
Thermal degradation analysis of light, medium and severe torrefaction of oil palm plantation residue

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Abstract: Oil palm plantation waste is a potential renewable energy resource in terms of energy security and carbon-neutral fuel. The wastes from the plantation sites, such as oil palm fronds (OPF) and oil palm trunks (OPT) are currently underutilised. In this work, the thermal degradation behaviour of the OPF and OPT, and evaluation on the kinetic and activation energy of the torrefied products are investigated. The samples undergo light (200°C), mild (250°C), and severe (300°C) torrefaction for 30 minutes in inert condition. Then, the samples are characterised using bomb calorimeter, FTIR, and TGA analyses. As a result, the calorific values of OPF and OPT improved to 22.46 MJ/kg and 25.48 MJ/kg from the raw samples. At mild torrefaction, the energy yield of the solid product was higher for both OPF and OPT. In conclusion, the mild condition process showed optimum characteristics for both OPT and OPF as the feedstock for energy application.

Keywords: energy; oil palm; torrefaction; thermal kinetics.

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1 Introduction

Energy consumption is one of the main aspects that has been discussed and debated; recently, due to the increasing world demand. The projection made by the United States Energy Information Administration (EIA) shows an increment in the world energy demand of 28% for the period 2015 to 2040 (IEO, 2017). There are two categories of energy – renewable and fossil fuel – for which the rate of demand for renewable fuel is four times faster than that of fossil fuel. The current main concerns across the world are the price fluctuation of oil and its high demand (Hosseini et al., 2018). Thus, energy security and sustainability have become a major emerging issue that could be addressed by diverting the source of energy towards renewable and sustainable fuel. Biomass is one of the renewable and sustainable fuels that have received high attention and interest worldwide. This source of energy is a unique energy that is available throughout the world. Thus, biomass can play a significant role as a promising alternative energy source in the future. The biomass can be utilised in three structures-solid (pellet, biochar, and briquette), liquid (bioethanol and biodiesel), and gaseous (biogas and syngas) (Flamos et al., 2011).

The oil palm industry is one of the contributors to Malaysia's economic activities by contributing 28% of world palm oil production. This industry generates a huge amount of solid waste since the palm oil products only contribute 10% of the total dry mass of the palms and the balance of 90% is considered as oil palm solid waste. The solid waste produced by the palm oil industry includes empty fruit bunches (EFB), palm kernel shells (PKS), and mesocarp fibre (MF) from the milling process, while oil palm fronds (OPF) and oil palm trunks (OPT) are the major waste from the plantations. Disposing of the oil palm waste could lead to pollution of the environment. OPT can be used as a raw material in the production of medium density fibreboard (MDF), plywood, and lumber for furniture manufacturing. Meanwhile, OPF is usually utilised to supply nutrients to the oil palm tree by decomposition on the ground (Abnisa and Wan Daud, 2014). Besides these applications, OPT and OPF have a high potential for use as biomass energy due to their lignocellulosic components (high carbon and hydrogen content). OPT and OPF, however, contain several undesired properties that limit their direct exploitation.

Raw OPT and OPF are classified as low-grade fuel, in which drawbacks, such as low energy value and bulk density, high moisture content, and poor grindability, will result in limitations in terms of their utilisation and thermal energy conversion, as well as present storage complications (Xue et al., 2014). The low value of energy is due to the high oxygen content that causes the production of a large amount of flue gas during the combustion process (Karsli and Saraç, 2018). Meanwhile, the high moisture content of raw biomass results in pellet disintegration, moss growth, and biodegradation after the pelletisation process (Uemura et al., 2011), as well as a decrease in the overall gasification temperature (Aziz et al., 2017). These characteristics and properties make biomass unacceptable for use as a fuel energy source. To overcome these problems and to increase the energy value, the biomass needs to undergo a pre-treatment process – chemical, mechanical, thermal, hydrothermal, or biological (Nhuchhen et al., 2014). The pre-treatment processes are important to change the structural and chemical compositions of the biomass by altering its amorphous and crystalline components (Shi et al., 2016).

The thermal pre-treatment process, which normally refers to the torrefaction process (dry and wet torrefaction) has been recognised as one of the feasible methods to improve the quality of biomass by converting it into suitable fuel energy with high energy density, hydrophobic characteristics, high compaction ability, low oxygen/carbon and hydrogen/carbon ratios, low moisture content, and lighter in weight.

Torrefaction is categorised as mild pyrolysis, which involves the thermal decomposition process of biomass in an inert atmosphere without the presence of oxygen. It is carried out in operating temperatures typically ranging from 200°C to 300°C with heating rates of less than 50°C/min (Harun et al., 2017). During this heating process, the lignocellulose components (hemicellulose, cellulose, and lignin) decompose into several types of volatile matter. These components will break down into condensable hydrocarbon, hydrogen, oxygen, and carbon-based gaseous through three major reactions – decomposition, devolatilisation, and depolymerisation (Regmi et al., 2017). The devolatilised product, namely, torrefied biomass, can improve fuel quality of the torrefied biomass and make it very attractive for further utilisation, such as combustion (Kraszkievicz, 2016) and gasification (Pérez et al., 2016) applications in general. Many studies have been done on the torrefaction of biomass, such as bagasse and rice husk (Chen et al., 2012), bamboo (Huang and Hsu, 2012), miscanthus (Anna and Scherer, 2018), eucalyptus (Ayoub et al., 2018), pine (Anna and Scherer, 2018), sawdust (Kamila et al., 2017), oil palm fibre (Sukiran et al., 2017), and others.

Although the torrefaction process of oil palm biomass has gained increasing interest among researchers, most of the studies emphasised the waste generated from the palm oil mills that often consists of PKS (Harun et al., 2017; Anna and Scherer, 2018), EFB (Sellapah et al., 2016; Harun et al., 2017; Mohd Fuad et al., 2018), and MF (Sellapah et al., 2016). However, the waste from the plantation sites, such as OPF and OPT, are currently underutilised. Hence, more research is required to understand the physical and chemical processes in terms of the torrefaction of OPF (Wahid et al., 2017) and OPT towards the better development of biomass Malaysia in terms of a renewable energy source. In general, the torrefaction process is classified into three types – light (200–235°C), mild (235–275°C), and severe (275–300°C) (Chen et al., 2018). Hence, the present work is intended to investigate the thermal degradation behaviour of the OPF and OPT with different torrefaction conditions. Furthermore, this study is to evaluate the kinetics and activation energy (EA) of torrefied products. The obtained results can provide a comprehensive insight into the degradation behaviour of lignocellulosic component of the biomass which is significantly related to the pre-treatment conditions. Besides, to the authors' knowledge, no study has been reported with regards to the torrefaction of OPT as a fuel source.

2 Materials and method

2.1 Materials

The raw OPF were collected from LCSB Lepar Oil Palm Mill, Gambang, Pahang, and the OPT were purchased from Regalis Asia Sdn Bhd, Kuala Lumpur.

2.2 *Methods*

2.2.1 *Raw material preparation*

The petioles of OPF are used after removing the leaflets. Both materials are further dried, processed, and ground to generate OPF and OPT in a fibrous form (~10 mm length) for experimental purposes. The samples are placed in a sealed container and stored indoors at room temperature until the experiments are carried out.

2.2.2 *Torrefaction process*

Two grams of the sample was weighed and inserted in the reactor. Nitrogen gas at a pressure of 1 atm is then flushed into the reactor for 15 minutes to eliminate the oxygen inside. This is followed by selecting the required pattern, which is first at a temperature of 200°C with a constant heating rate of 10°C/min by an electrically heated reactor. During the process, the exit gas is trapped in a cold bath to prevent the emission of harmful gas to the atmosphere. After a residence time of 30 minutes, the reaction is stopped by turning off the heater, and then the reactor is left to cool down to ambient temperature. The torrefied sample is collected, weighed, and stored in an airtight sample bottle before characterisation. The steps above are repeated by changing the temperature pattern at the control system of the reactor to 250°C and 300°C. Each experiment is repeated three times to obtain optimal results.

2.2.3 *Characterisation*

The calorific value (CV) is determined by a bomb calorimeter. The Fourier transform infrared spectroscopy (FTIR) analysis is conducted using a thermo scientific FTIR spectrometer with wavenumbers in the range of 4000 to 700 cm⁻¹ to determine the surface chemical bonding. Thermal gravimetric (TGA) analysis is carried out for both the raw and torrefied biomass with the temperature ranging between 25°C and 800°C at a heating rate of 10°C/min under nitrogen gas flow.

2.3 *Data analysis*

2.3.1 *Raw material preparation*

The biomass is subject to changes in the mass and energy yield during the torrefaction process. The mass and energy yields were calculated using equation (1), as proposed by (Bergman et al., 2005).

$$\text{Energy yield} = \frac{\text{Mass yield}}{\text{CV ratio}} \quad (1)$$

where

$$\text{Mass yield} = \frac{\text{Mass sample after torrefaction (g)}}{\text{Mass sample before torrefaction (g)}}$$

and

$$CV \text{ ratio} = \frac{CV \text{ sample after torrefaction} \left(\frac{J}{mol} \right)}{CV \text{ sample before torrefaction} \left(\frac{J}{mol} \right)}$$

2.3.2 Determination of kinetic parameters

For the torrefaction process, the kinetic parameters are evaluated through the temperature profile obtained from the thermogravimetric trends. The rate constant, k , can be acquired through equation (2), the Arrhenius equation;

$$k = A \exp\left(-\frac{E_A}{RT}\right) \quad (2)$$

where E_A is the EA (J/mol), A is the frequency factor (min^{-1}), T is the temperature profile ($^{\circ}\text{C}$) and R is the gas constant.

The rate of decomposition in the TGA process can be evaluated in the first-order reaction, through equation (3) as proposed by Kamila et al. (2017).

$$\frac{d\alpha}{dT} = \left(\frac{k}{\beta}\right)(1-\alpha)^n \quad (3)$$

where $d\alpha = \frac{W_i - W_s}{W_i - W_f}$, W_i is the initial weight of the sample (g), W_s is the actual weight of the sample (g), W_f is the final weight of the sample (g), dT = differential temperature ($^{\circ}\text{C}$), k is the rate constant, β is the heating rate constant ($^{\circ}\text{C}/\text{min}$) and n is the order of the reaction.

Therefore, by replacing and rearranging the rate constant in equation (2) in equation (3), and integrating the equation can be simplified as in equation (4).

$$\ln\left[-\frac{\ln(1-\alpha)^{1-n}}{T^2(1-n)}\right] = \ln\left[\frac{AR}{\beta E_A}\right] - \frac{E_A}{RT} \quad (4)$$

The plot of $\ln\left[-\frac{\ln(1-\alpha)}{T^2}\right]$ versus $1/T$ becomes a linear line if the order of reaction, n is equivalent to 1. The EA and the frequency factor (A) can be determined from the regression line's slope and intercept, respectively (Salema et al., 2019)

3 Results and discussion

3.1 Calorific values

CV analysis is performed on the biomass samples in both their untreated state and after torrefaction at 200°C , 250°C , and 300°C at a constant residence time of 30 minutes. As presented in Table 1, the results show that the reaction temperature is a significant

variable in the torrefaction of OPF and OPT. For both biomass types, increasing the severity of the torrefaction temperatures result in an increment in the CV. The experimental data of the OPF sample is also compared with the literature, which also proved a similar trend of the heating value over the torrefaction conditions.

Table 1 The CVs of the OPT and OPF after the torrefaction process

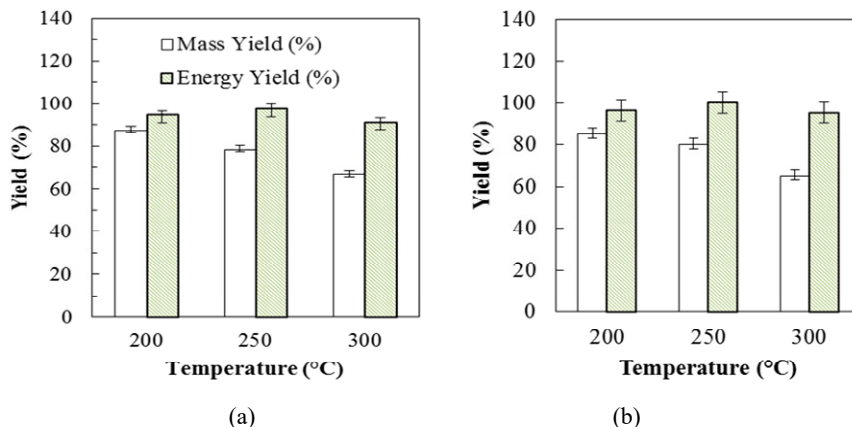
	<i>Condition</i>	<i>CV (MJ/kg)</i>		<i>References</i>
		<i>Experimental</i>	<i>Literature</i>	
OPF	Raw	16.41	17.74	Sukiran et al. (2017)
	200°C	17.77	18.31	
	250°C	20.45	19.28	
	300°C	22.46	23.79	
OPT	Raw	17.41	17.47	
	200°C	19.77	-	
	250°C	21.78	-	
	300°C	25.48	-	

The CV of raw OPT is higher than OPF at 17.41 MJ/kg compared to 16.41 MJ/kg, respectively. After torrefaction at 200°C, 250°C, and 300°C, the CV of OPF increases between 8 and 37%, respectively. The CV of OPT shows a similar trend, with an increment of between 14 and 46%.

3.2 *Mass and energy yield*

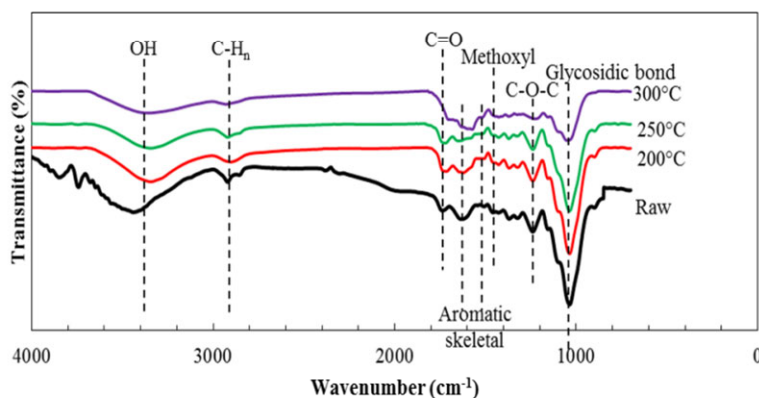
Figure 1 shows the effects of the torrefaction temperature on the mass and energy yields of OPF and OPT. In general, the mass yields of both OPF and OPT are decreased significantly with elevated torrefaction temperatures. It is found that the mass yields for both OPF and OPT are decreased at about 20% from 200°C to 300°C, which is mainly due to the volatile hydrocarbon release due to the rapid thermal decomposition of hemicellulose, cellulose, and some constituents of lignin.

In terms of the energy yield, both OPF and OPT are found to maintain their respective energy yield at greater than 90%, despite the difference in the severity of the torrefaction treatment (i.e., temperature). The energy yield of the torrefied OPF recorded the highest value at 250°C (97.6%), which is greater compared to the energy yields at 200°C (94.6%) and 300°C (91.1%). A similar trend is illustrated the energy yield of torrefied OPT. The gap relating to the mass and energy yield implies that the mass loss is balanced by the increasing CV with elevated torrefaction temperature (Sabil et al., 2013). Therefore, from this study, the temperature of 250°C gives the optimum torrefaction result for both OPF and OPT to acquire a high energy yield without a significant loss of mass.

Figure 1 Effect of temperature on mass and energy yield for (a) OPF and (b) OPT

3.3 Functional group analysis

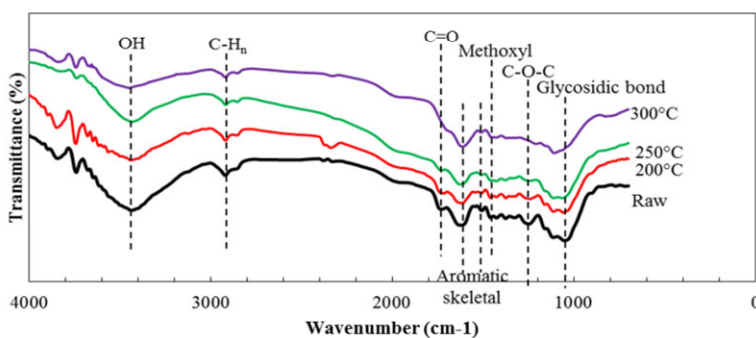
FTIR analysis is carried out to determine the functional group of torrefied samples at different temperatures respectively, in Figure 2 and Figure 3. The spectroscopy from the FTIR analysis is used to investigate the differences in the functional groups of the torrefied products. Abnisa et al. (2013) suggested that the vibrations of the functional group shifted towards lower intensity for the most severely treated samples. The trends recorded in Figures 2 and 3 illustrate the decrement in intensity at peak $\sim 3,500\text{ cm}^{-1}$, which is attributed to the reduction in the O-H groups in the samples at elevated temperatures, which shows that the water molecules within the solids are gradually removed. The loss of the O-H group also explains the improvement in the hydrophobic behaviour of torrefied OPF and OPT.

Figure 2 Spectroscopy behaviour of OPF products (see online version for colours)

In addition, the aliphatic CH_n groups, detected at $\sim 2,900\text{ cm}^{-1}$, also weakened by the increment in temperature, indicating that several long aliphatic chains in the raw samples are broken down. In the region below $2,000\text{ cm}^{-1}$, more changes in intensity can be observed between the raw and the torrefied samples. The chemical bonds detected under this wavelength are aromatic groups, and methoxyl and glycosidic groups (Garba et al., 2018).

The peak of the carbonyl (C=O) stretching vibrations is detected between wavelength $\sim 1,700$ and $1,750\text{ cm}^{-1}$. In raw OPF and OPT, the vibrations are generally due to the carboxylic acids in the hemicellulose structures, which can include xylan, xyloglucan, arabinoxylan, and galactoglucomannan (Ibrahim et al., 2013). The peak is attributed to the glycosidic bonds ($\sim 1,050\text{ cm}^{-1}$), indicates the presence of cellulose. This peak gradually weakens and disappears from 250°C to 300°C for both OPF and OPT, proving that the cellulose structure is partially degraded at this range of conditions. While the decrease in intensity for both the aromatic skeletal vibrations ($\sim 1,520\text{ cm}^{-1}$) and C-O-C aryl-alkyl ether linkages ($\sim 1,250\text{ cm}^{-1}$) indicate the decomposition process of the lignin structure.

Figure 3 Spectroscopy behaviour of OPT products (see online version for colours)



3.4 Thermal stability analysis

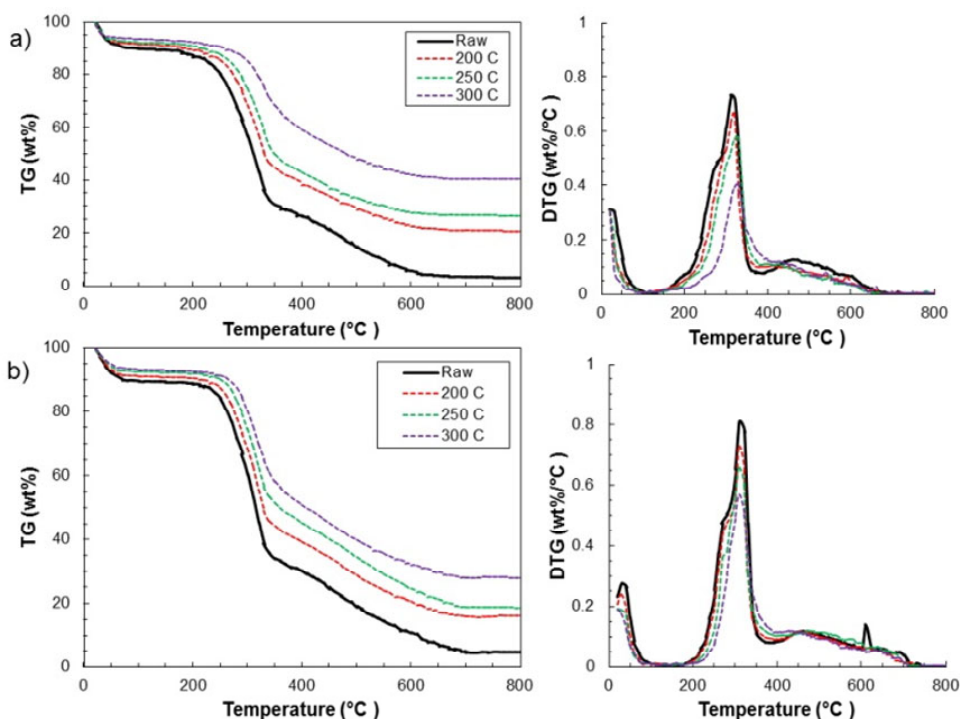
The lignocellulosic structure of biomass can be qualitatively determined from TGA analysis. According to Garba et al. (2018), the weight losses observed in the TG and DTG curves are found to be relevant to the composition of the cellulose, hemicellulose, and lignin fractions in the lignocellulosic biomass. Figure 4 shows the different stages of decomposition curves for OPF and OPT samples.

The first stage, which is at a temperature below 200°C , corresponds to the loss of water; the volatilisation of light molecules may have contributed to the weight loss in this stage. This is also supported by Talero et al. (2019), as mostly, at this stage, the sample undergoes a loss of moisture. At this early stage, the weight losses for all the samples are less than 11%, and the weight losses decrease with elevated torrefaction temperature, which results in the improved hydrophobicity of torrefied OPF and OPT. Initiation of torrefaction in OPF and OPT can be studied at temperatures of around 250°C based on the thermal decomposition of hemicellulose, cellulose, and lignin. Meanwhile, during the second stage of decomposition, devolatilisation process occurs. This stage is illustrated by the second stage of decomposition, which occurs at temperatures ranging between 200

to 500°C, where a remarkable slope in the TG trends is observed. This corresponds to the significant drop in weight of the samples due to the release of volatile hydrocarbon from the rapid thermal decomposition of hemicellulose, cellulose, and some parts of the lignin. In this study, the devolatilisation can be referred to as the thermal degradation of the biomass component. This indicates the major step in the thermochemical conversion process that involves the lignocellulosic biomass.

In this stage, the weight of the samples dropped below 40% due to the liberation of volatile hydrocarbon from the rapid thermal decomposition of hemicellulose, cellulose, and some parts of the lignin, which corresponds with the fact that (< 80 wt%) of the biomass is produced by a volatile fraction. This observation implies that the torrefaction treatment (between 200 and 300°C) has a marked impact upon the lignocellulosic structure, stemming from the thermal degradation of the hemicellulose and cellulose.

Figure 4 TGA and DTG curves of (a) OPF and (b) OPT at different torrefaction temperatures (see online version for colours)



Apart from that, the lignocellulosic structure of biomass can be identified using DTG as reported in a previous study (Kamila et al., 2017). Due to the different structures that exist in the lignocellulosic of biomass, it can be distinguished based on the distribution of the weight loss intensity of the biomass. In this research, the thermal decomposition of hemicellulose, cellulose, and lignin in OPF and OPT was found to occur at temperatures of 270°C, 310°C, and 370°C, respectively, as shown in Figure 4. This observation is supported by Sukiran et al (2017) who reported that the standard temperature decomposition of the three major elements of lignocellulosic (hemicellulose, cellulose, and lignin) is in the range of 150°C–350°C for hemicellulose; 275–350°C for cellulose,

and 250–500°C for lignin. Then, the final stage is between 450°C and 800°C, where the significant mass loss of about 30 wt% is from the organic residue (i.e., lignin) decomposition reaction. These are the maximum temperatures of decomposition for the OPF and OPT samples when the samples achieve complete combustion and decomposition. By referring to Figure 4, it can be seen that the weight loss in stage 3 is not as significant as in stage 2. This is primarily due to the steady disintegration of the remaining complex components, which are mainly from the lignin (Talero et al., 2019).

The DTG curves in Figure 4 represent the thermal degradation of the hemicellulose and cellulose of the OPF and OPT. The peaks of the hemicellulose gradually disappear with the increasing severity of torrefaction treatment for both the OPF and OPT. Meanwhile, the severely torrefied OPF and OPT (i.e., at 300°C) contribute the least weight loss, revealing that their resistance against thermal degradation is higher compared to samples torrefied at 200°C and 250°C.

The peak temperature of OPF and OPT, which is 310°C, shows the same values, which indicates that the presence of similar lignocellulosic constituent arrangements for both types of biomass. However, as seen in Table 2, the peak temperature of the torrefied OPF has a small increment, while the OPT shows a consistent peak. The decomposition intensity of raw OPF and OPT is slightly higher (0.73 and 0.81 wt%) compared to the torrefied OPF and OPT samples. With respect to the torrefied OPT and OPF, a constant reduction in the decomposition intensity is observed with the increment in the torrefaction temperature. The low value of decomposition intensity is believed to correspond to the thermal degradation of the hemicellulose and cellulose during the torrefaction process, which further reduces the decomposition of the torrefied OPF and OPT.

Table 2 The peak temperature and maximum decomposition intensity of raw/torrefied OPF and OPT

<i>Sample</i>	<i>*T_T (°C)</i>	<i>T_{peak} (°C)</i>	<i>Maximum degradation intensity (wt%/°C)</i>
OPF	0	310	0.73
	200	320	0.67
	250	330	0.58
	300	330	0.41
OPT	0	310	0.81
	200	310	0.73
	250	310	0.66

Note: *T_T is for torrefaction temperature, 0°C is equal to raw OPF/OPT.

3.5 Kinetic analysis

The reaction temperature is one of the main factors that influence biomass torrefaction. The EAs, pre-exponential factors, and mass loss of all the samples; the raw and torrefied OPF and OPT, in the second and third decomposition stages, are tabulated in Table 3, where the integration of the Arrhenius equation and the first-order reaction is utilised. The exemplary plot of the kinetic equations for OPT at a torrefaction temperature of 250°C is illustrated in Figure 5 at different torrefaction conditions.

In this work, thermal degradation occurs in three stages: stage 1 for the moisture and decomposition of volatile matter. Stage 2 is recorded between 220°C and 350°C, which shows the decomposition of hemicellulose and cellulose content from the OPF and OPT. While the final decomposition stage (stage 3) was expected to occur between 350°C and 600°C due to the removal of lignin in the lignocellulosic structure of OPF and OPT.

Table 3 Kinetic parameters in the second and third stages at different torrefaction temperatures

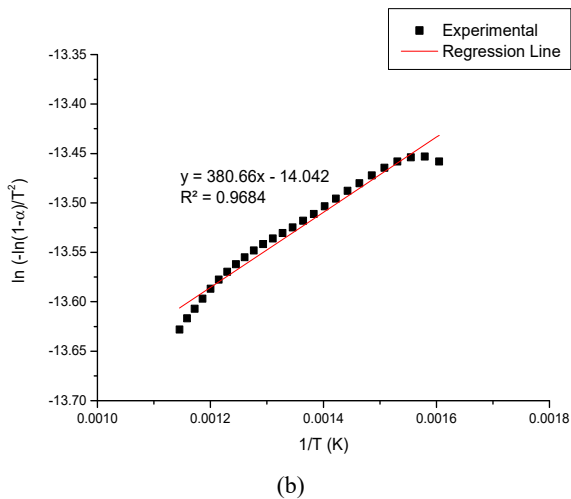
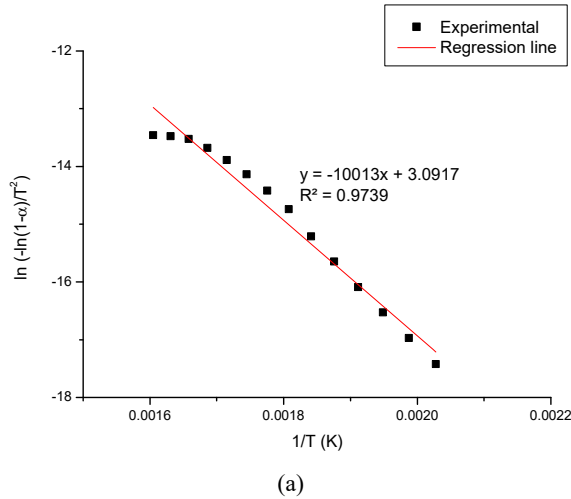
Sample	Condition	*Decomposition stage	Ea (J/mol)	A (min ⁻¹)	R ²	Mass loss (%)
OPF	0	Stage 2	46,464	9.983E+02	0.977	55.0
		Stage 3	-5,482	-4.025E-03	0.991	23.9
	200	Stage 2	49,617	1.797E+03	0.990	43.8
		Stage 3	-5,336	-4.024E-03	0.966	21.8
	250	Stage 2	52,288	2.812E+03	0.995	40.0
		Stage 3	-4,930	-4.037E-03	0.909	22.0
300	Stage 2	46,343	4.893E+02	0.984	23.5	
OPT	0	Stage 2	65,092	5.532E+04	0.959	54.3
		Stage 3	-5,580	-3.671E-03	0.992	23.4
	200	Stage 2	65,363	5.545E+04	0.975	45.8
		Stage 3	-4,757	-3.776E-03	0.984	24.4
	250	Stage 2	83,248	2.204E+06	0.974	40.4
		Stage 3	-3,165	-3.035E-03	0.968	26.8
300	Stage 2	68,015	6.974E+04	0.982	34.2	

Note: *Stage 2 for a temperature range of 220–350°C, stage 3 for a temperature of 350–600°C.

The EA in Table 3 indicates the minimum energy required to enhance the decomposition process of the raw and torrefied OPT and OPF. Based on the data calculated, the EA value for the OPF is lower than that for the OPT showing that OPF is a more suitable raw material for energy applications. In terms of the conditions, severe torrefaction only illustrates two stages of decomposition. This might be due to the higher amount of carbon content in the OPT and OPF after being torrefied at 300°C, as most of the volatile compounds in the lignocellulosic bonds have been decomposed. Also, the EA values (stage 2) for severe torrefied OPF and OPT are lower than those for the low and mild torrefaction process with minimum mass loss. In view of the changes in EA trends, the pre-exponential factor also changes to adjust for the significant reduction in chemical kinetics due to the growth of the exponential term.

The second stage of decomposition recorded a higher percentage of weight loss of between 23 and 44 wt% compared to the third stage (22–27 wt%). This indicates that the cellulose and hemicellulose content (stage 2) is higher compared to the lignin content, which indicates the degradation range in stage 3. The EA obtained for stage 2 shows an endothermic reaction, which means that energy needs to be provided to initiate the decomposition, which in stage 3, the EA resulted in an exothermic reaction due to the energy released from the decomposition of the lignin.

Figure 5 Linear regression of the Arrhenius plot for (a) Stage 2 and (b) Stage 3 of torrefied OPT at 250°C (see online version for colours)



5 Conclusions

In summary, the thermal degradation rate of the OPF and OPT is increased when the torrefaction conditions increased from light, mild to severe levels. In terms of characteristics, the CVs of OPF and OPT are improved from 16.41 MJ/kg (raw OPF) to 22.46 MJ/kg (torrefied OPF) and 17.41 MJ/kg (raw OPT) to 25.48 MJ/kg (torrefied OPT). Apart from that, the mass yields of both materials are found to decrease with an increase in the torrefaction temperature, indicating that degradation of lignocellulosic components, such as hemicellulose, cellulose, and lignin, occurs. At a mild torrefaction (250°C), the energy yield of the solid product is higher for both OPF and OPT, with 97.6% and 99.9% of the energy retained in the solid materials, respectively. The kinetic

study shows the mild condition of the torrefaction process leads to an evident decrease in the EA of the combustion process. In conclusion, mild condition of torrefaction process results in optimum energy yield and CVs for both OPT and OPF as the sustainable fuel feedstock for energy application.

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