Kinetic Study of Non-Isothermal Reactions on the Pyrolysis of Various Biomass Waste by using Thermogravimetric Data

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Article Info	Abstract
	Population growth causes an increase in the need for petroleum. However, petroleum as primary energy is currently increasingly limited in availability. Required alternative energy sources that can be renewed to overcome these problems, one of which is bio-oil. Bio-oil is produced by a pyrolysis process using biomass such as sugarcane bagasse, rice husk, and empty oil palm fruit bunches (EFB), by heating in the absence of oxygen. Kinetic studies on pyrolysis of this type of biomass (sugar cane bagasse, rice husk, and empty oil palm fruit bunches) were carried out using the thermogravimetric method. The Coats-Redfern method was used in this study. The purpose of this study is to obtain the most appropriate reaction kinetics model to represent the pyrolysis process for each type of biomass. In addition, to determine the optimal temperature used in forming bio-oil. Approximately 5 g of each biomass is used with a heating rate of 10°C/minute. Pyrolysis was carried out until the temperature reached 750°C. The results of the research on the selected kinetic model for each biomass is a geometric model with a correlation coefficient (R^2) close to 1 and the optimum temperature for producing bio-oil is around 550 - 600°C.

INTRODUCTION

As the population increases, energy consumption also increases. The increase in population is certainly directly proportional to energy needs due to high needs and mobility. Energy consumption in Southeast Asia almost doubled between 1995 and 2015, with an average rate of growth of 3.4% annually (Yana et al., 2021). In Southeast Asian countries, the fastest growing ones are Brunei Darusalam, Kamboja, and Vietnam. Indonesia, Malaysia, Thailand and Vietnam accounted for the majority of total final energy consumption (TFEC) in 2015 (IRENA, 2018). Conventional energy such as oil and gas is very limited and cannot be regenerated which will gradually decrease in number and have a bad impact on the environment. With this increase, it can encourage to find alternative energy sources. This is also in accordance with the government's policy contained in Law No. 30 that to realize the security of domestic energy supply, it is expected that by 2025 the role of new and renewable energy will be at least 23% of the national energy source mix (Yanti & Suarno, 2022). Biomass is an interesting alternative because it is easy to obtain locally in large quantities and relatively cheap (Dewayanto et al., 2014). Biomass can be used to provide a variety of energy, whether electric, heat or vehicle fuel. Biomass energy potential comes from seven commodities such as the forestry, agriculture and plantation sectors.

In producing energy derived from biomass can be obtained by various methods including centrifugation, coagulation, flocculation, filtration, flotation, and sedimentation (Barros et al., 2015). According to (Hutasoit et al., 2020)) in harvesting micro algae that will produce biomass is done by means of using low energy and and the floc produced is greater than others so that the separation of deposits is greater. Flocculations are also divided into various types, namely autoflocculation, physical flocculation, chemical flocculation, bioflocculation and natural flocculation. In addition to the previously mentioned methods, such a thermochemical technology as pyrolysis is quite promising in converting biomass. Pyrolysis technique is a thermochemical decomposition process that converts organic matter into charcoal, synthetic gases, biofuels with high temperature and pressure (Singh, 2018). Pyrolysis technique also has the potential to be developed because it has the availability of abundant sources of biomass materials, easy to develop, environmentally friendly and profitable from an economic aspect (Novita et al., 2021).

Pyrolysis of biomass is a complex process because it consists of many decompositions of reactions that take place simultaneously (Gözke, 2022). Therefore, it is necessary to have an approach that has been developed. Kinetic studies on biomass pyrolysis mostly use thermogravimetric techniques (Dewayanto et al., 2014). Thermogravimetric analysis (TGA) is a method used to understand the decomposition and chemical kinetics of biomass (Chilla & Suranani, 2022). Thermogravimetric techniques (TGA) are more convenient for predicting thermals from catalytic and non-catalytic biomass pyrolysis (Balasundram et al., 2017). Biomass pyrolysis in the process is strongly influenced by time, temperature, material moisture content, material size and chemical reactions. The difficulty of this process is also seen in the process of breaking down the chemical structure between solid materials into gaseous and liquid phases.

In determining the kinetic parameters of the pyrolysis results some researchers used a model approach and have not found a consistent model on the whole process (Dewi, 2017b). Quantitative thermal decomposition kinetics models are divided into two types, namely free models and based models. The free model is a method derived from the experimentation of isoconventional integrals at different heating rates using the Arrhenius equation (Sewry & Brown, 2002). The free model consists of the Kissinger-Akahira-Sunose (KAS), Friedman Method (FR), Flynn-Wall-Ozawa (FWO) methods. Previous studies have been conducted on thermal decomposition. On research (Dewi, 2017b) pyrolyzing propellant using the Kissinger-Akahira-Sunose (KAS) method obtained an average E value of 115.282 kJ/mol and an A of 7.261E+08/min on the RUM propellant type. However, using this method has a low R^2 value (below 0.9) compared to the FWO and Coats – Redfern methods. While in the research (Wibowo et al., 2022) pyrolysis on rice husk pellets and produced E of 206-226 kJ/mol and A of 2.07E+19 – 2.38E+19/min. Compared to the KAS and FR Methods, FWO has a small A value and the need to have a heating rate that varies in the FWO method. According to (Dewi, 2017b) that the free model enters into a variable rate so that kinetic parameters are obtained after several tests with different TGA heating rates. Whereas a single heating rate method such as the Coats – Redfern model, only needs to be tested once with a certain TGA heating rate so the value of the kinetic parameter can be known. The integral Coats – Redfren model is quite widely used and serves to describe the parameters of activation energy kinetics in pyrolysis reactions following reaction order kinetics (Majedi et al., 2015). In a research (Raza et al., 2022) that pyrolysis of date palm waste using the Coats – Redfern method produced better and detailed results. Coats – Redfern is more efficient than the previous two methods but more complex because it uses g(x) algebraic expressions so it is more accurate. That's also why Coats – Redfern is referred to as a fittings model. This article presents kinetic studies on catalytic pyrolysis (materials) to describe Coats–Redfern thermogravimetric data and models obtained from pyrolysis.

EXPERIMENTAL DESIGN

Materials

The materials used in this research were sugarcane bagasse, rice husk, and empty fruit bunches (EFB). Sugarcane bagasse was obtained from PTPN XII, Surabaya. The rice husks used were taken from local rice farmers in the area around Ungaran, Semarang. Meanwhile, EFB was

Table 1. Solid Thermal Decomposition Reaction Model Set

Model	Nucleation Model	Reaction model	$g(\alpha)$
1	P1/4	Power Law	$\alpha^{1/4}$
2	P1/3	Power Law	$\alpha^{1/3}$
3	P1/2	Power Law	$\alpha^{1/2}$
4	AE1/4	Avrami-Erofeyev	$[-\ln(1-\alpha)]^{1/4}$
5	AE1/3	Avrami-Erofeyev	$[-\ln(1-\alpha)]^{1/3}$
6	AE/12	Avrami-Erofeyev	$[-\ln(1-\alpha)]^{1/2}$
	Diffusion Model		
7	1DD	One dimensional diffusion	α^2
8	JAN	Diffusion control (Janders)	$[1-(1-\alpha)^{1/3}]^2$
9	CRA	Diffusion control (Crank)	$1-(2/3)\alpha-(1-\alpha)^{2/3}$
	Order Reaction and Geometric Model		
10	N1	Mampel (first order)	$-\ln(1-\alpha)$
11	N2	Second order	$(1-\alpha)^{-1}-1$
12	CC	Contracting cylinder	$1-(1-\alpha)^{1/2}$
13	CS	Contracting sphere	$1-(1-\alpha)^{1/3}$

(Ebrahimi-K & Abbasi, 2008)

obtained from Pangkalanbuun, Central Kalimantan.

Thermogravimetric Analysis

Non-isothermal TGA analysis was performed dynamically from an initial temperature of about 27°C heated to 110°C and kept at that temperature for 30 min to remove *moisture*. Then the sample was reheated to 750°C at a heating rate of 10°C/min under flow of 20 ml/min with a flow rate of N_2 as the gas used to clean the sample in TGA. The thermogravimetric equipment used during the research was the TGA Perkin Elmer 4000 Instrument. The experiment was conducted using 5 grams of samples.

Kinetic Model

The Coats – Redfern method is known as the most widely used method in examining the kinetics of a pyrolysis of biomass. This is because the Coats – Redfern method is more efficient than other methods. This method belongs to the single heating rate method, in which the value of the kinetic parameter can be known only by one test with a certain TGA heating rate (Dewi, 2017a). The basic equation for the Coats – Redfern method is expressed as shown in Eq. (1).

$$\ln\frac{g(a)}{T^2} = \ln\left[\frac{AR}{\beta E_a}\left(1 - \frac{2RT}{E_a}\right)\right] - \frac{E_a}{RT} \qquad (1)$$

Where, g (a) is the kinetic function, T is the temperature, A is the pre-exponential factor, R is

gas constant (0.008314 kJ/mol K), Ea stands for the activation energy and β is the heating rate.

Reaction area (α) or biomass conversion values are used to study the kinetics of reactions in a solid state, given by Eq. (2).

$$\alpha = \frac{m_0 - m_t}{m_0 - m_\infty} \qquad (2)$$

Where m_0, m_t dan m_t each of them is the initial sample mass, the sample mass at the time *t* and the sample mass at the end of the reaction. Equation (1) it is used to obtain the value of the constant *Arrhenius* (A) and the activation energy (Ea). From the equation can also be plotted between $\ln\frac{g(a)}{T^2}$ towards $\frac{1}{T}$, which can produced the *slope* value as activation energy (Ea) and the intercept as the Arrhenius coefficient (A). This equation remains to be examined further using reaction models. A reaction model that shows the value of the correlation coefficient (R^2) close to 1 then the model is better than other model.

Kinetic Parameters to Biomass

Data from the TGA pyrolysis analysis become kinetic parameters a catalytic pyrolysis. Eq. (1) is approached by using reaction models. The first-order reaction (Model no. 10, Table 1 is appropriate to assume the pyrolysis process, so that Eq. (3).

$$ln\frac{-ln(1 - a)}{T^2} = ln\left[\frac{AR}{\beta E_a}\left(1 - \frac{2RT}{E_a}\right)\right] - \frac{E_a}{RT} \quad (3)$$
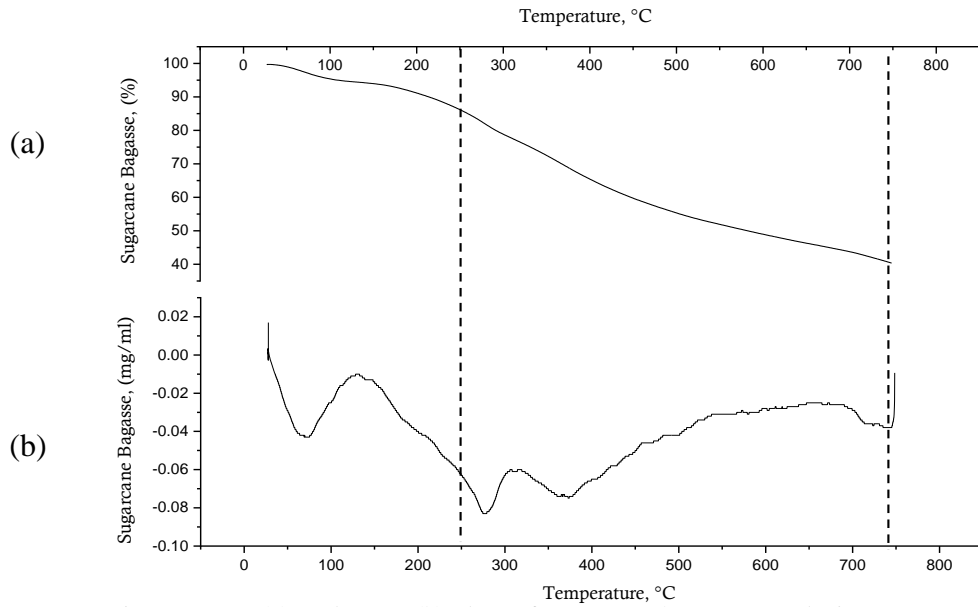
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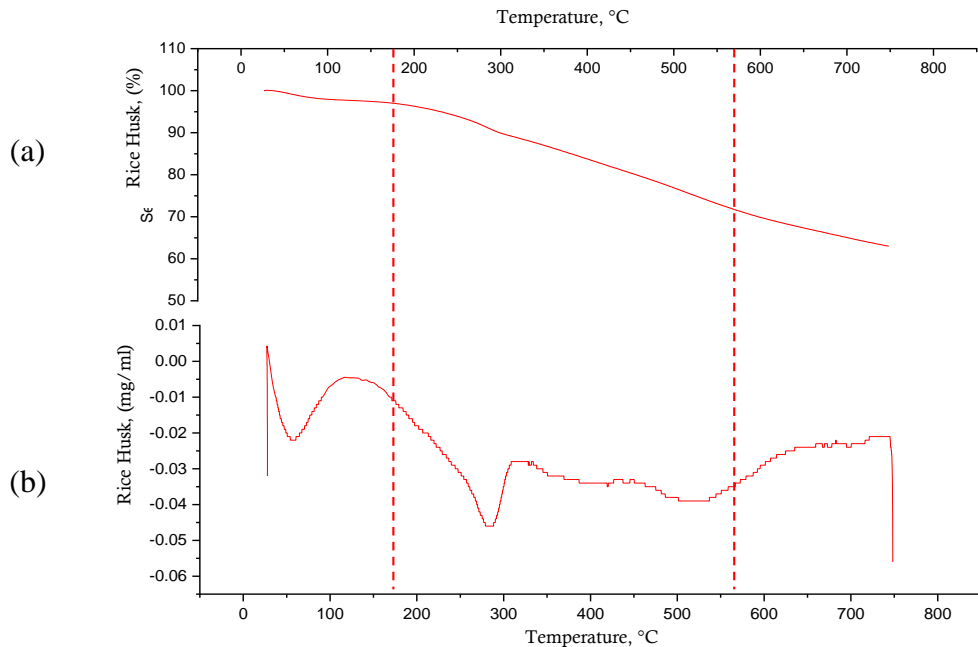Figure 1. TG (a) and DTG (b) plots of sugarcane bagasse pyrolysis.

Figure 2. TG (a) and DTG (b) plots of rice husk pyrolysis.

Because 2RT << 1, hence the Eq. (3) can be simplified to Eq. (4).

$$ln\frac{-ln(1-a)}{T^2} = ln\frac{AR}{\beta E_a} - \frac{E_a}{RT} \qquad (4)$$

According to (Vasconcelos et al., 2014) the better Coats – Redfern model is based on the adjustment of experimental data i.e. the correlation coefficient (R^2) is close to 1. The value of R^2 is derived from the conversion value of the experiment with the calculated conversion value (Dewayanto et al., 2014). The model of the decomposition mechanism is represented by an increasing large value of R^2. This is in line with the opinion of (Boumanchar et al., 2019) that the solution that has the highest correlation coefficient (R^2) will be selected. But there are some kinetics that give unrealistic results, namely the activation energy is negatively valued.

RESULTS AND DISCUSSION

Thermogravimetric Analysis

Figures 1,2, and 3 (a) show the TG plot and (b) show the pyrolysis DTG plots of various biomass materials namely sugarcane bagasse, rice husk, and EFB. This thermal behavior uses a
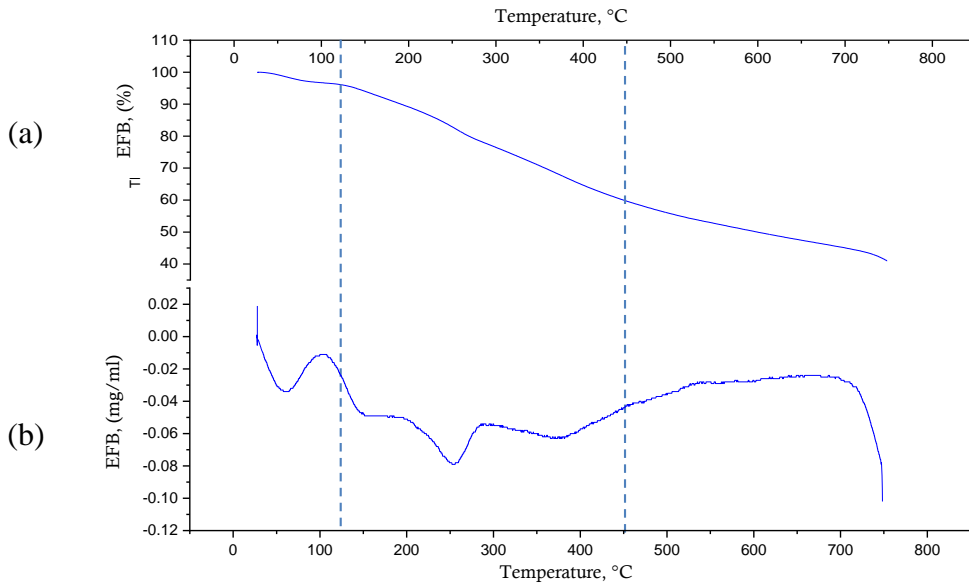
Figure 3. TG (a) and DTG (b) plots of EFB pyrolysis.

nitrogen flow rate with a heating rate of 10 °C / min.

Based on the thermal analysis curve, there are three regions analyzed. In the first stage, namely the drying process, namely the occurrence of slow evaporation of water. The second stage is a devolatilization process characterized by a very rapid decrease in mass. Then the third stage, namely the final combustion process, is characterized by a mass reduction that again slows down and tends to be stable (Subagyono et al., 2021).

Table 2. Stages of Biomass Pyrolysis Process.

Phase	Sugarcane Bagasse (°C)	Rice Husk (°C)	EFB (°C)
First	≤ 62 – 250	≤ 53 – 175	≤ 51 – 120
Second	± 250-750	± 175 – 570	± 120 – 450
Third	≥ 750	≥ 570	≥ 450

The results of the thermogravimetric analysis of each biomass material are different as shown in Table 2. The first stage of each ingredient is going on quickly or slowly. This is because the moisture content and volatile matter contained are different. Bagasse sugarcane runs slower in the first stage (250°C) than other biomass materials due to its higher moisture content and lower amount of volatile matter. In line with research (Alias et al.,

2014) that higher water content usually contributes to a lower amount of volatile matter due to the more heat required to evaporate the water first before biomass decomposition. The content of properties and composition can be seen in the Table 3.

The second stage refers to the successive decomposition of lignocellulose components, in which devolatilization of the lignin, cellulose and hemicellulose components contained in biomass occurs. Hemicellulose and cellulose belong to volatile substances that are easily condensed (Prasetio et al., 2020). Volatile substances contained in EFB are higher by 80.89% compared to other biomass materials, so the devolatilization process takes place faster. Therefore, the greater the volatile substances contained, the faster the devolatilization process takes place.

Devolatilization or mass weight loss is also associated with lignin decomposition, which is known to slowly degrade lignin over large temperature ranges (Mohamed & Hamzah, 2015). (Mohamed & Hamzah, 2015). The structure of lignin consists of a complex network of aromatic compounds, which causes lignin to be difficult to decompose so that it has high thermal stability (Setiawan et al., 2016). In line with the devolatilization process in the longer Bagasse Sugarcane, due to the larger contained lignin makes the lignin degraded run slowly and require a greater temperature. The lignocellulose compositions of the three biomass materials is shown n Table 4.

Table 3. Proximate Analysis and Ultimate Analysis

	Sugarcane Bagasse	Rice Husk	EFB
Proximate Analysis (% mass)			
Moisture Content	10.91 – 49	7.16 - 8.8	2.4 – 5.5
Volatile Matter	42.5 – 71.79	59.2 – 73.14	78.8 - 80.89
Fixed Carbon	7 – 11.33	4.38 - 14.6	12.6 – 13.4
Ash	1.5 – 5.97	15.31 - 26.2	2.3 - 4.02
Ultimate Analysis (% mass)			
C	23.7 – 42.07	35.6 – 46.83	42.6 - 44.72
H	3 – 5.60	3.58 - 4.5	5.7 - 6.58
O	22.8 – 52.01	47.1 - 59.7	39.5 - 42.96
N	0 – 0.26	0.19 – 2.43	0.80 – 1.7
S	0 – 0.06	0.02 – 0.05	0.17 - <0.3
Reference	(Daniyanto et al., 2016); (Hassan et al., 2020)	(Varma & Mondal, 2016); (Gajera et al., 2020)	(Alias et al., 2014); (Yan et al., 2019); (Ro et al., 2018)

Table 4. Lignocellulose Component.

Biomass	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Reference
Sugarcane Bagasse	42.8	27.6	26.4	(Zanatta et al., 2016)
Rice Husk	35	33	23	(Varma & Mondal, 2016)
EFB	57.8	21.2	22.8	(Prasetio et al., 2020)

Biomass Kinetic Study

Kinetic Study of Sugarcane Bagasse

Figure 4 presents a plot between ln g(a)/T^2 against 1/T in sugarcane bagasse. The results of the activation energy calculation from thermogravimetric analysis (TGA) and using the Coats – Redfern method with various kinetic models are shown in Table 5.

That the correlation coefficient (R^2 of stage 1 is in the range of 0.6716 – 0.9795 and stage 2 is in the range of 0.5632 – 0.9772. The Diffusion Control (JAN) kinetics model showed the highest R^2 at stage 1 with an activation energy value of -5.2308 kJ/mol while in stage 2 the Second Order kinetics model (N2) showed the highest R^2 with an Ea value of 23.571021 kJ/mol. However, the JAN kinetics model has an Ea value of less than zero, so it is not the best choice model. Therefore, for phase 1 the selected kinetic model is First Order (N1) with R^2 and Ea values of 0.9586 and 14.43061 kJ/mol, respectively.

Based on research (Jamilatun et al., 2022) the higher the pyrolysis temperature will make the resulting organic phase high, optimum at a

Table 5. Pyrolysis activation energy of Bagasse Sugarcane obtained by various kinetic models.

Model	Stage 1		Stage 2	
	Ea (kJ/mol)	R^2	Ea (kJ/mol)	R^2
Nucleation Model				
P1/4	10.9021	0.671	-4.30108	0.826
P1/3	10.1871	0.789	-4.3593	0.8752
P1/2	10.4166	0.906	-4.3593	0.9188
AE1/4	8.85441	0.710	9.50955	0.5632
AE1/3	10.0732	0.791	9.50955	0.6484
AE1/2	13.4113	0.868	8.831131	0.7751
Diffusion Model				
1DD	39.6976	0.850	4.645780	0.9398
JAN	-5.2308	0.979	-8.511	0.9636
CRA	1.50E-05	0.973	4.16E-06	0.9511
Reaction Order and Geometric Model				
N1	14.4306	0.958	8.841107	0.7097
N2	-4.49030	0.955	23.57102	0.9772
CC	-5.37508	0.893	-9.54946	0.9757
CS	-6.17306	0.890	-10.9379	0.9458

temperature of 550°C with a yield of 6.11%. The temperature increase of 300°C to 550°C made the organic phase yield increase from 3.57% to 6.11%. In addition to temperature that can affect the
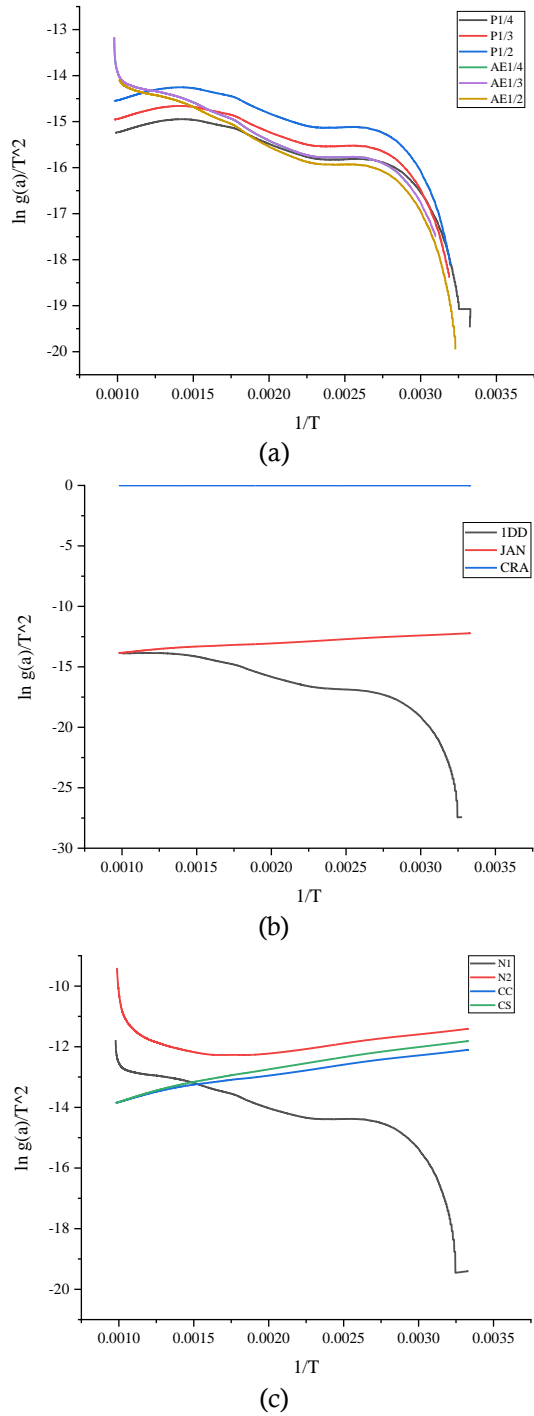
(a)

(b)

(c)

Figure 4. Nucleation model (a), diffusion model (b), and reaction order and geometric model (c) plot linear correlation of sgarcane bagasse pyrolysis between ln $(g(\alpha)/T^2$ against 1/T.

number of biooil yields, namely the influence of particle size. This is in accordance with the results of research (Erawati et al., 2013) that the cause of bio oil drops occurs because the size of the particle diameter is getting smaller making heat easily dispersed throughout the section. The larger the particle size makes the heating slow going, so the

average temperature on the sugarcane bagasse will be lower and result in less yield obtained.

Kinetic Study of Rice Husk

Figure 6 presented plot between ln $g(a)/T^2$ against 1/T on rice husk. The results of the calculation of activation energy from thermogravimetric analysis (TGA) and using the Coats-Redfern method with various kinetic models are shown in Table 6.

Table 6. Pyrolysis activation energy of rice husk obtained by various kinetic models.

Model	Stage 1		Stage 2	
	Ea (kJ/mol)	R^2	Ea (kJ/mol)	R^2
Nucleation Model				
P1/4	9.8778	0.710	0.70062	0.7921
P1/3	9.8778	0.815	0.70062	0.864
P1/2	9.8778	0.926	0.70062	0.9297
AE1/4	10.8805	0.693	16.8458	0.5096
AE1/3	10.8805	0.798	16.8525	0.5187
AE1/2	10.8805	0.913	16.8524	0.5033
Diffusion Model				
1DD	33.7523	0.903	14.760	0.9443
JAN	-5.6627	0.970	-6.4542	0.9965
CRA	-7.3475	0.009	-22.288	0.9475
Reaction Order and Geometric Model				
N1	10.8805	0.986	16.8524	0.6676
N2	11.9488	0.979	46.9225	0.9465
CC	-5.7523	0.963	-7.8401	0.9758
CS	-5.7523	0.949	-7.8401	0.9094

That correlation coefficient (R^2) stage 1 is in the range 0.0094 – 0.9862 and stage 2 is within the range 0.5033 – 0.9965. At stage 1 the First Order kinetic model (N1) shows the highest R^2 value, with an Ea value of 10.880532 kJ/mol. In stage 2 the Diffusion Control (JAN) kinetics model produced the highest R^2 value, however the Ea value was less than zero so the JAN model was not the best model. Therefore, the best kinetic model in stage 2 is N2 because it has an R^2 and Ea values corresponding to the values of 0.9465 and 46.92255 kJ/mol, respectively.

In the pyrolysis process, the main desired product is bio-oil. Bio-oil is a liquid consisting of two phases, namely in the form of oil (oil) and water (water). The amount of oil obtained is influenced by the temperature used. From the results of the experiments conducted (Wijayanti et al., 2020), the formation of a total yield of 3.27% (oil), 26.16% (water), 12.55% (gas), and 58.02% (char) at a temperature of 550 ° C, proves that the higher the

temperature used to eat, the greater the amount of oil produced.
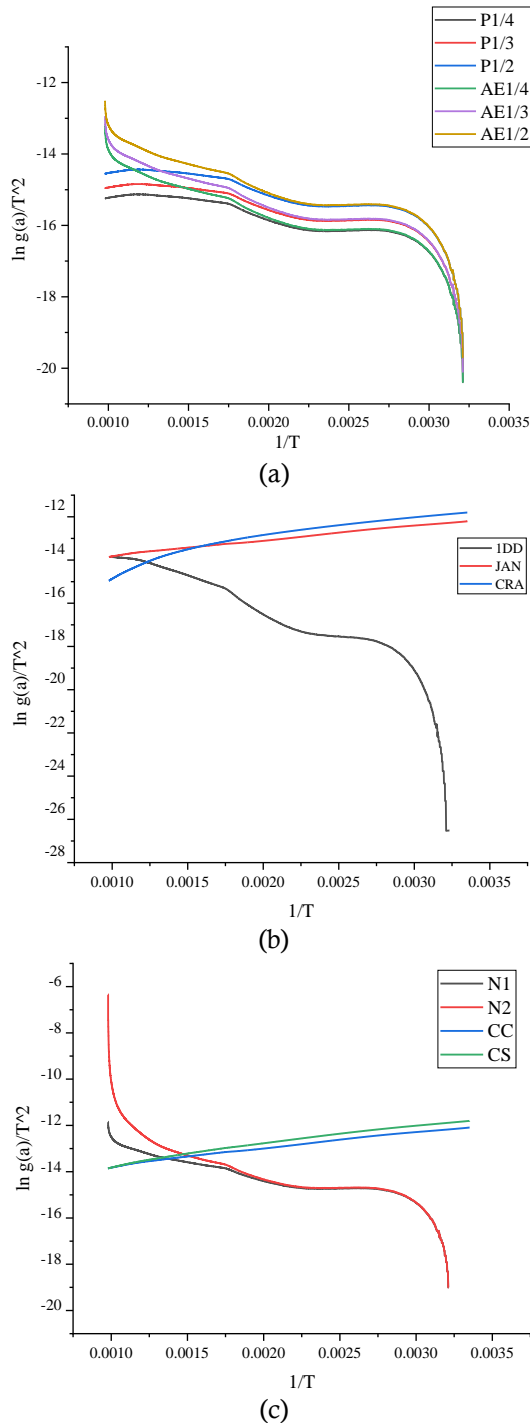

(a)

(b)

(c)

Figure 5. Nucleation model (a), diffusion model (b), and reaction order and geometric model (c) plot linear correlation of rice husk pyrolysis between ln $(g(\alpha)/T^2$ against $1/T$.

Kinetic Study of Empty Fruit Bunch (EFB)

Figure 6 Presented a plot between ln g(a)/T² towards 1/T on EFB. The results of the

activation energy calculation from thermogravimetric analysis (TGA) and using the Coats-Redfern method with various kinetic models are shown in Table 7.

Table 7. Pyrolysis activation energy of empty fruit bunch obtained by various kinetic models.

Model	Stage 1		Stage 2	
	Ea (kJ/mol)	R^2	Ea (kJ/mol)	R^2
Nucleation Model				
P1/4	15.8897	0.5121	-5.2531	0.855
P1/3	15.8897	0.6203	-5.2531	0.8958
P1/2	15.8897	0.9322	-5.2531	0.7733
AE1/4	17.450	0.4907	6.9806	0.6318
AE1/3	14.6634	0.637	6.57563	0.6638
AE1/2	14.8895	0.7708	6.3938	0.6605
Diffusion Model				
1DD	38.9594	0.7908	3.7748	0.9513
JAN	-4.9268	0.9943	-8.9508	0.9637
CRA	1.50E-05	0.9893	4.99E-06	0.9591
Reaction Order and Geometric Model				
N1	17.4502	0.8775	6.9806	0.7598
N2	19.1671	0.9967	30.0443	0.9699
CC	-5.1066	0.9912	-9.8995	0.974
CS	-6.0167	0.9512	-11.1557	0.9309

The best model in stage 1 is the Second Order kinetics (N2) model which produces an R^2 value of 0.9967 with an Ea value of 19.1671 kJ/mol. Stage 2 of the kinetic model that produces the highest R^2 value is the Contracting Cylinder (CC) but has an Ea value of less than zero. In stage 2, the one with the highest R^2 value is the Second Order (N2) of 0.9699 and the Ea value of 30.0443 kJ/mol. So that the best model for EFB at both stages is the Second Order kinetics model (N2).

EFB pyrolysis produces liquid in the form of a mixture of liquid product and oil phase. This oil phase is also commonly referred to as bio-oil. Based on research (Ferdiyanto et al., 2020) obtaining bio-oil with yields ranging from 6.94% - 9.96%, with the highest yield using temperature 600°C and the lowest yield at temperature 450°C. This is because the higher the pyrolysis temperature used, the more biomass substances such as cellulose, hemicellulose, and lignin will be decomposed.

CONCLUSION

Thermal decomposition of biomass (bagasse sugarcane, rice husk, empty fruit bunches) occurs in the temperature range 27 until 750°C. Pyrolysis kinetics are successfully modeled using
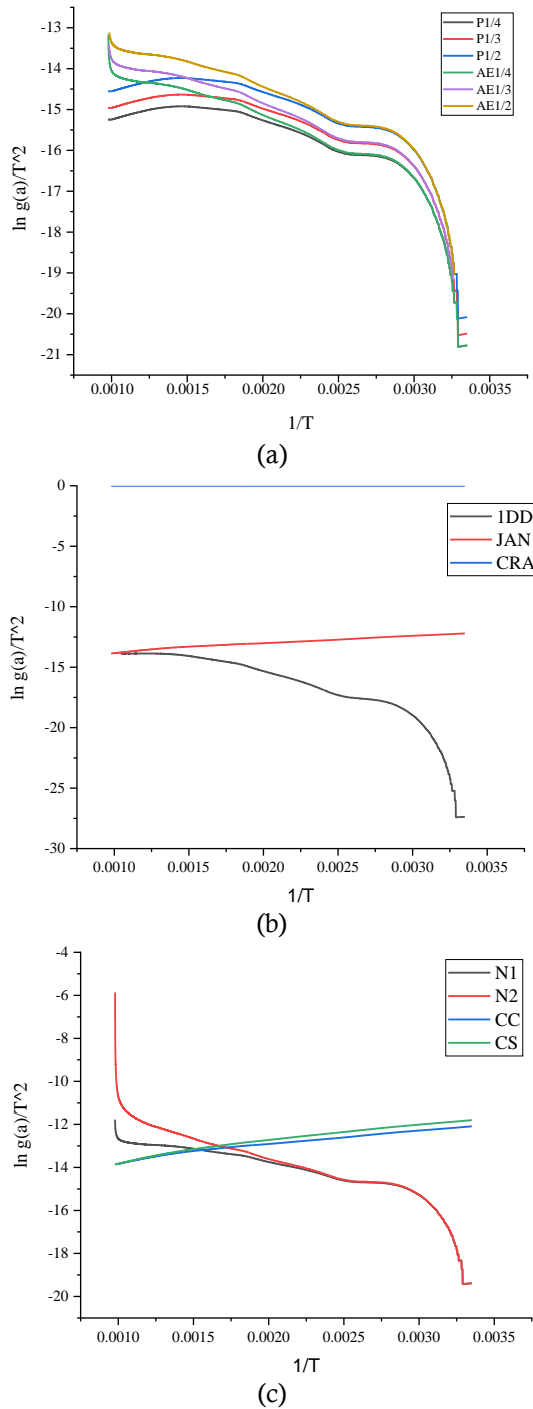
(a)

(b)

(c)

Figure 6. Nucleation model (a), diffusion model (b), and reaction order and geometric model (c) plot linear correlation of empty fruit bunch pyrolysis between ln $(g(\alpha)/T^2$ against $1/T$.

the Coats-Redfern method or also called the fittings model. Two stages of reaction with stage 1 (25-361°C) and stage 2 (361-750°C) identified during the main thermal decomposition of the substrate. Various reaction models are installed to be applied to the reactions of the first and second stages, respectively. In sugarcane bagasse the best model is the Order Reaction model. First Order Model (N1) at stage 1 that has value R^2 0.9586 and in stage 2 is a Second Order model which has value R^2 0.9772. The results of simulating reaction kinetics on rice husks with the best models, namely the First Order (N1) and Second Order (N2) models respectively in stage 1 and stage 2. The value of R^2 in stage 1 is 0.9862 and stage 2 is 0.9465. In empty fruit bunch (EFB) at both stages choose the Second Order (N2) model. In stage 1 it shows an R^2 of 0.9967, while in stage 2 it is 0.9699. Thus, it can be concluded that the best kinetic models are the First Order (N1) and the Second Order (N2) which are forms of the Reaction Order model. Increasing temperature affects the *yield* that produces bio-oil. The higher the temperature used, the greater the amount of bio-oil produced. Based on these 3 materials, the optimum temperature that can produce the highest amount of bio-oil is around 550 - 600 °C.

ACKNOWLEDGEMENTS

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