VOLUME 13 No. 2, 22 Juni 2024 Halaman 128-139

KINETIC AND COMBUSTION CHARACTERISTICS OF OIL PALM EMPTY FRUIT BUNCH BIOCHAR USING THERMOGRAVIMETRIC ANALYSIS

KINETIKA DAN KARAKTERISTIK PEMBAKARAN BIOCHAR TANDAN KOSONG KELAPA SAWIT DENGAN ANALISIS TERMOGRAVIMETRI

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Submitted: 2023-09-30; Revised: 2024-03-19; Accepted:2024-03-21

ABSTRAK

Penggunaan energi terbarukan merupakan fenomena mitigasi yang berdampak besar pada sektor tenaga listrik, dengan biomassa sebagai salah satu sumber yang secara langsung menggantikan penggunaan batu bara dalam berbagai aplikasi. Hal ini mengarah pada penggunaan biomassa yang berpotensi sebagai bahan berkarbon, yaitu Tandan Kosong Kelapa Sawit (TKKS). Untuk meningkatkan karakteristik TKKS, maka TKKS dapat dikonversi menjadi produk berbasis karbon melalui proses termokimia, seperti karbonisasi hidrotermal (HTC). Oleh karena itu, penelitian ini bertujuan untuk membandingkan karakteristik TKKS mentah dan biochar TKKS dengan menggunakan metode TGA. Laju pemanasan yang digunakan pada riset ini adalah 10 – 30°C/menit dengan interval 5 °C/menit. Pengaruh laju pemanasan terhadap parameter kinetik, serta karakteristik termal (DTG, TGA) dan pembakaran (T.ignition, T. Burn out) juga ditentukan. Pada penelitian ini, proses HTC dilakukan pada suhu 210°C dan 230°C. Hasil penelitian menunjukkan bahwa TKKS biochar memiliki temperatur penyalaan, temperatur pembakaran dan energi aktivasi yang lebih tinggi dibandingkan dengan TKKS mentah. Temperatur penyalaan untuk TKKS pada suhu 210°C dan 230°C adalah 297°C dan 298°C; temperatur pembakaran pada TKKS pada suhu 210°C dan 230^{0}C adalah 407°C dan 450°C; serta energi aktivasi pada TKKS pada suhu 210°C dan 230°C adalah 58,84 kJ/mol dan 62,16 kJ/mol. Selain karakteristik biomassa, laju pemanasan juga mempengaruhi pembakaran. Hal ini membuktikan bahwa peningkatan laju pemanasan menyebabkan temperatur penyalaan dan pembakaran yang lebih tinggi serta penurunan energi aktivasi. Hasil penelitian juga menunjukkan bahwa perbedaan laju pemanasan mempengaruhi temperatur puncak pada DTG.

Keywords: *Analisis termogravimetri; Energi aktivasi; Tandan kosong kelapa sawit; Laju pemanasan; Pembakaran.*

ABSTRACT

The usage of renewable energy is a mitigation phenomenon majorly impacting the power sectors, with biomass being one of the sources directly replacing coal in various applications. This leads to the portrayal of biomass having the potential to be a carbonaceous material, namely the Empty Fruit Bunch (EFB) of oil palm. To increase the characteristics of EFB, it can be converted into carbon-based products through thermochemical processes, such as hydrothermal carbonization. Therefore, this study aimed to compare the characteristics of feedstock and biochar EFB using the TGA method. The heating rate used in this study is 10 – 30°C/min at five °C/min intervals. The effect of heating rate on kinetic parameters and thermal (DTG, TGA) and combustion (T ignition, T burn

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out) characteristics was also determined. This study carried out the HTC process at temperatures of 210°C and 230°C. The results showed that biochar EFB had a higher ignition, burnout temperature, and activation energy than raw EFB. Ignition temperatures for EFB-HT210°C and EFB-HT230°C were 297°C and 298°C; burnout temperatures for EFB-HT210°C, EFB-HT230°C were 407°C and 450° C; and the activation energy for EFB-HT°210C, EFB-HT°230C were 58.84 kJ/mol and 62.16 kJ/mol. Besides the characteristics of biomass, the heating rate also affects combustion. This proved that increased heating rate caused higher ignition and burnout temperature and decreased activation energy. The results also indicated that the difference in heating rate influenced the peak temperature in DTG.

Keywords: *Thermogravimetric analysis; Activation energy; Empty fruit bunch; Heating rate; Combustion.*

INTRODUCTION

The issue of climate change is presently becoming a significant concern due to its being projected to have negative economic, community, and ecosystem impacts [1], [2]. This leads to the performance of various intensive mitigation actions in several sectors. In this context, renewable energy is considered a mitigation phenomenon that majorly impacts power sectors, with biomass being a source that partially substitutes coal in various applications [3]. This source is also widely used because of its abundant availability, low cost, and environmentally friendly characteristics.

Furthermore, Indonesia is the largest oil palm-producing country in the world, as observed in 2022, with a production of 45.5 million tons. This explains that the oil palm industry produces a large amount of solid waste consisting of 23% empty fruit bunches (EFB), 13% fiber, and 6.5% kernels [4]. Approximately 10.8 million tons of EFB are produced from this context with usage potential. EFB is also capable of being a carbonaceous material with a higher implementation value, accompanied by its conversion into carbon-based products through thermochemical processes, such as hydrothermal carbonization (HTC). This HTC process is widely used to improve the quality of EFB, including increasing the carbon content as well as decreasing the ash, potassium, and chlorine content [5], [6], [7].

Thermogravimetric Analysis (TGA) is an analytical method used to assess the weight loss of a sample at an increasing temperature. This method is commonly implemented to determine the decomposition patterns of carbon-based materials when heated while evaluating the kinetics and reaction parameters of energetic elements [8]. In this case, TGA is used to measure and compare solid fuels' thermal characteristics, combustion characteristics, characteristics, and kinetic parameters include, including biomass.

Several studies related to the use of TGA were also conducted to analyze combustion characteristics of biomass [9], [10]. From this context, a comparison of the decomposition characteristics and kinetic parameters of raw EFB and EFB from hydrothermal and washing processes was conducted by Patcharaporn et al. [11]. This showed that HTC and water-washing processes significantly affected the thermal characteristics parameters of EFB, leading to increased average activation energy and ignition and burnout temperatures. The heating rate is also considered an essential factor in the combustion process, with Hamzah et al. observing similar changes in the TG-DTG profile and thermal level [12]. Other reports subsequently proved that heating rate influenced combustion characteristics of the fuel [13], [14], [15].

Therefore, this study aims to compare the raw EFB and biochar EFB characteristics using the TGA method. It also aims to determine the effect of heating rate on kinetic parameters and thermal (DTG, TGA) and combustion (Ignition and Burnout temperature) characteristics. In this study, the HTC process is carried out at 210°C and 230°C with a ratio of EFB and water of 1:10.

METHOD

Material and Experimental Set Up

In this research, the raw material used was oil palm empty fruit bunch (EFB) from Kalimantan, Indonesia. These EFB samples were used after performing the pretreatment process in size reduction, with the average materials observed between 5-9 cm. The proximate and ultimate analysis of EFB samples before processing can be seen in Table 1. The EFB samples were subsequently processed using the hydrothermal carbonization process according to the experimental scheme in Figure 1.

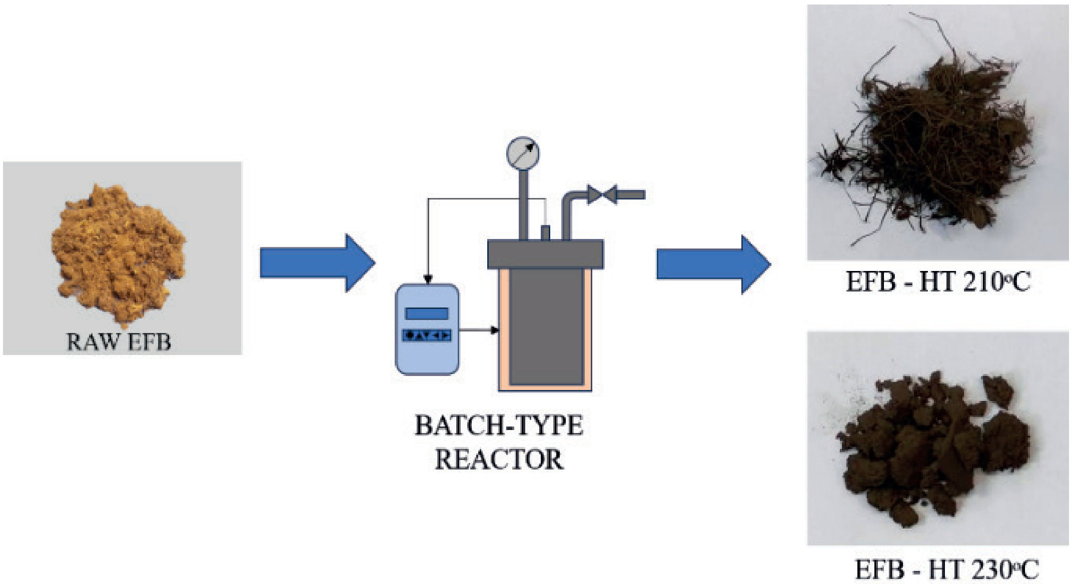


Figure 1.
Experimental Scheme of Hydrothermal Carbonization Process of EFB
Source: Researchers (2023)

The Hydrothermal Carbonization Process of EFB was carried out using a batch-type reactor with a capacity of 3 L and controlled temperature. In this process, the implemented solid-liquid ratio was 1:10 (weight basis), with the solid and liquid materials being 200 grams of EFB samples and 2000 grams of distillate water, respectively.

The operating conditions in the batch reactor were also 210°C and 230°C, with a heating rate of 4°C/min maintained for 60 min. A 12-hour cooling and filtration process accompanied this operation. The residue or solid product was oven-dried at 105°C for 24h and weighed to a constant mass. The dried solids or biochar produced were subsequently stored in sealed bags for characterization analysis.

The Characterization of EFB and Biochar EFB

Raw EFB and biochar EFB samples were characterized using proximate and ultimate analysis. Proximate analysis was used to determine the water content, volatile matter content, ash, and fixed carbon content, with the implemented standard methods being ASTM D3173, ASTM D3174, and ASTM D3175. Fixed carbon content was also determined from the MC, ash, and VM sum. Meanwhile, the ultimate analysis is used to determine the elemental content of carbon (C), hydrogen (H), oxygen (O), nitrogen (N), and sulfur (S). The implemented standard methods were ASTM D5373 for carbon, hydrogen, and nitrogen analysis and ASTM D4239 for sulfur analysis. Oxygen content was also determined by subtracting 100% from C, H, N, and S.

Thermogravimetric Analysis

Thermogravimetric analysis was carried out through a Shimadzu DTG 60-H analyzer, providing the mass loss curve against time (TG curve). The 14-15 mg sample with particle size <0.25 mm (passing 60 mesh) was placed on a platinum pan. The sample will be heated with controlled temperature changes from 35 °C to105 °C under inert conditions (nitrogen gas) and 106 to 900°C in oxidant air-atmosphere. This study maintained both gas flow rates at 20 mL/min. The sample was heated under non-isothermal conditions using five different heating rates of 10, 15, 20, 25, 30°C/min. The different heating rates were subsequently used to assess their impact on thermal analysis.

Furthermore, the data obtained were analyzed for thermal characteristics by developing thermogravimetric curves (TG) showing the sample mass loss and its derivatives (DTG) showing the mass loss rate. In addition, combustion characteristics were also analyzed by determining the ignition temperature (T_i) and Burnout temperature (T_b) using the intersection method [16]. TGA was carried out in this analysis using Shimadzu thermal software (TA-60WS) and Microsoft Excel© 2013.

Kinetic Analysis Calculation

TGA data of a single heating rate test could be used to calculate kinetic parameters by the Coats-Redfern method [17]. Therefore, this study implemented the Coats-Redfern method to determine the activation energy (E, kJ/mol). The activation energy was conducted in the combustion zone between the ignition and burnout temperature through the first-order reaction [17], [18]. In this study, the following equation emphasized the description of kinetic reaction [15].

$$\frac{d\alpha}{dt} = A \, exp \, (-\frac{E}{RT}) f(\alpha) \quad (1)$$

$$\alpha = \frac{(mo - mt)}{(mo - mf)} \quad (2)$$

Where α = mass conversion, f(α) = the hypothetical model of the reaction mechanism, A = the frequency factor (min-1), E = Activation energy (kJ/mol), R = the gas constant (8.314 kJ/mol.K), T = Absolute temperature (K), t = time (min), mo = the initial mass of the sample, mt = the mass of the sample at time t, and mf = the final mass. For non-isothermal conditions at a constant heating rate (β=dT/dt), the kinetic equation is expressed as follows [15].

$$\frac{d\alpha}{dt} = \frac{A}{\beta} \, exp \, (-\frac{E}{RT}) f(\alpha) \quad (3)$$

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

Assuming the kinetic reaction order is one, the derivation of equation 4 is expressed as follows [18]:

$$\ln ln \left(-\frac{ln \, ln \, (1-\alpha)}{T^2}\right) = \ln ln \left(\frac{AR}{\beta E}\left(1-\frac{2RT}{E}\right)\right) - \frac{E}{RT} \quad (5)$$

A plot was developed to determine the activation energy between $ln \, ln \left(-\frac{lnln \, (1-\alpha)}{T^2}\right)$ as the Y-axis and 1/T as the X-axis. Therefore, the plot obtained produced a linear line with slope -E/R [18].

RESULTS AND DISCUSSION

Proximate and Ultimate Characteristic of EFB and Biochar EFB

The proximate and ultimate analyses were conducted to determine changes in the characteristics of the materials (raw EFB, EFB-HT210°C, EFB-HT230°C), as presented in Table 1. This emphasized the existence of a change in the characteristics of EFB before and after Hydrothermal Processing. The hydrothermal process was effectively implemented to improve the fuel properties of EFB and could change the biomass's characteristics from hydrophilic to hydrophobic. Therefore, higher HTC temperature caused the lower moisture content attached to the biochar [19].

Table 1.
Proximate and Ultimate Analysis of raw EFB and Biochar EFB

	EFB-Raw	EFB-HT210°C	EFB-HT230°C
Proximate Analysis (%wt-as received)			
Moisture Content	9.92	3.18	2.83
Ash	9.46	7.98	12.43
Volatile Matter	66.49	65.15	56.61
Fixed Carbon	14.13	23.69	28.13
Ultimate Analysis (%wt-as received)			
C	43.54	52.05	52.47
H	6.06	5.82	5.30
O	40.15	34.92	28.14
N	0.84	0.82	1.10
S	0.02	0.2	0.19
GCV (kcal/kg)	3,887	4,688	4,902

Source: Experimental Data analysis (2023)

As shown in Table 1, the fixed carbon (FC) and ash content of biochar increased with higher HTC temperature due to the reduction of volatile matter during the HTC process. The occurrence of hydrolysis and decarboxylation processes were also observed, leading to the reduction of organic and volatile organic/matter [20]. HTC subsequently caused an increase in carbon content, accompanied by a decrease in hydrogen, nitrogen, and oxygen. This process decreased the oxygen-weight ratio, contributing to higher energy content and GCV values in biochar [21].

In addition, the GCV of raw EFB, EFB-HT210°C, and EFB-HT230°C were 3,887, 4,688, and 4,902 kcal/kg, respectively. This indicated that the HTC temperature variable was very influential in the hydrothermal process on the fuel properties of biochar [22]. The higher HTC temperature will decrease the volatile matter content that caused the increase of fixed carbon and GCV.

The Thermal Characterization of Raw EFB, EFB-HT210°C, and EFB-HT230°C on Heating Rate of 20°C/min

The thermal characteristics, combustion characteristics, and kinetic parameters obtained from TGA were considered suitable references for fuel selection in combustion. According to Figure 2 and Figure 3, the TG and DTG curves of raw EFB, EFB-HT210°C, and EFB-HT230°C were burned at a heating rate of 20°C/min. This heating rate was selected because heating rates between 20 and 30 C/min avoid thermal lag dominance and time-consuming measurements at high and lower heat levels, respectively [16]. The heating rate of 20°C/min is selected because it produces more accurate measurements compared to 30°C/min. The accuracy of this result is due to the smaller heating rate interval used.

Figure 2 shows the TG profile of EFB, EFB-HT210°C, and EFB-HT230°C, exhibiting a difference between raw and HT EFB. In this case, the curve shifted to the right in HT EFB, indicating a higher carbon and lower VM content.

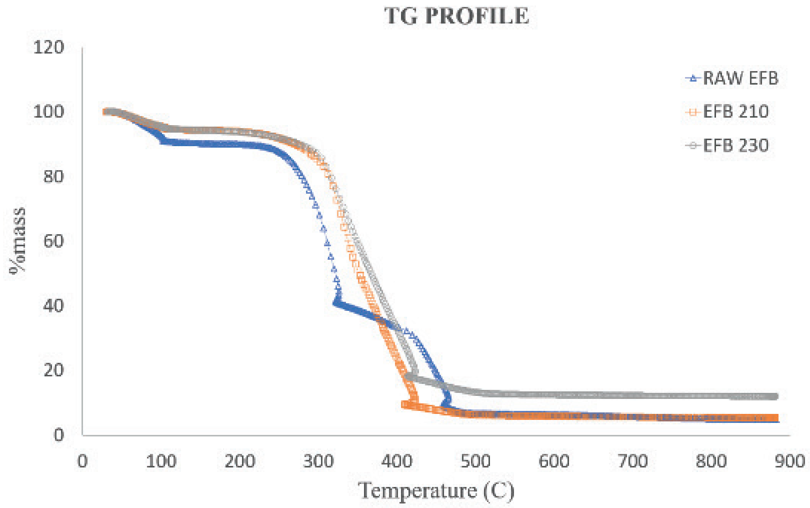


Figure 2.
The Comparison of TG Profile on Raw EFB and Biochar EFB
Source: Researchers (2023)

The DTG profile curve is presented in Figure 3. The DTG curve is divided into three stages. The first stage is the moisture content removal process around 35°C-280°C. Secondly, VM removal and char combustion were characterized by a rapid and significant decrease in mass at 280°C-415°C. In the third stage, combustible components burned out and provided ash residue [9].

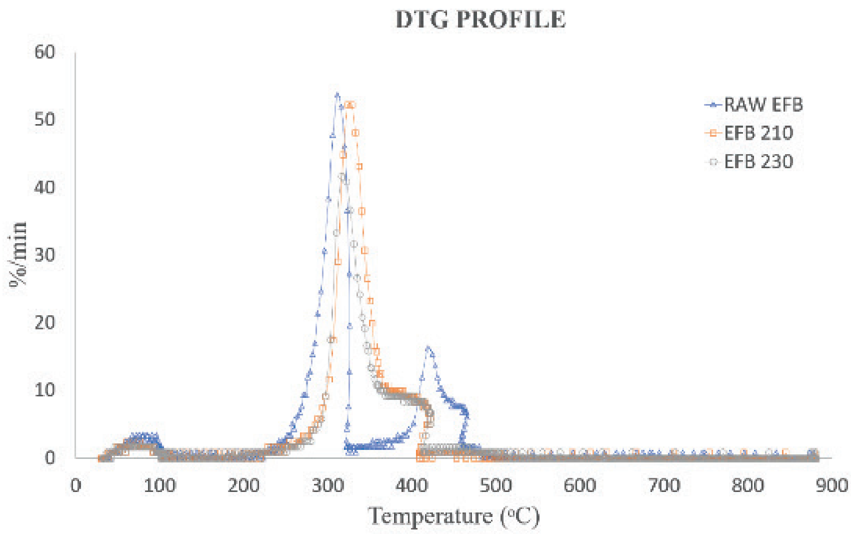


Figure 3.
The Comparison of DTG Profile on Raw EFB and Biochar EFB
Source: Researchers (2023)

Based on the results, the ignition and burnout temperatures were obtained from the TG-DTG curve. This indicated a sudden increase in the DTG curve characterized by the ignition temperature (Ti).

Meanwhile, the burnout temperature (T_b) emphasized the completion of oxidation on the curve [10]. In Table 2, raw EFB also had a lower T_i than HT EFB. For raw EFB was 279°C while for EFB-HT210°C and EFB-HT230°C were 297°C and 298°C. This is because the volatile matter and moisture content in raw EFB is higher than in the treated EFB. This proved that higher VM content led to lower ignition temperature. Like T_i, the burnout temperature in HT EFB had a higher value.

For raw EFB was 378°C while for EFB-HT210°C and EFB-HT230°C were 407°C and 450°C. This is prioritizing the increase in FC content due to the hydrothermal process. Fixed carbon was also considered the solid fuel component, prioritizing preserving thermal stability throughout its combustion. This significantly maintained thermal stability while reducing fuel weight loss during combustion [23]. Therefore, elevated levels of fixed carbon led to prolonged combustion and higher burnout temperatures. Activation energy is the minimum energy required to start a reaction; reactants will find it difficult to react if they have a high activation energy [24].

Table 2 shows an increase in activation energy after the hydrothermal process, which means that the raw EFB will be easier to react with than EFB treated. For raw EFB, the activation energy was 57.50 kJ/mol, while B-HT210 °C and EFB-HT230°C were 58.84 kJ/mol and 62.16 kJ/mol. Activation energy is related to volatile matter content: the higher the volatile matter, the lower the activation energy [17]. Table 1. Shows that raw EFB has higher VM, which results in lower activation energy than EFB treated by hydrothermal.

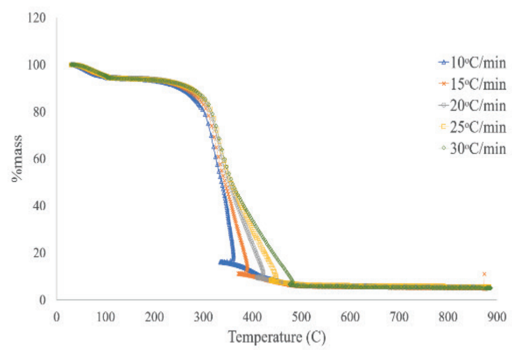
Table 2.
The Comparison of Temperature Ignition, Temperature Burnout and Activation Energy of Raw EFB and Biochar EFB

Sample	T_i (°C)	T_b (°C)	Equation	Ea (kJ/mol)
EFB-Raw	279	378	y = -6917x - 1,5339	57.50
EFB-HT210°C	297	407	y = -7077.7x - 1.9642	58.84
EFB-HT230°C	298	450	y = -7476.5x – 1.508	62.16

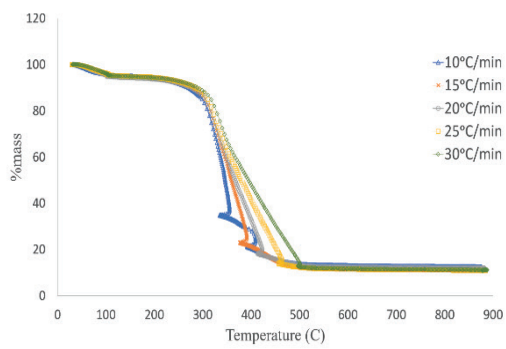
Source: Researchers (2023)

The Effect of Heating Rate on Thermal Characteristic (TG and DTG Profil of EFB-HT210°C and EFB-HT230°C

The heating rate was the rate at which the temperature increases in degrees per minute, measured on the Celcius or Kelvin scale [25]. The heating rate was a factor affecting the thermal profile of hydrothermal EFB combustion in both TG and DTG curve profiles. The changes in the thermal profile are shown in Figure 4 and Figure 5.



(a)

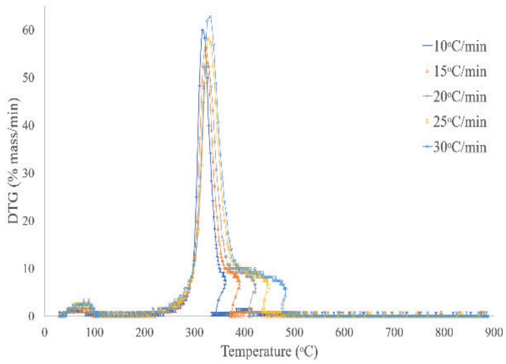


(b)

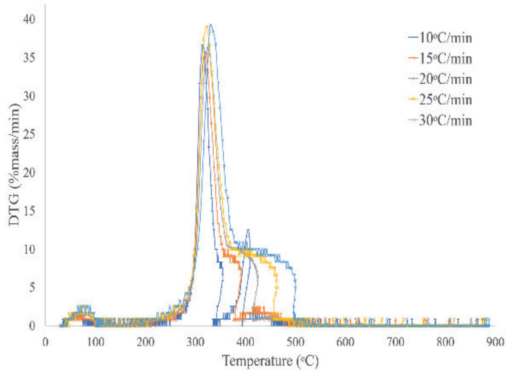
Figure 4.
The Effect of Heating Rate on TG Profile (a)EFB-HT210°C and (b) EFB-HT230°C
Source: Researchers (2023)

According to Figure 4, the TG profile curve indicated the same trend as the heating rate increases for both samples. This proved that the higher the heating rate, the more the thermal profile will shift to the right. Moreover, the reaction observed also shifted to a higher temperature. This result was in line with Hamzah (2021) and Ninduangdee (2022) [12], [15]. Showering leads to an extensive sample breakdown rather than quick thermal processes without considering the temperature [25].

Figure 5 also showed the DTG profiles of EFB-HT210°C and EFB-HT230°C, which exhibited changes in the three peaks. These changes manifested as peak shifts toward higher temperatures with increasing heating rates, as presented in Table 3.



(a)



(b)

Figure 5.
The Effect of Heating Rate on DTG Profile (a)EFB-HT210°C and (b) EFB-HT230°C
Source: Researchers (2023)

Table 3.
The Effect of Heating Rate on Peak Temperature in DTG Profiles

Sample	EFB-HT210°C					EFB-HT230°C				
Heating Rate (°C/min)	10	15	20	25	30	10	15	20	25	30
Tpeak (°C)	316	323	324	327	332	314	321	324	323	330

Source: Researchers (2023)

Based on Table 3, the effect of heating rate was found on the peak temperature of DTG profiles. Our results demonstrated that the peak temperature for EFB-HT210°C at 10, 15, 20, 25, and 30°C/min are 316, 323, 324, 327, and 332°C while for EFB-HT210°C at 10, 15, 20, 25, and 30°C/min are 314, 321, 324, 323, and 330°C. This indicated that the increase in heating rate caused the elevation of the peak temperature. The occurrence of a temperature shift was also due to ineffective heat transfer when the heating rate was carried out quickly. In this case, the transfer occurring to the interior part of the biomass particles decreased at a high heating rate.

Therefore, a higher temperature was required to achieve the same decomposition temperature or heating process between the external and internal parts of the biomass [12]. The results also showed that a slow heating rate provided sufficient time for thermochemical reactions to occur, leading to the simultaneous combustion of the biomass's internal and external surfaces at a specific temperature and period. This reaction led to the simultaneous decomposition of the exterior and interior components of the energy source [26]. According to Lu and Chen (2015), the rapid heating of particles on biomass surfaces caused a more considerable thermal lag, leading to lengthy complete combustion [16].

The Effect of Heating Rate on Combustion Characteristics (Ignition Temperature, Burnout Temperature, and Activation Energy) of EFB-HT210°C and EFB-HT230°C

The heating rate could affect the TG and DTG profiles and combustion characteristics, such as ignition/burnout temperature and activation energy. The effect of the heating rate was also observed in Table 4. For EFB-HT210°C at 10, 15, 20, 25, and 30°C/min, the ignition temperature were 293, 293, 297, 302 and 304°C; the burnout temperature were 393, 402, 407, 416 and 419°C; and the activation energy were 83.75, 71.66, 58.84, 57.62 and 48.70 kJ/mol. EFB-HT230°C at 10, 15, 20, 25, and 30°C/min, the ignition temperature were 289, 296, 298, 297, and 304°C; the burnout temperature were 410, 433, 450, 460, and 472°C; and the activation energy were 82.56, 70.13, 62.16, 46.18, 29.97 kJ/mol. The ignition and burnout temperatures increased with decreased activation energy at higher thermal levels. This proved that the decrease in activation energy was due to the power supply through fuel support and a high heating rate [27].

Table 4.
The Effect of Heating Rate on Tignition, Tburnout
and Activation Energy of EFB-HT210°C and EFB-HT230°C

Sample	Heating rate (°C/min)	T_i (°C)	T_b (°C)	Equation	Ea (kJ/mol)
EFB-HT210°C	10	293	393	y = -10073x + 3.4196	83.75
	15	293	402	y = -7744.4x – 0.604	64.38
	20	297	407	y = -7077.7x – 1.9642	58.84
	25	302	416	y = -4445.9x – 6.1173	36.96
	30	304	419	y = -3270x – 7.9927	27.19
EFB-HT230°C	10	289	410	y = -10548x + 3.9871	87.69
	15	296	433	y = -8435.3x + 0.2407	70.13
	20	298	450	y = -7476.5x – 1.508	62.16
	25	297	460	y = -5554.7x - 4.6965	46.18
	30	304	472	y = -4807.9x - 6.0543	39.97

Source: Experimental Data analysis (2023)

The increase in heating rate was also due to its effect on reaction time at a specific temperature. This indicated that a higher heating rate led to lower reaction times, shifting the response towards the high-temperature zone [17]. Moreover, the increase in burnout temperature with a higher heating rate was observed because of the elevated heat needed to attain an equivalent level of reaction. This condition was obtained from increased resistance within the particle's internal reactions, causing an elevation in combustion temperature [24].

CONCLUSION

In conclusion, EFB samples before and after HT processing were analyzed using TGA to compare thermal and combustion characteristics and kinetic parameters. The results showed that biochar EFB has a higher ignition temperature, burnout temperature, and activation energy than raw EFB. This increase was observed due to a decrease in volatile matter and moisture content and an increase in FC regarding hydrothermal treatment. The increase in heating rate also influenced combustion, causing the increase of the ignition and burnout temperatures, as well as decreased activation energy. Based on the results, the differences in heating rate affected the peak temperature in DTG. Considering the thermal profile and combustion characteristic, HTC at 230°C was the most favorable for producing solid fuel from EFB for cofiring.

Acknowledgments

The authors are grateful to the Energy Conversion Laboratory-Energy Conversion and Conservation Research Center for enabling the study to perform well. The authors thank the Energy and Manufacturing Research Organization - BRIN in the Program House of Renewable Energy (RP ET-03) for financial support.

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