 30 to 1200°C on a heating program of 10°C/min. The air atmosphere was kept continuously flowing during the combustion process at a rate of 100 ml/min. The mass loss (ML) of the sample during the temperature escalation was automatically recorded by a computer and resulted in the TG curve.

The rate of ML, which was represented as the DTG curve, was obtained by differentiating the TG data to the time or temperature.

2.3. Characteristic Parameters

The main issue, that of the combustion of solid material, determined the parameters to be set. These included the onset temperature (T_o) of reaction, which refers to the temperature at which volatile release and weight loss begin, the burnout temperature (T_b), which relates to temperature as mass stabilization, and the maximum rate temperature (T_{max}), which refers to the temperature at which the maximum ML rate (DTG_{max}) is reached, as well as the ML at a certain zone of the mass event of interest (Muthuraman et al., 2010).

To further understand the effect of additives on the thermal conversion of the biomass during combustion, the parameter ΔW was defined as follows (Liu et al., 2004; Xu et al., 2014):

$$\Delta W = W_c - W_w \quad (1)$$

where ΔW represents the incremental ML after the addition of additives into the SP sample; W_c is the ML of the blended SP additives, and W_w is the mass of the pure SP sample at a corresponding temperature.

2.4. Kinetic Method

The reaction rate for the solid fuel that was experiencing thermal breakdown under the isothermal condition can be expressed as the following Arrhenius equation (Sukarni et al., 2018a):

$$\frac{d\alpha}{dt} = A \exp\left(\frac{-E}{RT}\right) f(\alpha) \quad (2)$$

where A represents the frequency factor (min^{-1}), E denotes the activation energy (kJ/mol), R indicates the universal gas constant (8.314 J/mol K), T is the absolute reaction temperature (K), $f(\alpha)$ expresses the supposed model of the reaction mechanism, and α is the ML fraction expressed as $\alpha = (m_o - m_t)/(m_o - m_\infty)$, where m_o , m_t , and m_∞ denote the onset, present, and residual at the event of interest for the microalgae sample.

This study was conducted under the non-isothermal setting; therefore, the appropriate kinetic equation may be obtained by substituting Equation 2 with $dt = dT/\beta$, which results in an equation as follows:

$$\frac{d\alpha}{dT} = \frac{A}{\beta} \exp\left(\frac{-E}{RT}\right) f(\alpha) \quad (3)$$

where $\beta = dT/dt$ is the heating rate.

Choosing the n th order reaction model, $f(\alpha) = (1 - \alpha)^n$ and the rearrangement of Equation 3 generates:

$$\frac{d\alpha}{(1-\alpha)^n} = \frac{A}{\beta} \exp\left(\frac{-E}{RT}\right) dT \quad (4)$$

The linearization of Equation 4 was performed using the Horowitz–Metzger method and produced the following equation (Açikalin, 2012):

$$\ln g(\alpha) = \left(\ln \left(\frac{ART_{max}^2}{\beta E} \right) - \frac{E}{RT_{max}} \right) + \frac{E\varphi}{RT_{max}^2} \quad (5)$$

where:

$$\text{if } n = 1 \text{ then } g(\alpha) = -\ln(1 - \alpha)$$

$$\text{if } n \neq 1 \text{ then } g(\alpha) = (1 - (1 - \alpha)^{1-n})/(1 - n)$$

$$\varphi = T - T_{max}$$

By selecting the most proper n values, the activation energies and frequency factors could subsequently be calculated from the slope $\frac{E}{RT_{max}^2}$ and intercept $\left(\ln\left(\frac{ART_{max}^2}{\beta E}\right) - \frac{E}{RT_{max}}\right)$, in that order.

In order to deeply understand how the presence of additives affects the kinetic parameters during SP combustion, the mass activation energy E_m was introduced to thoroughly evaluate the sample reactivity (Shao et al., 2010),

$$E_m = F \times E \quad (6)$$

in which F refers to the amount of ML at the corresponding stage which being the concern, and E is the activation energy at a related stage.

3. RESULTS AND DISCUSSION

3.1. Effects of Additives on Thermal Behavior

The thermal behavior of SP and the various mixtures containing additives are presented in the TG-DTG curves of Figure 1. Stage I, which occurred at the temperature range of 30–165°C, relates to the elimination of moisture and light volatile evaporation, as discussed in various studies (Shuping et al., 2010; Xu et al., 2013). Stage II, which began at around 165–500°C, was the main combustion zone associated with the intense thermal decomposition and burning of organic components. Stage III was characterized by a distinctive basin with peaks at about 970°C that correlated with degradation of the volatile metal and carbonate compounds (López-González et al., 2014; Sukarni et al., 2015). Because combustion was principally aimed at the conversion of microalgal biomass to energy and took place in Stage II, this paper discusses the influence of additive primarily on the main combustion zone only. The characteristic temperature parameters in the main combustion zone in accordance with the TG-DTG curves are given in Table 1.

It can be seen from Table 1 that the addition of additives promoted the decreasing of onset temperature of degradation (T_o) and that this reduction was more in line with the increase in the additive mass ratio. This indicates that the presence of additives may have encouraged the initial degradation of the microalgae samples. The burnout temperature (T_b) also decreased in line with the increase in the additive mass ratio. These results show that the addition of additives could reduce the time required for combustion.

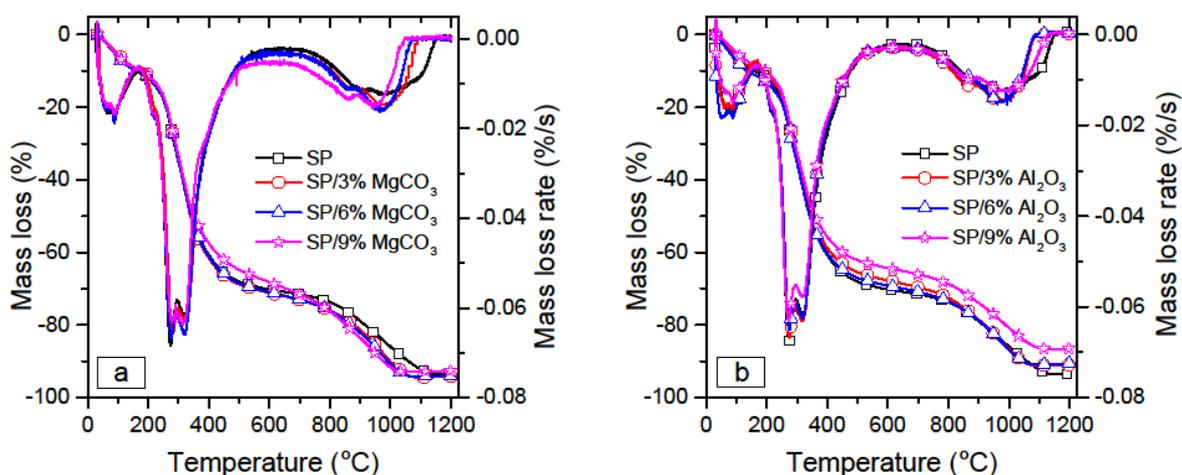


Figure 1 TG-DTG curves of SP and the mixtures of: (a) SP and $MgCO_3$; (b) SP and Al_2O_3
Table 1 Characteristic parameters of SP and its mixtures

Samples	T_o (°C)	T_b (°C)	T_{max-1} (°C)	DTG_{max-1} (%/s)	T_{max-2} (°C)	DTG_{max-2} (%/s)
SP	168.78	504.09	272.85	-0.06849	318.51	-0.06193
SP/3% $MgCO_3$	166.00	500.08	280.00	-0.06583	322.00	-0.06281
SP/6% $MgCO_3$	164.65	497.92	275.84	-0.06685	322.49	-0.06607
SP/9% $MgCO_3$	162.35	494.42	278.42	-0.06315	312.05	-0.06367
SP/3% Al_2O_3	165.85	501.42	275.00	-0.06687	318.11	-0.06310
SP/6% Al_2O_3	164.92	499.56	273.24	-0.06504	316.72	-0.06262
SP/9% Al_2O_3	162.44	497.40	272.04	-0.06303	315.32	-0.05764

T_{max-1} and T_{max-2} : temperature of the maximum rate at the 1st and 2nd peak, respectively

DTG_{max-1} and DTG_{max-2} : maximum mass loss rate at the 1st and 2nd peak, respectively

Figure 2 contains a magnification of the TG-DTG curves to indicate the temperature of the maximum rate (T_{max}) and the maximum ML rate (DTG_{max}). Two pronounced humps can be observed; the 1st peak corresponds to the degradation of carbohydrates and protein and the 2nd relates to lipid decomposition (López-González et al., 2014). T_{max} corresponds to the microalgae structure (Liu et al., 2004). As specified in both Table 1 and Figure 2, the presence of $MgCO_3$ affects the increase in the temperature of the maximum rate at the 1st peak (T_{max-1}), which indicates that $MgCO_3$ might inhibit the thermal degradation of carbohydrates and protein. The addition of Al_2O_3 has a similar impact on T_{max-1} , but with a lower temperature difference compared to $MgCO_3$. This phenomenon also confirmed that Al_2O_3 was strongly though prohibited the carbohydrates and protein thermal decomposition, but with the weak impact. From the perspective of DTG_{max} , it can clearly be seen that the presence of both $MgCO_3$ and Al_2O_3 reduced the maximum ML rate at the 1st peak (DTG_{max-1}). This means that the rate of thermal degradation of carbohydrates and protein was hindered by the presence of both additives.

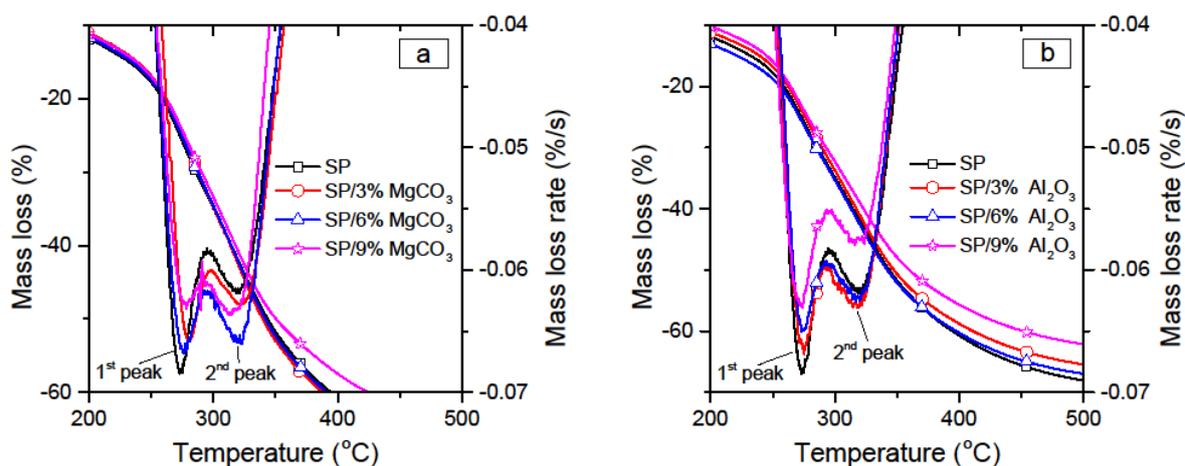


Figure 2 Magnification of TG-DTG curves to indicate the temperature of the maximum rate (T_{max}) and the maximum mass loss rate (DTG_{max}): (a) SP and $MgCO_3$; (b) SP and Al_2O_3

Figure 2 and Table 1 also indicate that T_{max-2} increased in line with the increasing $MgCO_3$ mass ratio, except for at the 9% ratio. In contrast, the addition of Al_2O_3 additive influenced the reduction of T_{max-2} , except at the mass ratio of 3%. This demonstrates that the presence of 9% $MgCO_3$, 6% and 9% Al_2O_3 promotes lipid degradation, and it is mainly related to the thermal cleavage of hydrocarbon chains of fatty acids (López-González et al., 2014). It can also be clearly seen that at the 2nd peak, the higher level of $MgCO_3$ in SP results in a higher DTG_{max-2} . This confirms that the thermal degradation of lipid became severe in line with the increasing $MgCO_3$

mass ratio. Meanwhile, Al_2O_3 induced a massive increase in $\text{DTG}_{\text{max}-2}$ except for at the mass ratio of 9%, which actually reduced $\text{DTG}_{\text{max}-2}$. This explains that the addition of 3% and 6% Al_2O_3 has a positive impact on the rate of lipid decomposition, whereas at 9% the opposite was true.

Figure 3 represents the incremental ML after the addition of additives into the SP sample with various ratios of additives in the main combustion zone. It illustrates that ΔW of 3% MgCO_3 started to increase from 252°C onward and then began falling at 336°C. The ΔW of 6% MgCO_3 remained close to 0 at temperatures below 202°C and then increased in line with the escalation in temperature between 202 and 336°C before decreasing as the temperature continued to rise. This confirms that increasing MgCO_3 from 3% to 6% promoted the catalytic effectiveness toward the lower temperature and that both these ratios of MgCO_3 have had a decreasing effect on the thermal conversion of microalgae at 336°C. Whereas the 9% MgCO_3 had an active effect within the temperature range of 247–341°C.

For Al_2O_3 , it was clearly observed that the mass ratio of 6% made a greater contribution to the microalgal conversion than that of the 3% and 9% mass ratios. It was detected that 3% Al_2O_3 might enhance the conversion of biomass samples in the temperature range from 252 to 353°C, whereas 9% Al_2O_3 inhibited the conversion process. Overall, it can be understood from this discussion that the addition of both MgCO_3 and Al_2O_3 additives up to a mass ratio of 6% provides further evidence with respect to the conversion of microalgal biomass.

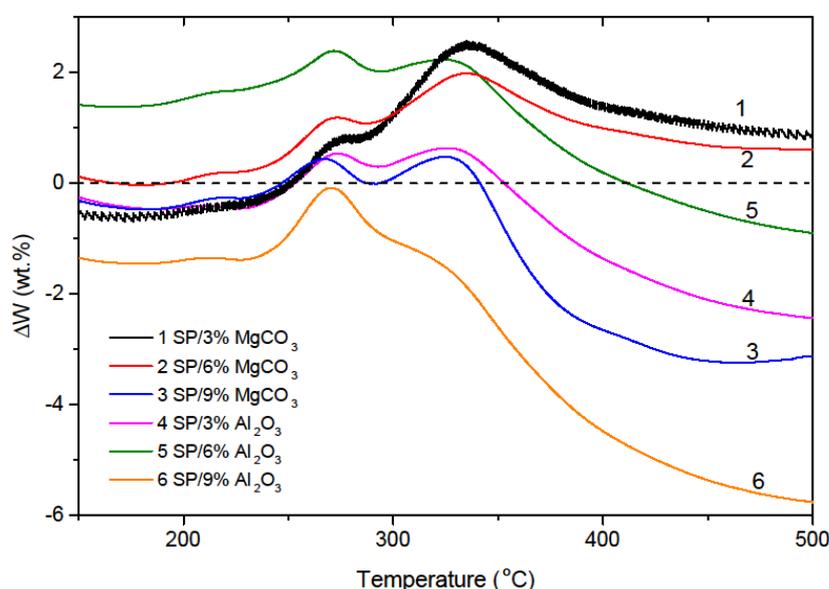


Figure 3 Incremental mass loss after the addition of additives into the SP sample in the main combustion zone

3.2. Effects of Additives on Kinetic Parameters

In accordance with the characteristic temperature parameters as listed in Table 1, the kinetic evaluation was performed using Equation 5. Since biomass combustion is a complicated chemical reaction involving decomposition and the oxidation of various elements at different temperatures, the kinetic evaluation assumed that thermal reaction took place separately within a certain temperature zone as a single-step reaction of the n th order (Sukarni et al., 2018a).

Using the method proposed by Açıkalın (2012), the most proper n value could be obtained from the best linear plotting of $\ln g(\alpha)$ versus φ at the conversion range (α) of 0.05–0.95. This plotting resulted in the coefficient correlation R^2 . Subsequently, $R^2 - n$ curves were drawn as presented

in Figure 4a, and their peaks denoted the highest R^2 values, in which each highest R^2 value was associated with the most proper n . These most proper n values were then used to create final plots of $\ln g(\alpha)$ versus φ and the results are shown in Figure 4b. The results of the kinetic evaluation in terms of activation energies and the frequency factors that were calculated from the respective slopes and intercepts are tabulated in Table 2.

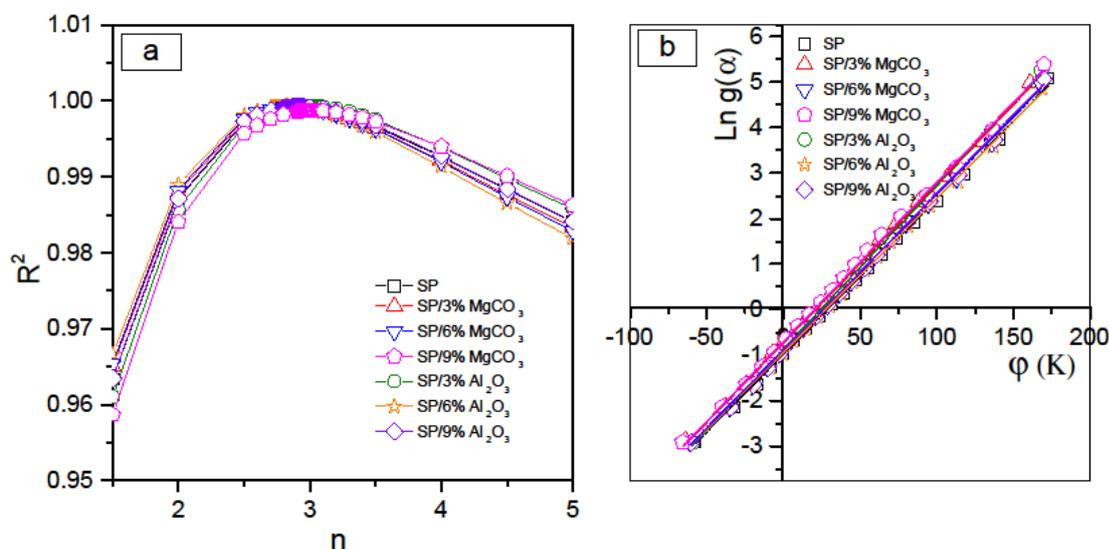


Figure 4 The Horowitz–Metzger parameters: (a) The $R^2 - n$ curve; and (b) Linear regression of the plot $\ln g(\alpha)$ versus φ

Table 2 The kinetic parameters of SP and its mixtures

Sample	Temperature range (°C)	n	A (1/min)	R^2	E (kJ/mol)	F (wt,%)	E_m (kJ/mol)
SP	168.78-504.09	2.91	1.14E+10	0.999	84.61	57.88	48.97
SP/3% MgCO_3	166.00-500.08	2.87	1.57E+10	0.999	88.75	59.29	52.62
SP/6% MgCO_3	164.65-497.92	2.86	1.28E+10	0.999	85.61	58.46	50.05
SP/9% MgCO_3	162.35-494.42	3.00	1.60E+10	0.998	88.08	55.21	48.63
SP/3% Al_2O_3	165.85-501.42	2.98	1.36E+10	0.999	89.44	55.90	49.99
SP/6% Al_2O_3	164.92-499.56	2.82	1.17E+10	0.999	83.79	55.64	46.62
SP/9% Al_2O_3	162.44-497.40	2.91	1.24E+10	0.999	85.15	53.63	45.67

Table 2 indicates that in the main combustion zone, the addition of additives could increase the ML amount by 1.41 and 0.58 (wt,%) for the 3 and 6% MgCO_3 , respectively. The activation energies increased by up to 4.83 kJ/mol with the addition of additives. A reaction with higher activation energy indicates that a higher temperature or longer reaction time is needed (Liu et al., 2004) and vice versa. These results show that the presence of additives might increase the whole reaction time during the SP combustion. A similar result was reported by Xu et al. (2014) for the catalytic pyrolysis of *Chlorella vulgaris*. Table 2 also shows that activation energy varies in line with the different mass ratios and types of additives. These results confirm that the different materials and various mass ratios have different catalytic effects. It is also observed from Table 2 that mass activation energy E_m increased with the increasing ML amount for 3% and 6% MgCO_3 , E_m increased alongside the decreasing ML amount for 3% Al_2O_3 , and E_m decreased in conjunction with the decreasing ML amount for 9% MgCO_3 as well as 6% and 9% Al_2O_3 . These

results show that the catalytic effectiveness of additives was encountered for 3 and 6% MgCO₃. The addition of additives also led to an increase in the frequency factors (A). These results were well aligned with those reported in the previous literature, which showed that catalytic pyrolysis using alkali and alkaline earth metal compounds in the temperature range of 150–430°C induced an increase in frequency factors (Xu et al., 2014). Due to the strong correlation between the kinetic parameters and the chemical structure of the materials, the change in activation energies and frequency factors demonstrates how the compartment structure of SP might be rearranged due to the presence of additives. The high correlation coefficient (R^2) characterizes the great degree of matching between the experimental data and fitting results.

4. CONCLUSION

The catalytic effects of MgCO₃ and Al₂O₃ in the decomposition process of *Spirulina platensis* during combustion have been studied using a thermal analyzer on a heating program of 10°C/min. In the main decomposition and combustion zone, it was found that the presence of MgCO₃ and Al₂O₃ inhibited the thermal degradation of carbohydrates and protein, and slowed down their rate of thermal degradation. Increasing the mass ratio of MgCO₃ and Al₂O₃ up to 6% induced severe thermal degradation of lipids. The addition of MgCO₃ and Al₂O₃ up to a 6% mass ratio had a positive impact on microalgal thermal conversion mainly at temperatures above 200 °C and below 340°C, whereas the addition of 9% of both additives was not recommended as they made little contribution to enhancing the thermal conversion process. The presence of additives led to an increase in activation energies of up to 4.83 kJ/mol, thus indicating that the presence of additives might increase the entire reaction time. Similarly, the additives influenced the increasing frequency factors. The change in both kinetic parameters (activation energies and frequency factors) confirmed that the thermal conversion dynamics of the material had also been changed due to the presence of additives.

5. ACKNOWLEDGEMENT

This research is supported by the Directorate of Research and Community Services, Ministry of Research, Technology, and Higher Education, Government of Indonesia under the scheme of “*Penelitian Dasar Unggulan Perguruan Tinggi*” 2019, with grant number 19.3.82/UN32.14.1/LT/2019.

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