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Effect of Temperature on Calorific Value of Pyrolyzed Empty Fruit Bunch (Efb) Derived Biochar

Siti Sarah Aliah Mohd Najib[1], Mahaletchumi A/P Ganesan[1], Norhana Mohamed Rashid[1]* Mohd Nazri Mohd Sokri[1], Nabilah Zaini[2] and Noorshawal Nasri[1]

[1]Energy Engineering Department, School of Chemical and Energy Engineering, Faculty of Engineering, Universiti Teknologi Malaysia, 81310 UTM Johor Bahru, Johor

[2]SHIZEN Conversion and Separation Technology ikohza, Department of Chemical and Environmental Engineering, Malaysia-Japan International Institute of Technology, Universiti Malaysia Kuala Lumpur, 54100 Kuala Lumpur, Wilayah Persekutuan Kuala Lumpur

ABSTRACT

The residues from the oil palm industry are the main contributors to biomass waste in Malaysia, and these wastes require extra attention with respect to handling. A survey of the literature indicates that most of them are handled with unsatisfactory practices that negatively impact the environment. Therefore, it is very important that they be utilized for more beneficial purposes, particularly in the context of the development of biofuels via pyrolysis technology. Due to its high carbon content, rich in lignin and low cost, empty fruit bunch (EFB) shows potential to be a good precursor for the production of biochar. The pyrolysis temperature greatly affects biochar properties and its potential usage. Many researches work on biochar have been carried out to assess its potential by investigating its characteristics. The most common thermochemical technique to produce biochar is pyrolysis, during which the organic components are decomposed at adjustable temperature in a nitrogen-limited atmosphere. The focus of this study is to identify the effect of temperature (300, 350, 400, 450 and 500 °C) on calorific value of pyrolyzed EFB derived biochar. Eight experimental runs were conducted. The results were completely analyzed by Analysis of Variance (ANOVA). The model was statistically significant. The factor studied which temperature was significant with p-values < 0.0001. The value of R^2 was 0.9633 which indicated that the temperature showed high correlation to the calorific value of biochar from EFB pyrolysis process. A quadratic model equation was developed and employed to predict the highest theoretical calorific value. The maximum biochar calorific value was achieved at pyrolysis temperature of 500 °C. Char yield was obtained highest at 300°C around 53.36 wt% and started to decrease as temperature increase. Result of this experiment revealed that the calorific value of biochar increases as the temperature increases while the yield percentage of biochar decreases as the temperature increases. The yield of biochar decreases with temperature because of the secondary tar reactions of the volatiles, such as thermal cracking, that favors the increase of gas yield.

***Corresponding author**

Norhana Mohamed Rashid, Energy Engineering Department, School of Chemical and Energy Engineering, Faculty of Engineering, Universiti Teknologi Malaysia, 81310 UTM Johor Bahru, Johor. Tel: +60129446141; E-mail: norhanarashid@utm.my

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Introduction

According to data published by United Nations the population of the world in 2013 was 7.7 billion and it is expected an increase of 1 billion until 2025 and will reach 9.7 billion by the year 2050 [1]. As the population of the world is increasing energy demand is also getting higher. Since energy is required for industrial, agricultural and transportation development, power generation and many more different sectors, energy is very crucial for development of any country. The major source for the energy is the fossil fuels which are buried deep inside the earth. These sources are limited and will not be able to fulfil the world's increasing energy demand for a very long time. Furthermore, it is a known fact that carbon dioxide, gas released when fossil fuels are burnt, is one of the primary gas responsible for global warming. Rise in temperature of earth has resulted in melting of polar ice caps, flooding of low-lying areas and rise in sea levels. If such conditions continue, our Earth might face some serious consequences in near future. Therefore, an interest is developing among the researchers to find alternate source of energy. Recently, biomass has attracted much attention and is considered to be a promising renewable source of energy [2].

Biomass presents a clean, renewable strength source that might dramatically enhance the environment, economy and energy security. It is one of the most promising sources of alternative energy which can solve the problem of energy crisis in world up to some extent due to its potential availability. Biomass is mainly derived from the agriculture or forestry sector Interest in biomass utilization in Malaysia has developed at least since the last two decades. As for the oil palm industry, the oil palm utilization committee (OPTUC) established in 1991, spurred discussions on strategies for commercial exploitation of oil palm biomass, updating and dissemination of information and review on the technologies and supply [3].

Malaysia is one of the largest producers of palm oil and has an abundance of oil palm residues produced throughout the year

[4]. According to Malaysia Palm Oil Board (MPOB), the total production of crude palm oil in Malaysia was 3.4 million tonnes in 2016, hence about 25.5 million tonnes of oil palm wastes were generated since 75 wt% of the solid wastes were produced from 10 wt% of crude palm oil [5]. The empty fruit bunch (EFB), a solid residue which accounts for 20% of the fresh fruit weight, is one of the palm biomass produced in abundance after oil extraction at palm oil mills [6]. Energy potential of empty fruit bunches is attractive as it contains neither chemical nor mineral additives, and depending on proper handling operations at the mill, it is free from foreign elements such as gravel, nails, wood residues, and waste Due to its high carbon content, rich in lignin and low cost, EFB shows potential to be a good precursor for the production of charcoal. The charcoal derived from EFB is commonly known as biochar [7].

Biochar is defined as "a carbon (C)-rich solid material from the thermo-chemical conversion of biomass at temperatures below 900 °C, in an oxygen-limited environment" [8]. The techniques for producing biochar mainly include pyrolysis, gasification, and hydrothermal carbonization, etc. The most common thermochemical technique to produce biochar is pyrolysis, during which the organic components are decomposed at adjustable temperature in an oxygen-limited atmosphere. Pyrolysis, in the past decade has developed itself as the most promising thermochemical method to produce energy from the biomass. Pyrolysis is a process in which thermal degradation of the chemical constituent of the biomass is made, keeping the reaction atmosphere inert to obtain the energy [9]. Pyrolysis of the EFB results in three products which are biochar, bio-oil and gas. Biochar is a solid carbon rich by-product of thermal stabilization of biomass or any other organic matter [10].

Statistical method such as response surface methodology (RSM) enables researchers to design the experiments and evaluating the interactions among factors and responses throughout the study. More researches in recent time have used this approach that combines experimental design, regression modeling techniques, and optimization tool to predict the maximum yield for bioproducts of interest [11]. According to a research conducted by Muhamadin, one factor design was used to optimize the medium formulation such as molasses, nitrogen sources, and glutamic acid as the one factor design allows researchers to study the effect of a factor when the conditions of other factors vary. Thus, it leads a better understanding of how the existing process inputs influence the performance of the process [12].

Studies on the biomass pyrolysis in the recent past have revealed that the production of the biochar depends upon several factors like biomass properties like particle size, reaction temperature, reaction holding time and heating rate [13]. Besides this biochar yield, properties of the biochar also have been found to vary with different process conditions like temperature, residence time, heating rate, particle size etc [14]. The main focus of this study is to determine the effect of pyrolysis operated at different temperature on calorific value of EFB biochar and to show the importance of temperature effect towards biochar properties via RSM.

Experimental

Sample Preparation

Empty Fruit Bunch (EFB) was used as a raw material that collected from oil palm industry in Kahang, Johor. Firstly, the EFB processed by cutting to small size and then dried at 100°C for 2 days. Then, the dried EFB was grinded to and sieved into size between 250μm to 1.5mm and then stored.

Sample Characterization

Few samples from grinded and sieved EFB was send to thermogravimetric analysis (TGA), Fourier Transform Infra-Red (FTIR) analysis and calorific test. The TGA analysis was conducted to identify the thermal behaviour of the raw EFB. By analyzing the TGA.DTG graph, the parameters such as ignition temperature (Tign), maximum temperature (Tmax) and transition temperature (Tg) can be deduced [15]. The calorific value of raw EFB was determined using bomb calorimeter. The elemental and functional group composition of raw EFB was analyzed using Fourier Transform Infra-Red (FTIR) spectroscopy.

Pyrolysis Experiment

The schematic diagram of the fixed-bed reactor system used for the pyrolysis studies was shown in Figure 2.1. The major components of the reactor system included a vertical fixed bed reactor tube, furnace, gas cylinder (nitrogen gas, N_2), condenser and thermocouple to monitor the reaction temperature and a cooling circulation unit. A flow meter is to control flow of N_2 into the reactor. Nitrogen was flowed at 1 litter per minute. The pyrolysis process operated at different temperature, 300, 350, 400, 450 dan 500 °C based on TGA results.

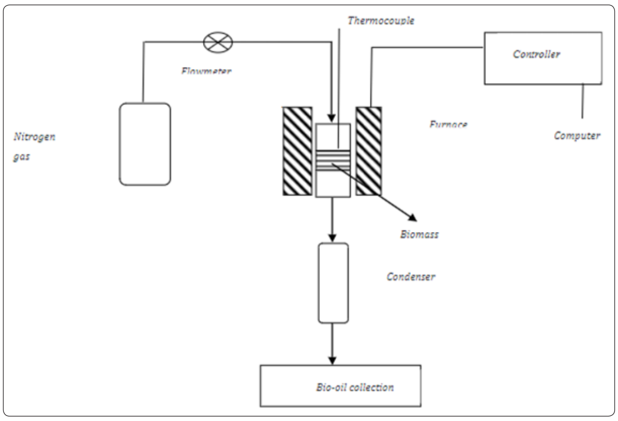

Figure 2.1: The tubular fixed bed reactor set-up

The pyrolysis procedure involves three phases; initial phase, constant phase and the cooling phase. During the initial phase, the sample was heated at a rate 10 °C per minute. In the constant phase, the sample is left to be heated for 2 hours. After that, the heating process stopped. The sample need to be left to cool down.

There was weight loss when compared before pyrolysis process. The yield of the chars was calculated by using formula as shown below:

$$\text{Char Yield} = \frac{\text{Mass of char (g)}}{\text{Mass of EFB sample}} \times 100\% \qquad [1]$$

After the yield percentage was calculated, the biochar samples were sent for calorific test.

Response Surface Methodology

Design of Experiments (DoE) was utilized to investigate the effect of pyrolysis temperature of EFB towards the production of biochar and its calorific value. A standard RSM analysis in conjunction with one factor design was used to develop experimental runs. In single factor experiments, ANOVA models are used to compare the mean response values at different levels of the factor [16]. Each level of the factor is investigated to see if the response is significantly different from the response at other levels of the factor. The analysis of single factor experiments is often referred to as one-way ANOVA [17]. In this experiment, the temperature of pyrolysis is the factor and calorific value, yield percentage of biochar are responses. The pyrolysis temperature was chosen as the studied parameter with considered at three level; low (300°C), central (400°C) and high (500°C). Concomitantly, 8 experimental runs were suggested. The results were analyzed by Analysis of Variance (ANOVA) using Design Expert software version 6.0.4.

Results and Discussion

Characterization of Raw EFB

After grinding and sieving process, the characterization of raw EFB was conducted. Thermogravimetric analysis (TGA) is used to analyze the thermochemical properties of EFB. Furthermore, FTIR was conducted to identify the functional group presence in raw EFB. Bomb calorimeter was used to determine the calorific value of samples.

Thermogravimetric Analysis (TGA)

The TGA analysis was performed on grinded raw EFB. Based on the TGA analysis results, the TGA/DTG curves of raw EFB was plotted. The plot was shown in Figure 3.1. The parameters ignition temperature (Tign), maximum temperature (Tmax), transition temperature (Tg) were deduced from the TGA/DTG curves. From Figure 3.1, it can be deduced that raw EFB decomposition occurs in four (4) stages; drying (A), heating (B), devolatization (C) and char aggregation (D).

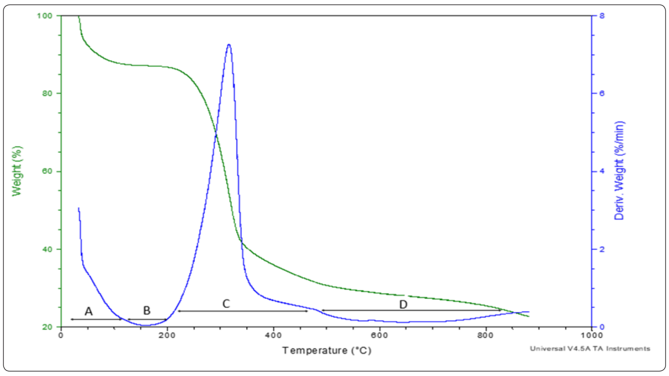

Figure 3.1: TGA/DTG curve of EFB

The fuel ignition temperature (Tign) signifying the onset of devolatization was 207°C. The peak temperature of devolatization was 318°C and is denoted as glass transition (Tg) or maximum temperature (Tmax) as deduced from the TGA/DTG graph. In addition, two peaks were observed in the DTG curve of the sample. The small peak between observed from 20°C to 127°C denoting the drying process of the reaction. Sample weight loss during this stage was 28.03%. The second peak between 207°C and 498°C signifies the devolatization of the sample. The TGA result of raw EFB obtained were almost similar as the TGA result in a research conducted by Nyakuma [18]. During this stage of the reaction, the condensable and non-condensable matter in the fuel is thermally decomposed into gases, char and tar. Sample weight loss during this step of the reaction is around 66% of initial sample weight. The TGA/DTG temperature and weight loss profile of the sample is presented in Table 3.1.

Table 3.1: TGA/DTG profile of raw EFB

Stage	Process	Onset (°C)	End (°C)	Weight Loss, Wt %
A	Drying	20	127	28.03
B	Heating	127	207	34.09
C	Devolatization	207	498	66.03
D	Char aggregation	498	880	71.79

The results in Table 3.1 indicate that the thermal decomposition of raw EFB occur from 20°C to 880°C resulted in ~ 72% of the sample. Hence, higher heating rates and temperatures greater than 880°C are required to ensure complete decomposition of raw EFB into the desired products of thermal conversion. According to a journal by Mr.Nyakuma, temperatures greater than 900°C are required to ensure complete decomposition of raw EFB into the desired products of thermal conversion. Hence, the TGA result for the raw EFB is acceptable.

Fourier-Transform Infrared Spectroscopy (FTIR)

The elemental and functional group composition of the raw EFB was analyzed using Fourier Transform Infra-Red (FTIR) spectroscopy. The distribution of elements and functional groups in the fuel is vital for determining the composition and distribution of pyrolysis products. The FTIR spectra for the raw EFB was presented in Figure 3.2.

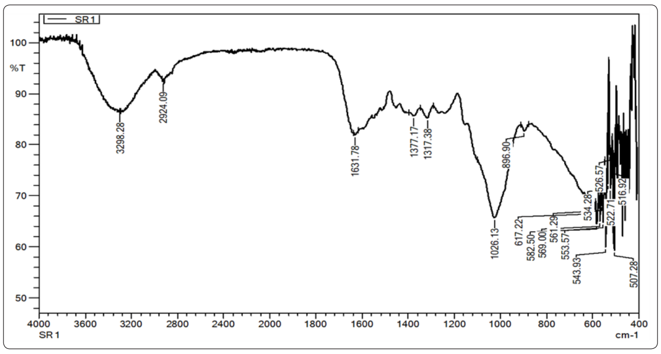

Figure 3.2: FTIR Spectra of EFB Briquette

A strong and broad absorption was observed at wavenumber 3298.28 cm-1 which is related to the stretching of O-H group indicate the presence of alcohols. The other prominent one around wavenumber 2924.09 cm-1 is due to the C-H stretching vibrations found in CH_3 and CH_2. Other than that, the medium intensity band observed in 1631.78 cm^{-1} is due to C=C stretching from aromatic ring of lignin. In addition, the area of 1500 to 600 cm-1 is called the fingerprint area of spectra which has many sharp and well defined absorption bands due to the various functional groups presence in each component of raw EFB. The elemental and functional group composition of the raw EFB obtained from this FTIR analysis was similar to a previous study conducted by Wei Ting [19]. He distinctive bands in the fingerprint region and the components to which these peaks are shown in Table 3.2.

Table 3.2: FTIR Wavenumber Characterization of Native Raw EFB

Wavenumber (cm^{-1})	Group
3298.28	O-H stretching
2924.09	C-H stretching
1631.78	C=C stretching
1377.17,1317.38	C-H deformation vibrations for alkenes
1026.31	Ether groups
896.90	C-H bending

Calorific Value

The calorific value of raw EFB was determined using bomb calorimeter. The calorific value of raw EFB is 3505.05 kcal/kg. The The calorific value for EFB is consistent with findings in journal by Helwani [20].

Design and analysis of results by using response surface methodology

Table 3.3 shows the yield percentage of biochar and calorific value at various bed reactor temperatures from 300 to 500ºC. It shows that the product yields and calorific value are influenced by the process temperature. The maximum biochar calorific value was achieved at pyrolysis temperature of 500 °C. Char yield was obtained highest at 300°C around 53.36 wt% and started to decrease as temperature increase. The trend of HHV and yield percentage from temperature 300° to 500° are in consistent with journal by Hanif [21]. As mentioned earlier, in single factor experiment like this, ANOVA models are used to compare the mean response values at different levels of the factor. Each level of the factor is investigated to see if the response is significantly different from the response at other levels of the factor. In this study, the responses analyzed using the one factor design in RSM are biochar's yield percentage and it's calorific value. The results of ANOVA for biochar yield percentage and HHV are summarized in Table 3.4. The ANOVA 99.99% confidence level indicated that biochar yield percentage and energy contents are affected by temperature. Values of "Prob > F" less than 0.0500 for both responses indicate that the model terms are significant. The effect of pyrolysis temperature on yield percentage and calorific value of biochar were discussed in upcoming sections.

Table 3.3: Yield Percentage and Calorific Value of Different Pyrolysis Temperature

Standard Order	Temperature (°C)	Higher Heating Value, HHV (Kcal/Kg)	Yield Percentage (%)
1	300	4712.89	53.3597
2	300	4712.89	53.3597
3	350	4749.36	52.4212
4	400	4780.55	42.9771
5	400	4780.55	42.9771
6	450	4880.57	41.9856
7	500	5278.9	36.4247
8	500	5278.9	36.4247

Table 3.4: Analysis of Variance for Adjusted Model for Yield Percentage and HHV of biochar

Source	Sum of Squares	Degree of Freedom	Mean square	F value	p value
Biochar Yield Percentage (Process order, linear)					
Model	339.52	1	339.52	103.61	<0.0001
Residual	19.66	6	6.55		
Lack of Fit	19.66	3	6.55		
Pure Error	0.000	3	0.000		
Corrected Total	359.19	7			
R2: 0.9453					
R2 Adjusted: 0.9361					
Higher Heating Value (kcal/kg) (Process order, quadratic)					
Model	3.980E+005	2	1.990E+005	92.86	<0.0001
Residual	10714.10	5	5357.05		
Lack of Fit	10714.10	2	5357.05		
Pure Error	0.000	3	0.000		
Corrected Total	4.087E+005	7			

Effect of Temperature on Yield Percentage

Table 3.3 shows the biochar yield at different pyrolysis temperatures. The yield percentage of biochar was decreased as the pyrolysis temperature was increased from 300–500°C. The highest EFB biochar yield percentage was 53.36% (at 300°C) and the lowest was 36.42% (at 500°C). The results of ANOVA for biochar yield percentage are summarized in Table 3.4 while Figure 3.3 shows the yield percentage model of EFB biochar. The results show that the predicted responses using the quartic model are close to the experimental values recorded, with adjusted R-squared of 0.9361. The lack of fit was not significant, indicating that the model fit was acceptable. From ANOVA, the value of R^2 was 0.9361 which indicated that the selected parameter, temperature contributed 93.61 % to the production of the biochar. The linear model equation for yield percentage value in terms of temperature as follows:

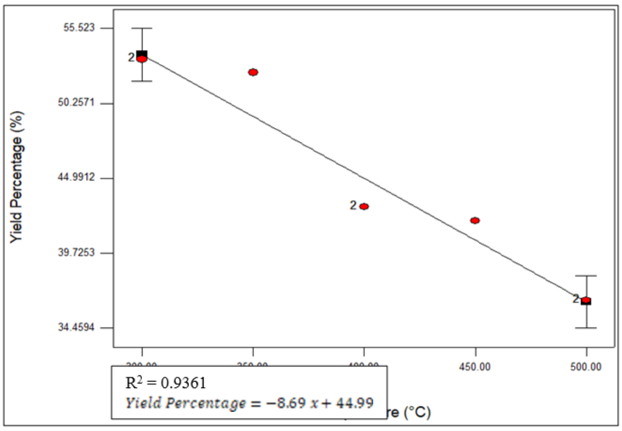

Figure 3.3: Yield Percentage Model of Biochar

The pyrolysis temperature range of 300–400°C was chosen because at above 450°C the biochar yield was reduced due to decomposition of volatile fractions as well as intermediate melt in the biochar structure [22]. The temperature of 500°C is the highest temperature that increases the biomass decomposition and decrease the potential for biochar formation. Clauston (2014) reported that the yield of biochar depended on destructive reaction of cellulose and polymerization process of biochar. Besides that, a decrease in yield with increasing temperature can be due to either greater primary decomposition of EFB and RH or secondary decomposition of the biochar residue at higher temperatures. As the pyrolysis temperature increases, the final solid residue initially decreases, as a result of the competition between charring and devolatilization reactions, of which the latter is more favoured [23]. According to Mr.Tripathi, increasing the temperature in pyrolysis affects the biochar yield in a negative way as the increase in the temperature allows the thermal cracking of heavy hydrocarbon materials, leading to the increase of liquid and gaseous and decreases in the biochar yield. Low temperature is suitable for high biochar yield because at high temperature, energy given to the biomass may exceed the bond cessation energy which supports the release of the volatile components of the biomass. These volatile constituents of biomass come out in the form of gases resulting in less char yield. Although there are many literatures available with the study of effect of temperature on biochar yield but finding the suitable temperature for biochar production is a difficult task because the optimized temperature for the high biochar production depends upon nature, composition and type of biomass [24].

Effect of Temperature on Calorific Value

According to the results presented in Table 3.4, quadratic was considered an appropriate process order for biochar HHV. From ANOVA, the value of R^2 was 0.9633 which indicated that the selected parameter, temperature contributed 96.33 % to the production of the biochar. There was a good correlation between the calorific value of biochar and temperature. The developed model fits into a quadratic equation. Figure 3.3 shows the energy content models of pyrolysis product, biochar. The quadratic model equation for calorific value in terms of temperature as follows.

$$HHV = 4771.41 + 266.14\,x + 222.96\,x^2 \qquad [3]$$

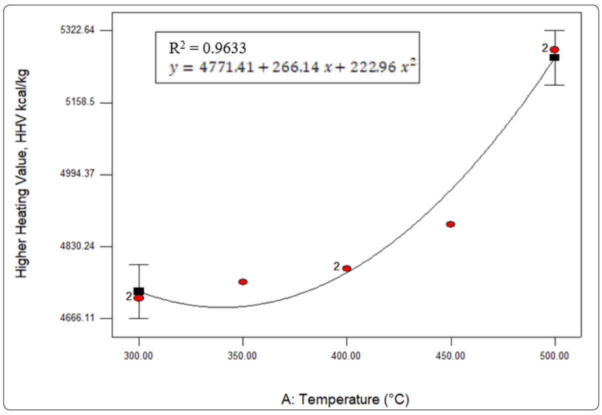

Figure 3.4: Energy Content Models of Biochar

HHV is defined as the maximum amount of energy that can be released upon combustion of 1 kg of the sample. Improving the HHV and increasing the energy density are the main purpose of carbonization [25]. The HHV ranged from 4712.89 to 5278.9 kcal/kg, implying that the energy content in the biochar samples increased by 34–50.61% as compared to the raw EFB.The increase of heating value of the biochar at temperature up to 450°C can largely be attributed to optimum extraction of the combustible elements from the biomass, particularly carbon and hydrogen during pyrolysis course, which are favourably elevated to the biochar product. Similarly, the elevating heating value of syngas may be due to secondary tar reactions of the volatiles, such as thermal cracking, that favors the increase of gas yield [26]. Because of the increase in gas yield at high temperature, the char yield subsequently decreased as the operating temperature increases. This is an indication that, at higher temperature such as 500 °C, the energy contents of the biomass were elevated to liquid and gas products. Similar trends for the pyrolysis yields of different biomass were also observed by many researchers [27]. This study only study the effect of temperature from 300-500°C. Additional experiments are needed to be performed at higher temperatures to analyse the effect of higher pyrolysis temperature on calorific value of EFB biochar.

Conclusion

In the range of 300 to 500 °C, the calorific value of biochar became the maximum at 500 °C, but in terms of yield percentage it shows a decline. Beyond 400°C, yield of biochar decreases with temperature because of the secondary tar reactions of the volatiles, such as thermal cracking, that favors the increase of gas yield. By analyzing the ANOVA table, it can be concluded that the temperature has high effect on yield percentage and calorific value of biochar. Since the results are consistent with the past results, the tendency that the yield decreases as the temperature increases is considered to be correct. The behavior of the decrease also reflected the result of TGA. This study only focuses the effect of one parameter which is temperature while there are more other factors like particle size, holding time which can be considered to optimize the pyrolysis process.

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