Thermogravimetric analyses (TGA) of three oil palm biomass pyrolysis: Kinetics and reaction mechanisms

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Abstract. Oil palm biomass generated from the plantation site (i.e., oil palm trunk (OPT) and oil palm frond (OPF)) are an attractive source for pyrolysis given their abundant availability. Despite this potential, there is no comprehensive study on the pyrolysis of OPT and OPF. In this work, the pyrolysis of OPT, OPF, and PKS are compared and analysed by thermogravimetric analysis (TGA). The kinetics and reaction mechanisms of these biomass during pyrolysis were determined using Kissinger-Akahira-Sunose (KAS) model-free method and Šesták-Berggren function. Pyrolysis of the three oil palm biomass is complex and exhibits different thermal behaviours and strong overlapping reaction between the pseudo-components, which can be attributed to their different constituents. The apparent activation energy of OPT, OPF, and PKS are 17.83-116.58, -45.36-225.60, and 56.50-90.42 kJ mol⁻¹. The variation in the apparent activation energy indicates that pyrolysis of the three oil palm biomass involves multiple reaction mechanisms. Thermal decomposition of the pseudo-hemicellulose and pseudo-cellulose in the three oil palm biomass are co-controlled by two reaction mechanisms, i.e., phase boundary reaction and nucleation, with phase boundary reaction as the dominant mechanism.

Introduction
The rapid depletion of fossil fuels has prompted researchers to search for renewable alternative resources to replace fossil fuels. Being the world’s second largest palm oil exporter, Malaysia generates up to 51.2 million tonnes of oil palm biomass in 2017 [1]. Oil palm biomass such as oil palm trunks (OPT), oil palm fronds (OPF), palm kernel shells (PKS), empty fruit bunches (EFB), and mesocarp fibre (MF) are a promising source for renewable energy production given their abundant availability and high energy value [1]. Amongst the various types of oil palm biomass, OPT and OPF are normally left as mulch in the plantation site or disposed as waste without further utilisation. It is more desirable to convert these biomass into bio-fuels that can add value to the economy and environment.

Biomass can be converted into value-added bio-fuels via various thermochemical processes, amongst which are pyrolysis, combustion, and gasification. Pyrolysis is the thermal decomposition of organic materials under inert atmosphere to produce bio-oil, bio-gas, and bio-char [2]. Pyrolysis has attracted wide attention since its product can be easily stored and transported [2]. Furthermore, pyrolysis is the initial process that takes place in both combustion and gasification [3]. Therefore, thorough understanding of the pyrolysis process is essential to achieve efficient use of oil palm biomass in any thermochemical conversion process [3].

The thermal decomposition behaviour of oil palm biomass is complex. An in-depth understanding of the dominant reaction mechanisms of oil palm biomass pyrolysis through kinetic modelling is essential for the biomass reactor design and optimisation. The kinetic modelling of the pyrolysis of oil palm biomass, especially EFB, MF, and PKS has been performed using both model-free and model-fitting methods [4, 5]. Nonetheless, the reaction mechanism that governs the pyrolysis of oil palm biomass cannot be determined using only model-free method. The Coats-Redfern model-fitting method for example can be used to determine the reaction mechanism function. However, it is only limited to several functions. The Šesták-Berggren function, which is a numerical model that can represent all models in a single expression was proposed by Šesták and Berggren [6] is more suitable given the unlimited functions and kinetic parameters that can be obtained using this function.
Our previous work [7] has studied the pyrolysis of five oil palm biomass (i.e., OPT, OPF, PKS, EFB, and MF) at a single heating rate. To date, the pyrolysis behaviour, kinetics, and reaction mechanism of OPT, OPF, and PKS under different heating rates has not been studied, which is essential for the effective reactor design and operation. In this study, pyrolysis of oil palm plantation biomass, i.e., OPT and OPF are compared with that of palm oil mill biomass, i.e., PKS at different heating rates. Subsequently, kinetic modelling is performed using model-free method to determine the apparent activation energy followed by reaction mechanism determination using Šesták-Berggren function.

Experimental section

Materials and methods
Oil palm trunk (OPT) and oil palm fronds (OPF) used in this study were obtained from oil palm plantation estate in Serian, Kuching while palm kernel shells (PKS) were obtained from Felcra Jaya Samarahan Palm Oil Mill, Samarahan. The three biomass were dried in a convection oven at 105 °C for 24 hours to reduce their moisture content. The dried samples were then milled using a variable speed rotary mill (Fritsch, Model PULVERISETTE 14) and sieved to 125-250 μm using a sieve shaker (Fritsch, Model ANALYSETTE 3 PRO). The milled samples were collected and stored in a sealed bag, which was kept in a desiccator for further use.

Thermogravimetric analysis (TGA)
Thermogravimetric analysis of the three biomass were performed using a simultaneous thermal analyser (STA) (Perkin Elmer, STA 8000). Approximately 5 g of sample with 125-250 μm particle size was placed in a crucible and heated from 30 to 900 °C at three different heating rates (20, 30, 40 °C min⁻¹). Nitrogen gas at 20 mL min⁻¹ flow rate was used as the purging gas to induce inert atmosphere for pyrolysis.

Kinetic analysis
Pyrolysis of oil palm biomass is complex and involves multiple overlapping reactions, which can be identified from the peak appearance in their thermal profile. Each reaction (peak) was thus assigned to different apparent activation energies, which depend upon the temperature and the extent of conversion [8]. Kinetic analysis in this work was performed in two stages, i.e., (1) apparent activation energy estimation using Kissinger-Akahira-Sunose (KAS) model-free method; and (2) reaction mechanism determination by model-fitting with Šesták-Berggren function.

Model-free method. The thermal decomposition of oil palm biomass can be described by the solid-state reaction in Eq. 1 [8].

\[
\frac{d\alpha}{dt} = A \exp \left( - \frac{E_a}{RT} \right) f(\alpha)
\]  

(1)

where \(A\) is the pre-exponential factor, \(E_a\) is the apparent activation energy, \(R\) is the universal gas constant, \(T\) is the absolute temperature, \(f(\alpha)\) is the reaction mechanism function, and \(\alpha\) is the extent of reaction that is given by Eq. 2.

\[
\alpha = \frac{w_0 - w_t}{w_0 - w_f}
\]

(2)

where \(w_0, w_t, w_f\) is the initial, instantaneous, and final weight of the samples, respectively.

Substituting the heating rate \(\beta = \frac{dT}{dt}\) into Eq. 1 gives,

\[
\frac{d\alpha}{dT} = \frac{A}{\beta} \exp \left( - \frac{E_a}{RT} \right) f(\alpha)
\]

(3)

Integrating Eq. 3 gives,

\[
g(\alpha) = \int_0^\alpha \frac{d\alpha}{f(\alpha)} = \frac{A}{\beta} \int_0^T \exp \left( - \frac{E_a}{RT} \right) dT = \frac{AE_a}{\beta T} p(x)
\]

(4)

where \(p(x)\) is the temperature integral without analytical solution and \(x = E_a/RT\). The integral equation (Eq. 4) can be solved using Kissinger-Akahira-Sunose (KAS) approximation method, leading to the KAS model represented by Eq. 5 [8].
The apparent activation energy for each extent of reaction (\(\alpha\)) can be obtained from the slope by linear plots of \(\ln(\beta/T^2)\) against \(1/T\) at different heating rates (i.e., 20, 30, and 40 \(^\circ\)C min\(^{-1}\)) and extent of conversion step size (\(\Delta\alpha\)) of 0.05. The apparent activation energy for each reaction (peak) was obtained by averaging the activation energies in the peak temperature range corresponding to the extent of reaction range. For example, if the temperature range for Peak I is 200-400 \(^\circ\)C and the extent of conversion corresponding to this temperature range is 0.1-0.75, the apparent activation energy within that extent of conversion range is averaged and is used to estimate the reaction mechanism function.

\[ f(\alpha) = a^n(1-\alpha)^m[-ln(1-\alpha)]^p \]  \(\text{Eq. 6}\)

where \(m\), \(n\), and \(p\) represent the different reaction mechanism. Seven reaction mechanism functions can be obtained from the different combinations of \(m\), \(n\), and \(p\) \(\text{[6]}\).

Substituting \(\text{Eq. 6}\) into \(\text{Eq. 3}\) and subsequently linearising both sides of the equation with the natural log gives,

\[ \ln \left( \frac{\beta}{T^2} \right) = \ln \left( \frac{AE_a}{Rg(\alpha)} \right) - \frac{E_a}{RT} \]  \(\text{Eq. 5}\)

The apparent activation energy that was determined from the KAS method was substituted into \(\text{Eq. 7}\) while \(\ln A\) can be obtained from the intercept. Assessment of the reaction mechanism function fitting was performed based on the adjusted-coefficient (i.e., adjusted-R\(^2\)) due to the consideration of the variation in the numbers of variables (i.e., 1-3 variables) in the seven reaction mechanism functions.

**Results and discussion**

**Thermogram analysis**

Figure 1 display the pyrolysis weight loss profiles (TGs) and the corresponding derivative weight loss profiles (DTGs) of OPT, OPF, and PKS at heating rates of 20, 30, and 40 \(^\circ\)C min\(^{-1}\). The peak appearance in the DTG profile that corresponds to the maximum mass loss rate is identified as Peak I, II, III, and IV and the temperature ranges for the peak appearance are listed in Table 1. An increase in the heating rate shifts the TG and DTG profile towards higher temperature (i.e., towards the right). In addition, increasing the heating rate results in lower weight loss as observed in the TG profile (Figure 1). Such phenomenon is attributed to the poor heat transfer from the atmosphere to the interior of the solid. At high heating rate, the biomass has shorter residence time to devolatilise, hence resulting in higher solid residue \(\text{[9]}\). It should be noted that although increasing the heating rate skewed the TG and DTG profile towards higher temperature side, it does not change the shape of both the TG and DTG profile.

Pyrolysis of OPT, OPF, and PKS occurred via three main stages based on the TG profile, i.e., (1) surface and bound moisture and light volatiles removal (30-150 \(^\circ\)C), (2) rapid devolatilisation (150-450 \(^\circ\)C), and (3) char formation from the slow devolatilisation of lignin (> 345 \(^\circ\)C). The TG profile reveals that the onset temperature for the rapid devolatilisation to occur was lowest for OPF followed by OPT and PKS, indicating that OPF was the easiest oil palm biomass sample to devolatilise. In addition, PKS has the highest solid char residue relative to OPT and OPF as indicated by the lowest weight loss, thus making PKS a suitable option for bio-char production.

Stefanidis et al. \(\text{[10]}\) reported that the pyrolysis of pure hemicellulose, cellulose, and lignin occurs at 200-320, 280-360, and 140-600 \(^\circ\)C, respectively. The multiple peak appearance in the DTG profile is postulated to represent the thermal decomposition of the biomass pseudo-components, i.e., pseudo-hemicellulose, pseudo-cellulose, and pseudo-lignin. Although the temperature range for the pyrolysis of pure lignocellulosic components cannot represent the thermal decomposition of actual pseudo-component in oil palm biomass, it can be used as a reference to guide the identification of pseudo-component decomposition of oil palm biomass.
Figure 1 Thermal weight loss profile (TG) of (a) OPT, (b) OPF, and (c) PKS and the derivative weight loss profile (DTG) of (c) OPT, (d) OPF, and (f) PKS under inert atmosphere at three different heating rates (i.e., 20, 30, and 40 °C min⁻¹).

Table 1 Temperature range of each peak during pyrolysis.

| β (°C min⁻¹) | Peak temperature range (°C) |
|-------------|----------------------------|
|             | I  | II | III | IV |
| OPT         |    |    |     |    |
| 20          | 240-345 | 345-590 | 730-860 |    |
| 30          | 252-373 | 373-620 | 735-875 |    |
| 40          | 265-390 | 390-630 | 745-900 |    |
| OPF         |    |    |     |    |
| 20          | 205-260 | 260-330 | 330-402 | 402-610 |
| 30          | 210-270 | 270-340 | 340-417 | 417-620 |
| 40          | 220-280 | 280-355 | 355-432 | 432-635 |
| PKS         |    |    |     |    |
| 20          | 270-350 | 350-415 | 415-510 | 695-752 |
| 30          | 280-360 | 360-430 | 430-525 | 725-782 |
| 40          | 285-375 | 375-450 | 450-540 | 710-790 |

As mentioned, the peak corresponding to the maximum weight loss rate is identified as Peak I, II, III, and IV, which excludes the first peak below 150 °C. The first peak below 150 °C (not labelled) for all three samples corresponds to the surface and bound moisture and light volatile removal. Pyrolysis of OPT comes with three peaks, while pyrolysis of OPF and PKS exhibit four peaks. Peak I and II in OPF and Peak I in PKS correspond to the thermal decomposition of pseudo-hemicellulose, which took place at 205-355 and 270-375 °C, respectively. Pyrolysis of OPT however, does not exhibit any peak that can be assigned to the thermal decomposition of pseudo-hemicellulose due to the different pseudo-component(s) and/or mineral constituent(s) in OPT relative to those in OPF and PKS.

Following pseudo-hemicellulose decomposition is cellulose decomposition. The pseudo-cellulose decomposition of OPT, OPF, and PKS is indicated by Peak I, Peak III, and Peak II, respectively. The temperature range during which the pseudo-cellulose of OPT, OPF, and PKS decomposes are 240-390, 330-432, and 350-450 °C, respectively. The thermal decomposition of the pseudo-cellulose overlaps with the thermal decomposition of pseudo-hemicellulose and pseudo-lignin.
Pseudo-lignin decomposition started at low temperature after moisture removal and can only be identified from the slow mass loss rate represented by the flat curve in OPT and OPF that corresponds to Peak II and IV, respectively, while pseudo-lignin decomposition of PKS is represented by a sharp shoulder in Peak III. The offset temperature for the thermal decomposition of pseudo-lignin in OPT, OPF, and PKS is 630, 635, and 540 °C, respectively. Further inspection of the DTG profile of OPT and PKS reveals an isolated peak above 700 °C, which is peak III in OPT and Peak IV in PKS. This peak can be attributed to the self-gasification of char [3, 11].

**Kinetic analysis**

**Apparent activation energy estimation.** Table 2 lists the apparent activation energies for the pyrolysis of OPT, OPF, and PKS and the coefficients for the extent of conversion range of 0.1-0.9 using KAS method. The correlation coefficients for the fitting using KAS method range between 0.8079-0.9461, 0.8855-0.9995, and 0.0023-0.9997 for OPT, OPF, and PKS, respectively. It should be noted that the poor fitting was obtained for the pyrolysis of PKS at the extent of conversion of 0.25 and above 0.75. Similarly, [12] reported that poor correlation coefficients were obtained for the pyrolysis kinetics of PKS using KAS method at extent of conversion below 0.07 and above 0.89. Hence the poor fitting of PKS during pyrolysis at the extent of conversion of 0.25 and above 0.75 can be attributed to the difficulty to fit complex reaction.

**Table 2** Apparent activation energy distribution of the pyrolysis of three oil palm biomass using KAS method.

| α   | OPT          |          | OPF          |          | PKS          |          |
|-----|--------------|----------|--------------|----------|--------------|----------|
|     | Ea (kJ mol⁻¹) | R²       | Ea (kJ mol⁻¹) | R²       | Ea (kJ mol⁻¹) | R²       |
| 0.10| 39.54        | 0.8171   | 30.35        | 0.9944   | 57.50        | 0.9701   |
| 0.15| 42.93        | 0.9309   | 35.05        | 0.9975   | 59.28        | 0.9817   |
| 0.20| 48.97        | 0.9043   | 53.27        | 0.9995   | 56.50        | 0.9714   |
| 0.25| 46.34        | 0.8670   | 59.40        | 0.9965   | 65.54        | 0.2315   |
| 0.30| 55.60        | 0.9204   | 61.62        | 0.9965   | 67.38        | 0.9812   |
| 0.35| 55.04        | 0.8765   | 73.20        | 0.9904   | 69.21        | 0.9904   |
| 0.40| 60.44        | 0.8933   | 70.34        | 0.9967   | 76.15        | 0.9904   |
| 0.45| 57.05        | 0.8770   | 84.08        | 0.9904   | 77.96        | 0.9967   |
| 0.50| 59.74        | 0.9210   | 93.03        | 0.9977   | 79.60        | 0.9997   |
| 0.55| 65.47        | 0.9365   | 87.70        | 0.9643   | 81.06        | 0.9995   |
| 0.60| 61.87        | 0.9213   | 90.50        | 0.9793   | 77.44        | 0.9995   |
| 0.65| 67.77        | 0.9367   | 100.20       | 0.9904   | 83.73        | 0.9995   |
| 0.70| 60.86        | 0.9260   | 93.51        | 0.9794   | 90.42        | 0.9948   |
| 0.75| 38.20        | 0.8967   | 112.52       | 0.9775   | 62.19        | 0.7295   |
| 0.80| 17.83        | 0.8079   | 115.71       | 0.9904   | 13.88        | 0.1056   |
| 0.85| 53.75        | 0.9461   | 225.60       | 0.9214   | -0.65        | 0.0006   |
| 0.90| 116.58       | 0.8788   | -45.36       | 0.8855   | -1.67        | 0.0023   |

The apparent activation energies estimated from the KAS method with correlation coefficient greater than 0.8 for pyrolysis of OPT, OPF, and PKS range from 17.83-116.58, -45.36-225.60, and 56.50-90.42 kJ mol⁻¹, respectively. The large variation in the apparent activation energy with the extent of reaction indicates that different reaction mechanisms were present during the pyrolysis of the three oil palm biomass. Negative apparent activation energy was obtained at extent of conversion of 0.9 for OPF and at 0.85-0.90 for PKS. The negative apparent activation energy obtained from could be attributed to the kinetic method used in this study as reflected by the poor fitting at higher extent of conversion. In addition, the negative apparent activation energy could be attributed to the complex reactions of OPF and PKS. It is also speculated that the negative apparent activation energy obtained from this study was...
due to the spontaneous reactions caused by the free radicals. However, further investigation is needed to support this speculation.

To determine the apparent activation energy for each peak, the extent of conversion corresponding to the peak temperature range in Table 1 is determined. Subsequently, the apparent activation energy within the range of the extent of conversion is averaged. It should also be noted that the apparent activation energies for the peaks related to the pseudo-hemicellulose and pseudo-cellulose are determined, since the fitting correlation coefficient was poor at the higher extent of conversion. The average apparent activation energies of OPT for Peak I is 54.27 kJ mol⁻¹, OPF for Peak I, II, and III are 32.70, 66.98, and 114.85 kJ mol⁻¹, respectively, and PKS for Peak I and II are 64.51 and 81.70 kJ mol⁻¹, respectively.

Reaction mechanism. The reaction mechanism for the pyrolysis of the three oil palm biomass can be described using the Šesták-Berggren function containing three exponents, i.e., m, n, and p, which are associated to the mechanism diffusion, phase boundary reaction, and nucleation, respectively. Results reveal that pyrolysis of the three oil palm biomass in this work is best described by the reaction mechanism function containing exponent n and p, which indicates that the reaction was co-controlled by phase boundary reaction and nucleation. It should be noted that the reaction mechanisms are only determined for pseudo-hemicellulose and pseudo-cellulose (i.e., Peak I, II, and III) only since the apparent activation energy obtained at higher extent of conversion, which is associated to pseudo-lignin (i.e., Peak IV) had relatively poor fitting. Closer inspection to the numerical values of the parameters in Table 3 reveals that the thermal decomposition of both the pseudo-hemicellulose and pseudo-cellulose in all three oil palm biomass are dominated by the phase boundary mechanism as evident from the larger n value relative to p. Our previous study [7] that reports on the pyrolysis kinetics of five oil palm biomass (i.e., OPT, OPF, PKS, EFB, and MF) under single heating rate of 10 °C min⁻¹ also revealed that phase boundary mechanism dominated the thermal decomposition of pseudo-hemicellulose and pseudo-cellulose with nucleation as the side reaction.

Table 3 Reaction mechanisms of three oil palm biomass pyrolysis determined from Šesták-Berggren function.

| β (°C min⁻¹) | I  | II          | III         |
|-------------|----|-------------|-------------|
|             | lnA| n | p | Adj-R² | lnA| n | p | Adj-R² | lnA| n | p | Adj-R² |
| OPT         | 20 | 14.13 | 0.88 | 0.17 | 0.8094 | 18.83 | 5.22 | 1.15 | 0.9846 | 32.56 | 8.11 | 6.64 | 0.9435 |
|             | 30 | 15.23 | 2.27 | 0.50 | 0.8205 | 18.00 | 3.99 | 0.65 | 0.9956 | 32.26 | 7.78 | 6.30 | 0.9560 |
|             | 40 | 14.69 | 1.86 | 0.25 | 0.7175 | 17.29 | 2.86 | 0.42 | 0.9698 | 28.44 | 4.11 | 3.62 | 0.9698 |
| OPF         | 20 | 15.50 | 26.57 | 1.67 | 0.9880 | 18.83 | 5.22 | 1.15 | 0.9846 | 32.56 | 8.11 | 6.64 | 0.9435 |
|             | 30 | 10.50 | 12.74 | 0.20 | 0.9815 | 18.00 | 3.99 | 0.65 | 0.9956 | 32.26 | 7.78 | 6.30 | 0.9560 |
|             | 40 | 10.34 | 12.40 | 0.17 | 0.9980 | 17.29 | 2.86 | 0.42 | 0.9698 | 28.44 | 4.11 | 3.62 | 0.9698 |
| PKS         | 20 | 16.13 | 3.18 | 0.13 | 0.9951 | 22.94 | 5.24 | 4.83 | 0.9528 | 21.30 | 3.58 | 3.22 | 0.9765 |
|             | 30 | 16.00 | 2.83 | 0.05 | 0.9865 | 26.56 | 8.87 | 7.08 | 0.9432 | 21.30 | 3.58 | 3.22 | 0.9765 |
|             | 40 | 16.03 | 2.85 | 0.09 | 0.9975 | 21.30 | 3.58 | 3.22 | 0.9765 |

Conclusion
The pyrolysis behaviours and kinetics of OPT, OPF, and PKS under three different heating rates of 20, 30, 40 °C min⁻¹ were studied using thermogravimetric analysis (TGA). The resultant thermal weight loss profiles (TGs) and derivative weight loss profiles (DTGs) reveal that pyrolysis of the three oil palm biomass is complex as reflected by their multiple overlapping peak appearance. This was also indicated in the variation in apparent activation energy at different extent of conversion using the Kissinger-Akahira-Sunose (KAS) method which ranges between 17.83 and 116.58, -45.36 and 225.60, and 56.50 and 90.42 kJ mol⁻¹ for OPT, OPF, and PKS, respectively. Thermal decomposition of the pseudo-
hemicellulose and the pseudo-cellulose in the three oil palm biomass was co-controlled by phase boundary reaction and nucleation, with phase boundary reaction as the dominant mechanism.

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