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Kinetic Study on Palm Oil Waste Decomposition

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1. Introduction

Malaysia is the largest producer of palm oil and contributes 43% of worldwide production (Shuit et al., 2009). Beside palm oil, palm oil industry generated 169.72 million metric tons solid wastes which contribute 85.5% of total biomass waste produced in the country (Khan et al., 2010). This huge amount of wastes can be converted into valuable chemical feed stocks and fuels due to environmental problems associated with conventional fossil fuels.

It is well known that lignocellulosic biomass mainly consists of hemicellulose, cellulose and lignin. The usual proportions (wt%) vary as 40-50% cellulose, 20-60% hemicellulose and 10-25% lignin (Yang et al., 2007). The thermal decomposition of these individuals is important since they influence the basics of thermochemical conversion processes such as pyrolysis, combustion and gasification. Decomposition of these components is intensively studied in the literature. Demirbas et al. (2001) observed the ease of lignocellulosic biomass components decomposition as hemicellulose > cellulose >>> lignin. Based on different reasoning, Yang et al. (2007) proposed different decomposition regions of 220-300 °C, 300-340 °C and >340 °C for hemicellulose, cellulose and lignin, respectively. Lignin is the last to decompose due to its heavy cross linked structure (Guo & Lua, 2001).

Several techniques are available to study the kinetics of biomass decomposition. Among these, thermogravimetric analysis (TGA) is the most popular and simplest technique (Luangkiattikhun et al., 2008), based on the observation of sample mass loss against time or temperature at a specific heating rate. TGA provides high precision (Várhegyi et al., 2009), fast rate data collection and high repeatability (Yang et al., 2004) under well defined kinetic control region.

Very few attempts have been carried out to study the kinetics of empty fruit bunch (EFB) and palm shell (PS) using TGA. Guo & Lua (2001) presented the effect of sample particle size and heating rate on pyrolysis process and kinetic parameters for PS. They concluded a first order reaction mechanism for the decomposition of PS at different heating rates. They also suggested higher heating rates for faster and easy thermal decomposition of PS. Yang et al. (2004) studied activation energy for decompositions of hemicellulose and cellulose in EFB and PS by considering different temperature region for first order kinetic reaction. They evaluated average activation energy and pre-exponential factor from single-step decompositions of hemicellulose and cellulose. Luangkiattikhun et al. (2008) considered the

effect of heating rate and sample particle size on the thermogram behaviour and kinetic parameters for palm oil shell, fibre and kernel. They observed that there is no significant effect of particle size on the thermogram behaviour at lower temperature i.e. <320 °C for palm oil shell. They further proposed nth order reaction mechanism to evaluate the kinetic parameters based on different models.

Previous works reported on EFB and PS kinetics were based on single heating rate in which activation energy is only a function of temperature. The present work evaluate the kinetic parameters based on a method, which requires at least three sets of experimental data generated at different heating rates. This method allows the dependence of activation energy on temperature and conversion at a desired heating rate (Vyazovkin & Wight, 1999) Secondly, lignin decomposition in EFB and PS is not intensively studied at relatively high heating rates. Present work considers lignin decomposition in EFB and PS to understand the effect of lignin content on kinetic parameters and decomposition rate. Furthermore, pure lignin decomposition is studied based on its thermogram analysis and kinetic parameters.

In this work, the kinetics of biomass decomposition which includes EFB, PS, pure cellulose and lignin were investigated using TGA under non-isothermal conditions. The detail thermogram analysis was presented to understand the decomposition of cellulose, hemicellulose and lignin as major components in lignocellulosic biomass. The decomposition kinetics of cellulose and lignin were studied under single-step first order kinetic model. Meanwhile, the decomposition of EFB and PS were reported based on single-step nth order kinetic model. Activation energy, pre-exponential factor and order of reaction were determined and discussed in comparison to the values reported in the literature.

2. Materials and methods

2.1 Materials preparation and experimental procedure

Cellulose in fibrous powder form and lignin in brown alkali powder form were purchased from Sigma Aldrich Sdn. Bhd., Malaysia. EFB and PS were collected from local palm oil industry in Perak, Malaysia. Biomass samples were dried at 105°C and the weighted was monitored at one hr interval, until the readings became constant. Samples were then grinded to particle size of 150-250µm. The method for drying, characterization and analysis were given in previous work (Abdullah et al., 2010). The biomass, pure cellulose and lignin properties are given in Tables 1 and 2.

The biomass decomposition experiments were carried out in EXSTAR TG/DTA 6300 (SII, Japan). N₂ was used as inert gas with a constant flow rate of 100 ml/min for the entire range of experiments. The sample initial weight used in all experiments was within the range of 3-6 mg. TG experiments were performed at heating rate of 10, 30 and 50 °C/min. All samples were first heated from 50 °C to 150 °C where it was kept constant for 10 min to remove moisture content, and then heated up to the final temperature of 800 °C. All experiments were carried twice for reproducibility. No significant variations were observed in the second experimental measurements.

2.2 Kinetic parameters determination

The biomass decomposition rate under non-isothermal condition is described (Cai & Bi, 2009).

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

$f(\alpha)$ depends on the reaction mechanism as listed in Table 3 and α is the mass fraction reacted.

$$\alpha = \frac{w_0 - w}{w_0 - w_f} \quad (2)$$

Where, 0 and f shows initial and final sample weight

$$g(\alpha) = \int_0^\alpha \frac{d\alpha}{f(\alpha)} = \frac{A}{\beta} \int_0^\alpha \exp\left(-\frac{E}{RT}\right) dT = \frac{AE}{\beta R} p\left(\frac{E}{RT}\right) \quad (3)$$

$p(E/RT)$ function has no exact analytical solution, and therefore different approximations are reported to evaluate the function (Budrugeac et al., 2000). The method developed by Flynn-Wall (Flynn & Wall, 1996), Ozawa (1965) using Doyle's approximation (1961) is the most popular and commonly used by several researchers for biomass decomposition (Cai & Bi, 2009; Hu et al., 2007; Zhouling et al., 2009).

$$p\left(\frac{E}{RT}\right) = 0.0048e^{-1.0516\left(\frac{E}{RT}\right)} \quad (4)$$

(3) is then rearranged for β

$$\ln\beta_i = \ln\left(\frac{A_\alpha E_\alpha}{R g(\alpha)}\right) - 5.331 - 1.0516 \frac{E_\alpha}{RT_{\alpha,i}} \quad (5)$$

Where; w = sample weight (mg); β = heating rate (K/min); R = universal gas constant (8.314×10^{-3} kJ mol⁻¹ K⁻¹)

To determine the activation energy, $\ln\beta_i$ vs. $1/T_{\alpha,i}$ is plotted for different α values and heating rates (i) to give a straight line and the slope of which gives the activation energy (Doyle, 1961; Ozawa, 1965; Zsakó, & Zsakó, 1980; Flynn & Wall, 1996).

Analysis	EFB	PS
Volatiles	84.61	81.03
Ash	5.50	4.10
Fixed Carbon (by difference)	9.89	14.87
C	40.73	49.65
H	5.75	6.13
N	1.40	0.41
S	0.22	0.48
O (by difference)	51.90	43.33
Cellulose ^a	38.30	20.8
Hemicellulose ^a	35.30	22.7
Lignin ^a	22.10	50.7

^a Kelly-Yong et al. (2007)

Table 1. Biomass analysis (wt% dry basis)

Analysis	Cellulose	Lignin
C	43.09	47.71
H	5.96	4.53
N	0.13	0.04
S	0.14	4.24
O (by difference)	50.67	43.40

Table 2. Pure cellulose and Lignin properties (wt % dry ash free)

Function	$f(\alpha)$	$g(\alpha)$
First order reaction	$1-\alpha$	$-\ln(1-\alpha)$
Second order reaction	$(1-\alpha)^2$	$(1-\alpha)^{-1}-1$
Third order reaction	$1/2(1-\alpha)^3$	$(1-\alpha)^{-2}-1$
nth order reaction	$(1-\alpha)$	$1-(1-\alpha)^{1-n}/1-n$

Table 3. Different $f(a)$ and $g(a)$ values based on kinetic control regime (Ahmad et al., 2009)

2.3 Model for kinetic parameter determination

The following assumptions are considered for the decomposition of EFB, PS, pure cellulose and lignin.

- Reaction is purely kinetic controlled.
- The decompositions follow single-step processes.
- First order reaction kinetics is considered for pure cellulose and lignin and PS and EFB kinetics are assumed to be nth order.
- No secondary reaction takes place among the gaseous products.

3. Results and discussions

3.1 Thermogram analysis

The TG and DTG curves for cellulose, lignin, EFB and PS at different heating rates are shown in Figures 1-4. The effect of different heating rate can be described by a lateral shift appeared at high heating rates. These lateral shifts are due to the thermal lag effect between surrounding and biomass particles (Yang et al., 2004; Luangkiattikhun et al., 2007). As a result, conversions are delayed at high heating rates. Thermal lag effect is due to the small heat conductive property of biomass particles (Zhang et al., 2006).

In the DTG curves (Fig. 1-4, b) for all samples, high decomposition rate was observed at 50 °C/min, which shows the increase of thermal decomposition rate of biomass at high heating rates.

The investigated EFB exhibited the decomposition rate corresponds to -41 wt%/min which is higher than -33 wt%/min of PS at 50 °C/min (see Fig. 5). The high decomposition rate for EFB and PS appeared at 342 and 382 °C, respectively. It is important to consider that 60 wt% of EFB and PS is decomposed at 400 and 429 °C for 50 °C/min. These results depict relatively easy and fast decomposition for EFB as compared to PS. This fast decomposition of EFB may be attributed to the comparatively high volatiles matter and low lignin content present in EFB as compared to PS. Conversely, pure cellulose and lignin decomposition rate

is the highest and lowest among all species which is -124 and -19 wt%/min at 50 °C/min, respectively. Furthermore, the highest decomposition rate for cellulose and lignin is observed at 386 and 418 °C.

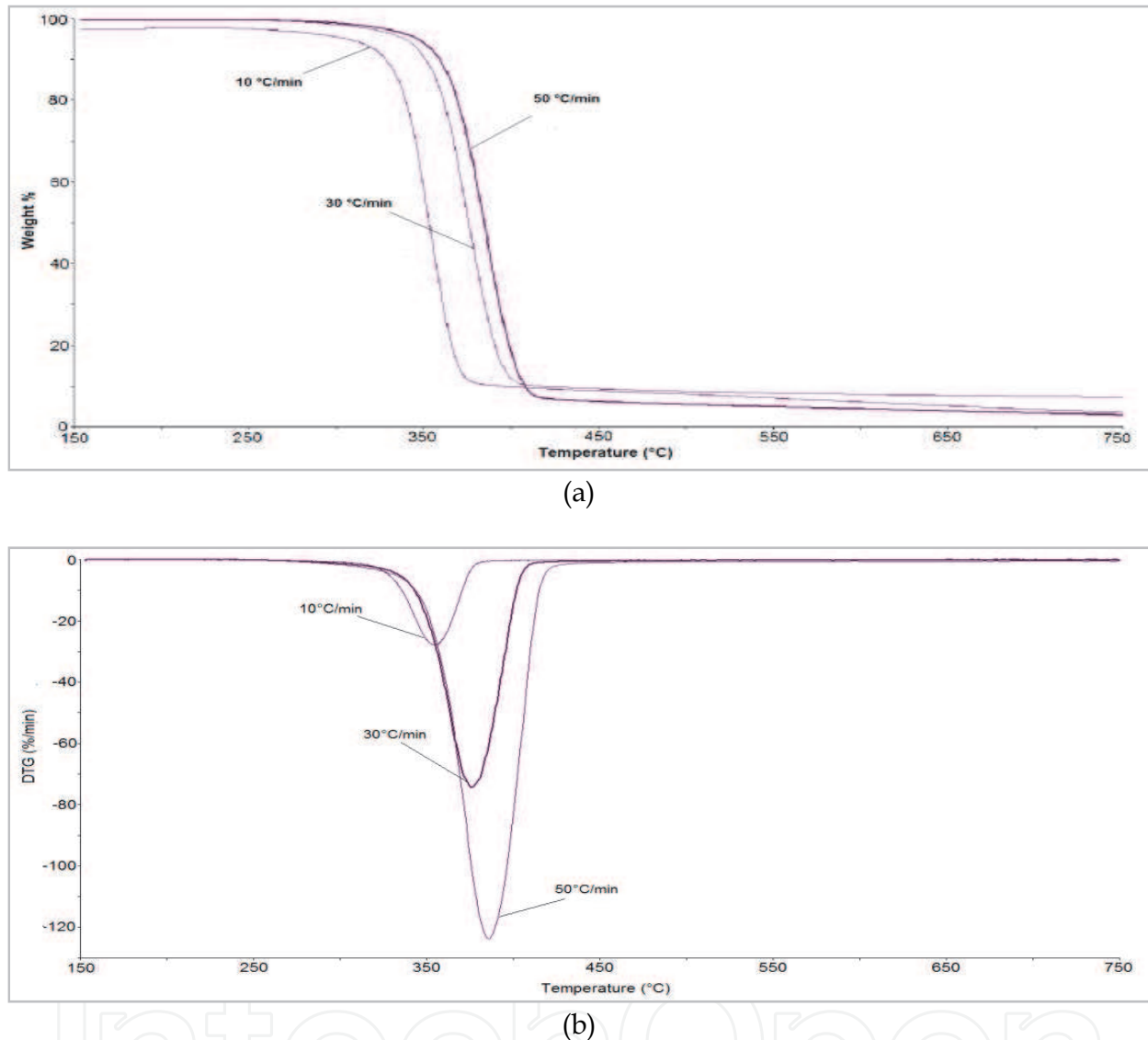
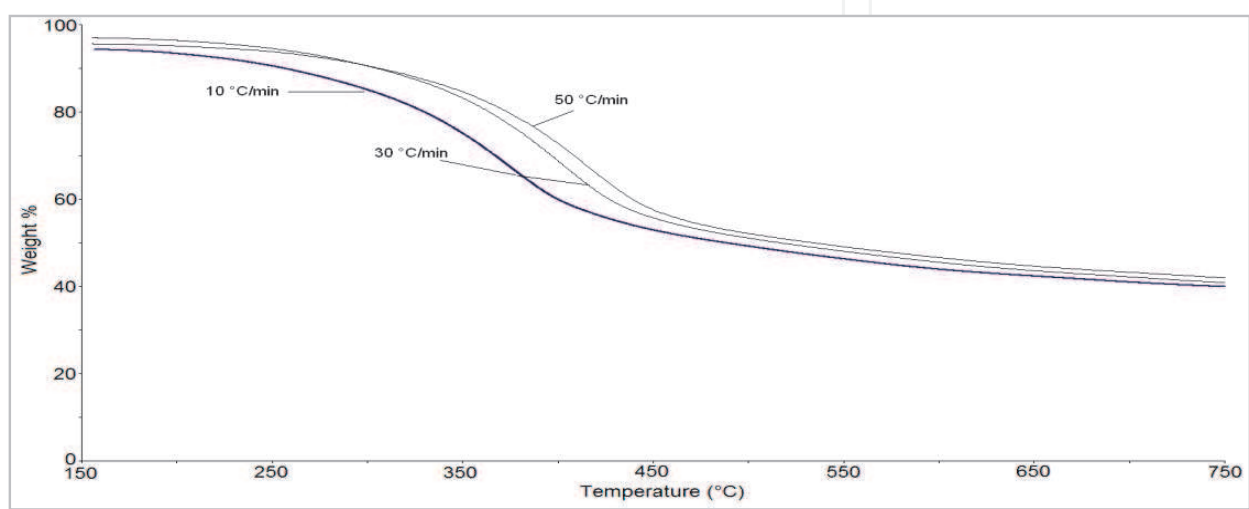


Fig. 1. Cellulose (a) TG and (b) DTG curves

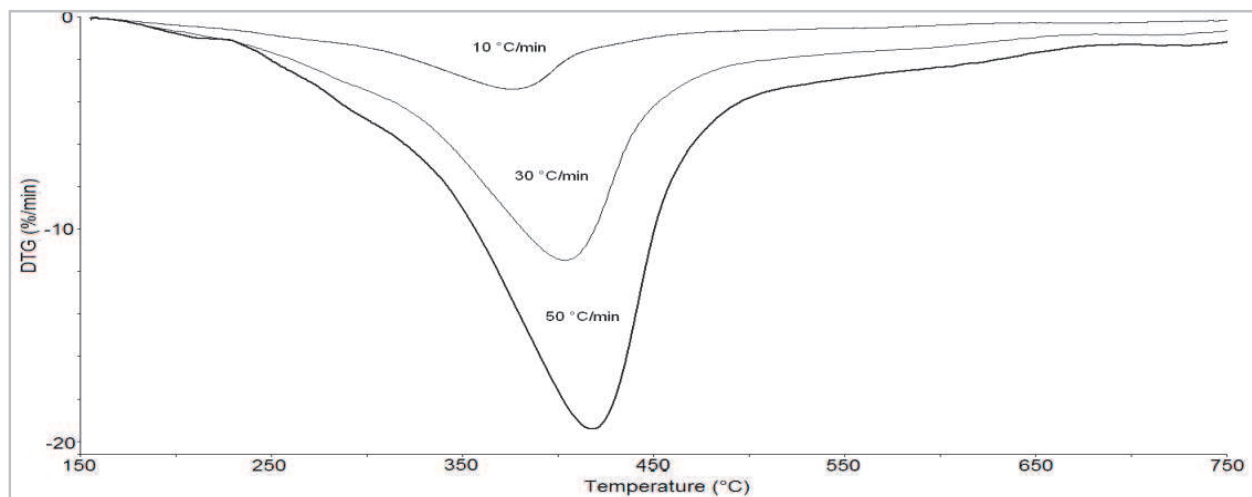
The TG and DTG curves for EFB and PS are given in Figures 3-4. In these figures, the first peak represents the decomposition of hemicellulose. The second peak, which is sharper, gives the highest rate corresponds to the cellulose decomposition. The decomposition range of hemicellulose and cellulose of EFB is between 240-300 °C and 300-340 °C, respectively, at heating rate of 10 °C/min. Decomposition rate of hemicellulose in PS falls almost in the same temperature region as for EFB but higher decomposition range for cellulose (340-370 °C). It is important to consider that the cellulose decomposition rate in PS is in the same temperature region as pure cellulose (340-370 °C at 10 °C/min). The tail at high temperature shows lignin decomposition as found by Yang et al. (2004) and Luangkiattikhun et al. (2008). In the present study, at 10 °C/min, no lignin decomposition was observed for EFB and PS. Similar observation is reported by Yang et al. (2004) for heating rate of 10 °C/min at

temperature >340 °C. At higher heating rates, there is some small lignin decomposition observed for EFB and PS which is in the range of 450-530 °C and 680-750 °C, respectively. Different region for lignin decomposition in EFB and PS may be due to different lignin structure and composition in both species.

Among all species, lignin decomposition produced highest residual fraction of $\sim 40\%$ followed by $\sim 27\%$ of EFB and PS and $<7\%$ for cellulose, respectively. High residual fraction for lignin shows its high resistance to thermal decomposition which can be seen by its lowest decomposition rate.

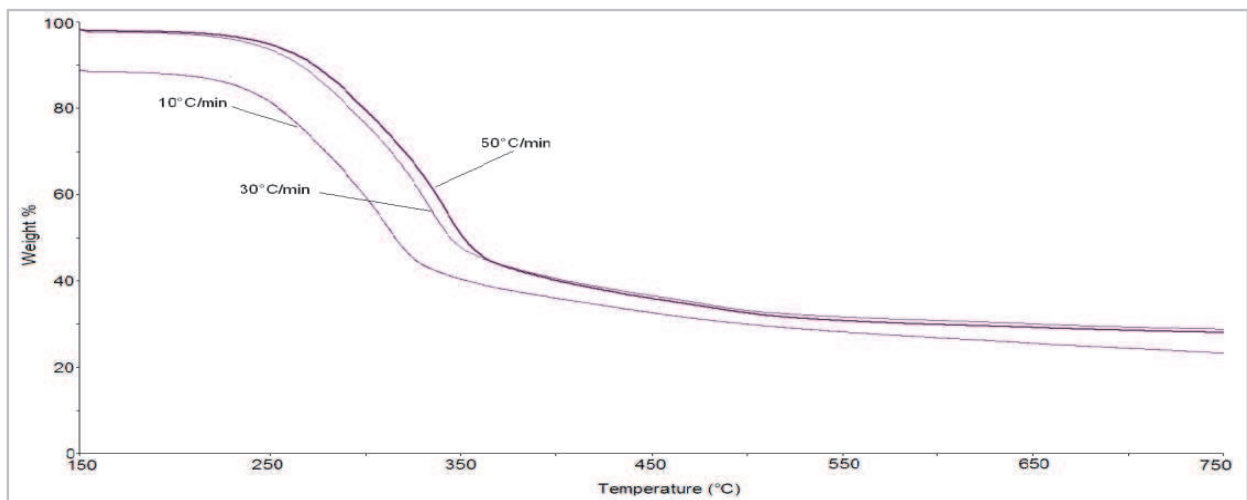


(a)

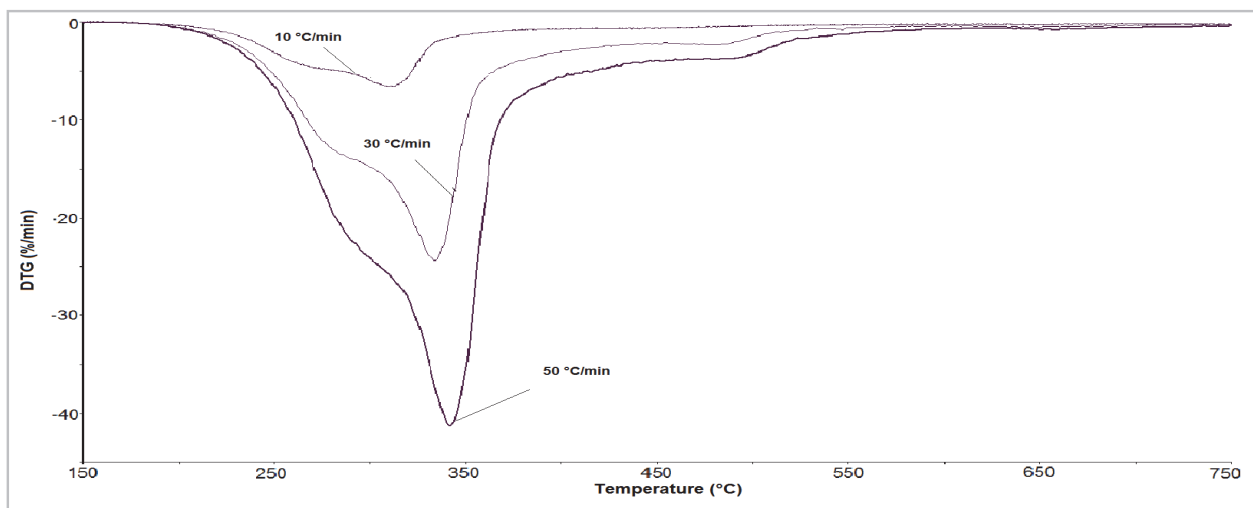


(b)

Fig. 2. Lignin (a) TG and (b) DTG curves

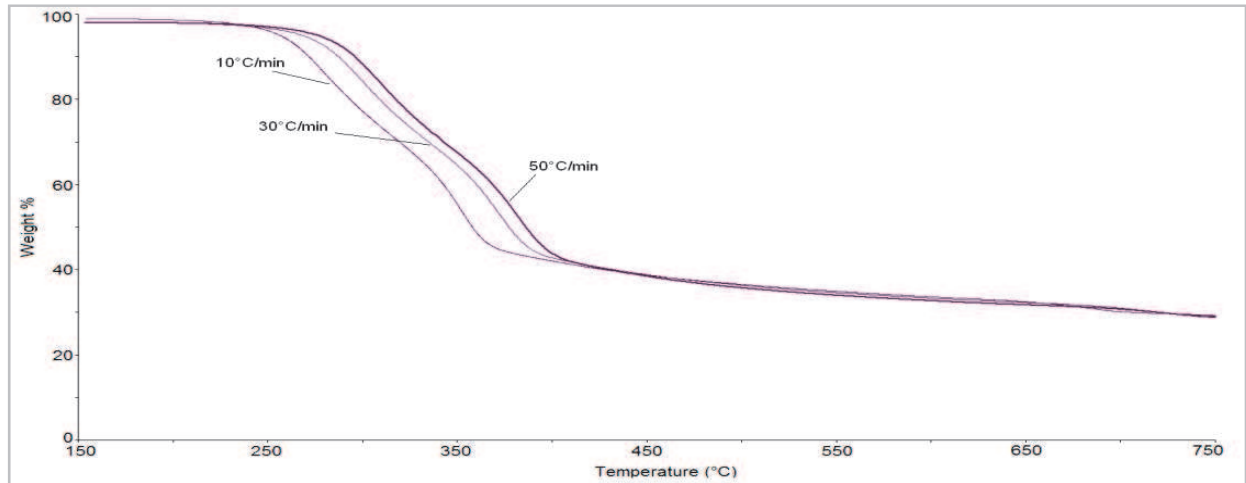


(a)

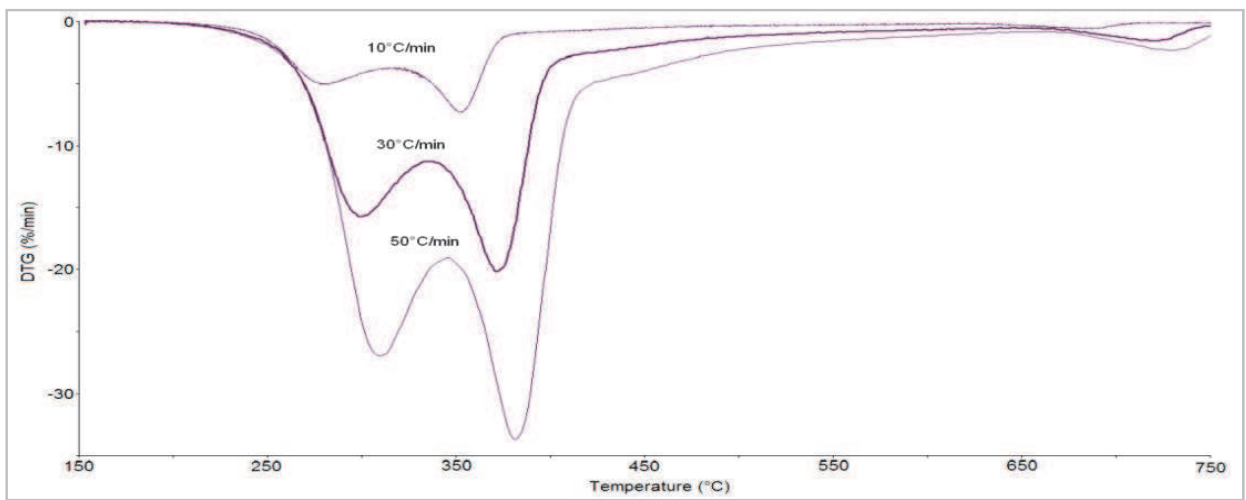


(b)

Fig. 3. EFB (a) TG and (b) DTG curves



(a)



(b)

Fig. 4. PS (a) TG and (b) DTG curves

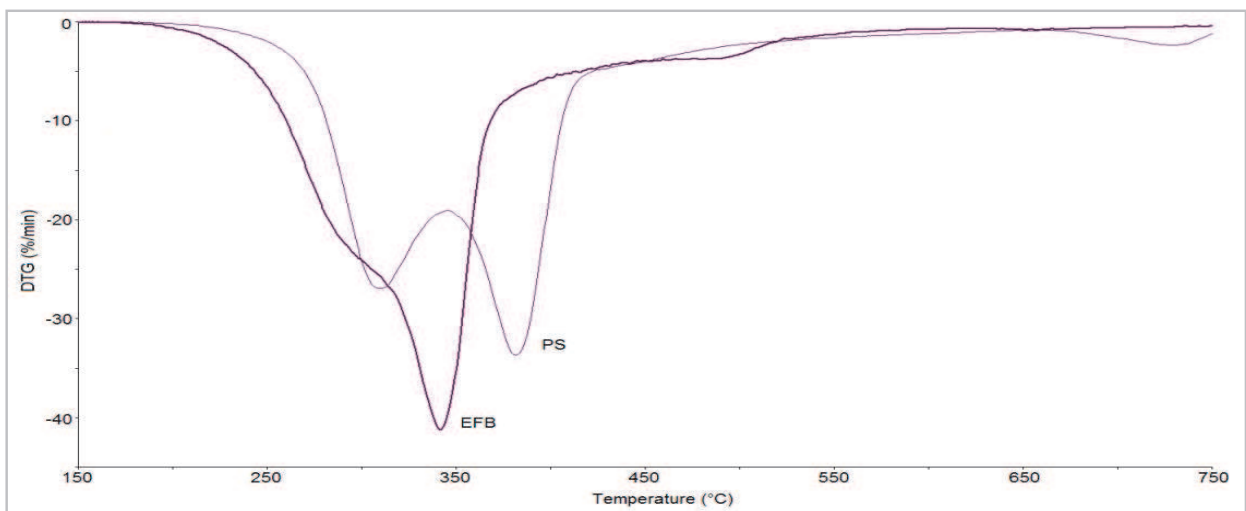


Fig. 5. EFB and PS DTG curves at 50 °C/min

3.2 Kinetic parameters

Kinetic parameters are evaluated using equation (5) at a heating rate of 10, 30 and 50 °C/min. Figures 6-9 shows the plot of $\ln\beta_i$ against $1/T$ at different mass fraction reacted (α) for cellulose, lignin, EFB and PS. The higher $\ln\beta_i$ values shows high heating rate (50 °C/min) followed by 30 and 10 °C/min. Kinetic parameters evaluated at each α are given in the Tables 4-7. For pure cellulose and lignin, the kinetic parameters are determined at $\alpha=0.1$ to 0.8 and $\alpha=0.1$ to 0.6, respectively. EFB and PS kinetic parameters are evaluated at $\alpha=0.1$ to 0.7. Among all samples, pure lignin produced highest residual fraction and hence kinetic parameter determined up to $\alpha=0.6$. The correlation coefficients (R^2) determined are higher than 0.991 for all cases.

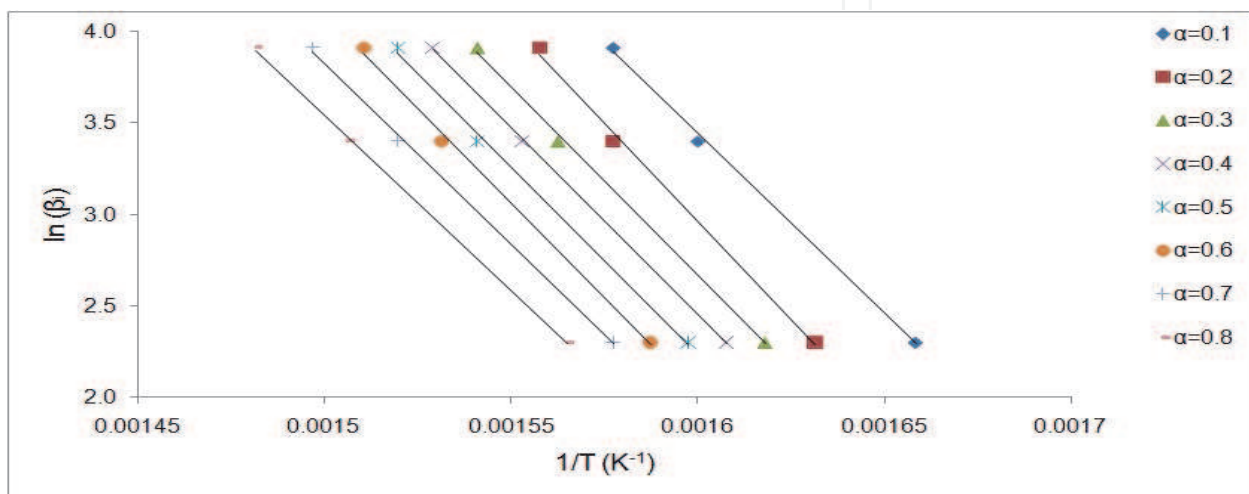


Fig. 6. The dependence of $\ln(\beta_i)$ on $1/T$ at a different α values of cellulose (solid lines shows linear fitting)

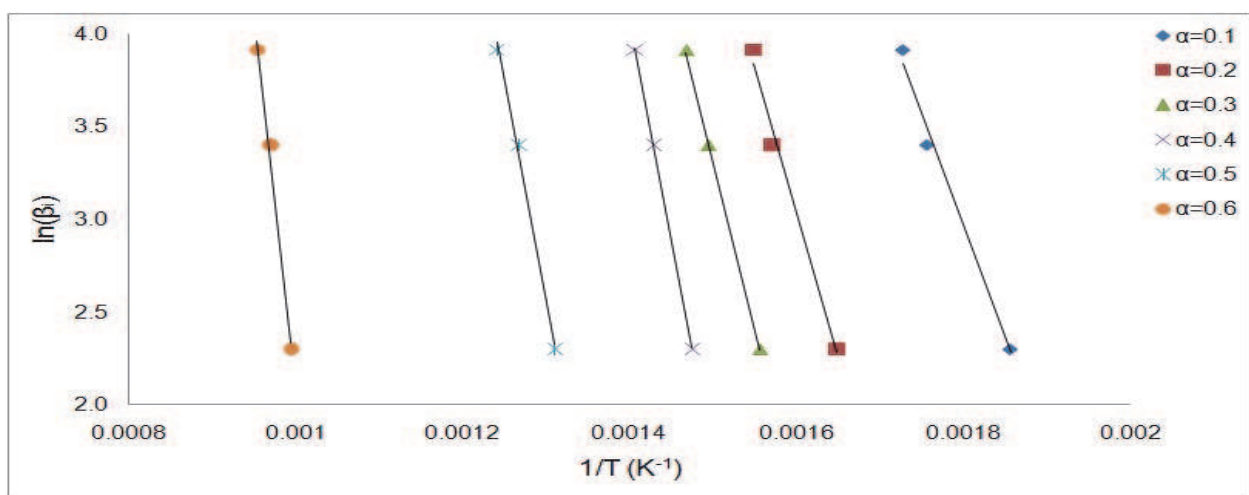


Fig. 7. The dependence of $\ln(\beta_i)$ on $1/T$ at a different α values of lignin (solid lines shows linear fitting)

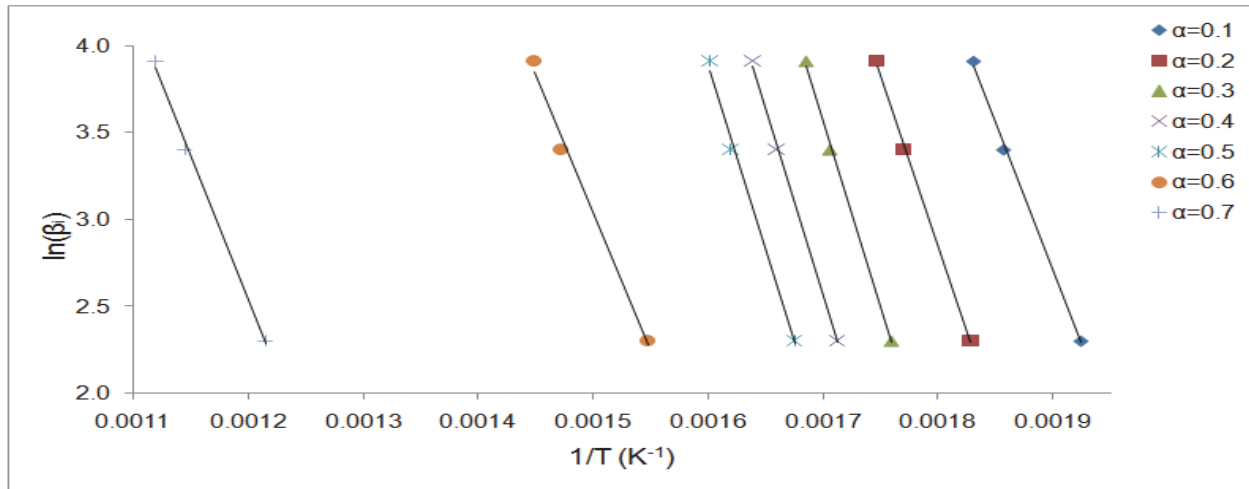


Fig. 8. The dependence of $\ln(\beta_i)$ on $1/T$ at a different α values of EFB (solid lines shows linear fitting)

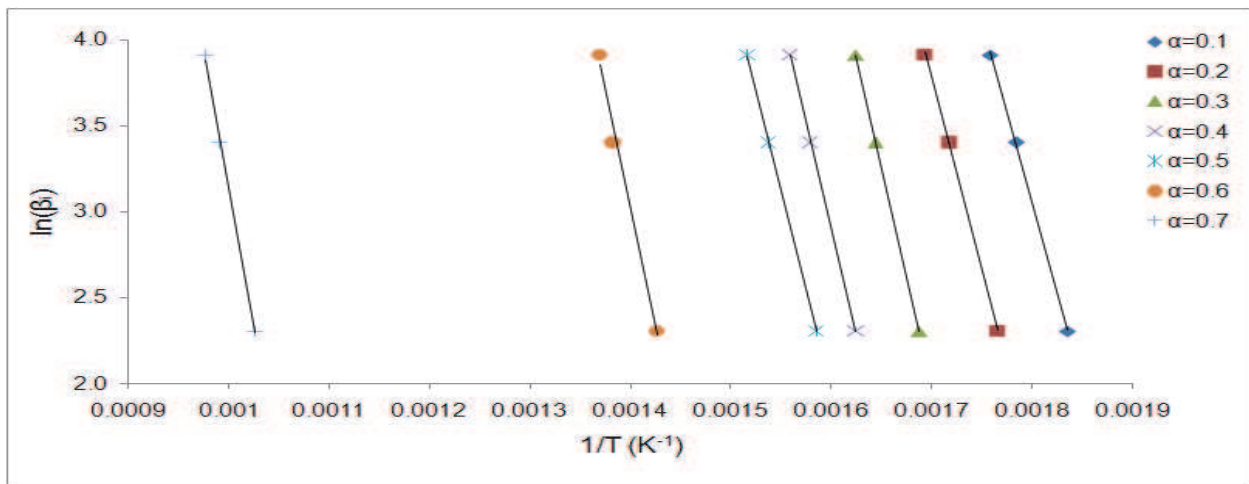


Fig. 9. The dependence of $\ln(\beta_i)$ on $1/T$ at a different α values of EFB (solid lines shows linear fitting)

α	E (kJ/mol)	R ²	A (m ⁻¹)
0.1	155.31	0.996	3.9×10^{13}
0.2	170.14	0.996	3.9×10^{13}
0.3	162.95	0.998	1.1×10^{13}
0.4	161.12	0.999	9.2×10^{12}
0.5	161.64	0.997	1.1×10^{13}
0.6	163.66	0.997	1.8×10^{13}
0.7	157.03	0.998	5.3×10^{12}
0.8	151.53	0.991	1.9×10^{13}
Average	160	0.997	1.2×10^{13}

Table 4. Kinetic parameters of cellulose at different α values

α	E (kJ/mol)	R ²	A (m ⁻¹)
0.1	95.01	0.990	9.1E+10 ⁷
0.2	123.28	0.990	4.4E+10 ⁹
0.3	143.67	0.999	7.9E+10 ¹⁰
0.4	183.89	0.999	3.8E+10 ¹³
0.5	184.22	0.995	1.2E+10 ¹²
0.6	318.48	0.992	1.2E+10 ¹⁶
Average	175	0.994	2.06E+10 ¹⁵

Table 5. Kinetic parameters of lignin at different α values

α	E (kJ/mol)	R ²	A (m ⁻¹)
0.1	135.88	0.998	3.9×10 ¹³
0.2	154.59	0.996	6.0×10 ¹⁴
0.3	169.01	0.998	4.5×10 ¹⁵
0.4	171.96	0.998	4.7×10 ¹⁵
0.5	169.92	0.993	9.1×10 ¹³
0.6	125.60	0.997	9.4×10 ¹⁰
0.7	131.69	0.997	4.0×10 ⁹
Average	151	0.997	1.4×10 ¹⁵

Table 6. Kinetic parameters of EFB at different α values

α	E (KJ/mol)	R ²	A (m ⁻¹)
0.1	165.20	0.999	6.5×10 ¹⁵
0.2	176.23	0.999	1.9×10 ¹⁶
0.3	200.13	0.999	6.2×10 ¹⁷
0.4	194.15	0.998	5.4×10 ¹⁶
0.5	186.27	0.999	7.6×10 ¹⁵
0.6	214.97	0.992	5.9×10 ¹⁶
0.7	255.68	0.998	4.0×10 ¹⁴
Average	199	0.998	1.1×10 ¹⁷

Table 7. Kinetic parameters of PS at different α values

The kinetic parameters for EFB, PS, pure cellulose and lignin are determined as listed in the Table 8 and compared with experimental works reported. The average activation energy of cellulose is 160 kJ/mol and pre-exponential factor is $1.2 \times 10^{13} \text{ m}^{-1}$ for first order kinetic model. Several researchers found that first order kinetics fit well for cellulose decomposition reaction (Várhegyi et al., 1997; Grønli et al., 1999; Hu et al., 2007).

Activation energy evaluated for cellulose in the present study is in good agreement with the work reported by Zhang et al. (2009) for a first order kinetic model. Nevertheless, the value is comparatively low corresponding to values reported by Varhegyi et al. (1997), Grønli et al. (1999) and Yang et al. (2004). The reason may be different source of cellulose and different method followed by the authors. Secondly, this may be due to relatively high heating rates used in the present study. Meanwhile, Hu et al. (2007) reported high activation energy (233 kJ/mol) using Flynn-Wall-Ozawa method at low heating rates of 2.5, 5, 10 °C/min. Grønli et

al. (1999) observed the effect of different heating rates on activation energy for cellulose and found low activation energy at high heating rates. Similarly, much lower activation energies were found by Milosavljevic et al. (1995) at high heating rates. The same effect was also observed for pre-exponential factor. This is due to the heat transfer limitation between sample particles and the surroundings at high heating rates. Varhegyi et al. (1997) suggested utilization of low sample mass to minimize the heat transfer limitations at high heating rates.

Biomass	Kinetic Parameters			R ²	Reference
	E (kJ/mol)	A (m ⁻¹)	n (-)		
Cellulose	160	1.2×10 ¹³	1	0.997	This study
	175	7.16×10 ¹²	1	0.999	Zhang et al. (2009)
Lignin	175	2.06×10 ¹⁵	1	0.994	This study
	171	2.73×10 ²⁵	1	-	Morugun et al. (2008)
EFB	151	1.4×10 ¹⁵	5.3	0.997	This study
	61	3.14×10 ²	1	0.991	Yang et al. (2004)
PS	199	1.1×10 ¹⁷	5.0	0.998	This study
	111	5.27×10 ⁷	2.54	-	Luangkiattikhun et al. (2008)

Table 8. Comparison of kinetic parameters

Pure lignin is decomposed with average activation energy of 175 kJ/mol and pre-exponential factor of 2.06×10¹⁵ m⁻¹ for first order kinetics model. These kinetic parameters are in good agreement with work reported by Murugan et al. (2008).

EFB and PS are decomposed with the average activation energy of 151 and 199 kJ/mol and reaction order of 5 and 5.3, respectively. The corresponding average pre-exponential factors evaluated for EFB and PS are 1.4×10¹⁵ and 1.1×10¹⁷ m⁻¹, respectively. These values are somehow larger than those reported by Yang et al. (2004) for first order reaction kinetics. Luangkiattikhun et al. (2008) and Guo & Lua, (2001) observed lower activation energy and pre-exponential factor based on single-step nth order and first order kinetic model for PS. However, in these studies, comparatively high values were obtained using two step kinetic models.

Based on kinetic parameters, it is easy to decomposed EFB as compared to PS, pure cellulose and lignin. The order of decomposition from fast to slow is EFB > cellulose > lignin > PS.

4. Conclusion

A biomass decomposition study has been carried out to investigate different breakdown region and kinetic parameter evaluation for EFB and PS. As major components of biomass, pure cellulose and lignin decomposition kinetics were also studied. TG and DTG curves were studied in detail to understand the major decomposition region in EFB, PS, pure cellulose and lignin. The kinetic parameters of EFB and PS are found to be higher compared to reported values in the literatures. This difference may be due to the different methods for kinetic parameter determination and relatively high heating rates used in the present study. Based on the kinetic parameters, PS was difficult to decompose as compared to EFB. The

possible reason is may be relatively high lignin content present in PS. This high lignin content was also responsible for low decomposition rate of PS as compared to EFB. Pure lignin had the lowest decomposition rate among all the species. Moreover, lignin content in PS was decomposed at high temperature as compared to EFB based on higher heating rates.

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This book aspires to be a comprehensive summary of current biofuels issues and thereby contribute to the understanding of this important topic. Readers will find themes including biofuels development efforts, their implications for the food industry, current and future biofuels crops, the successful Brazilian ethanol program, insights of the first, second, third and fourth biofuel generations, advanced biofuel production techniques, related waste treatment, emissions and environmental impacts, water consumption, produced allergens and toxins. Additionally, the biofuel policy discussion is expected to be continuing in the foreseeable future and the reading of the biofuels features dealt with in this book, are recommended for anyone interested in understanding this diverse and developing theme.

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