

Valorisation of Napier Grass and Agricultural Residues via Pyrolysis – A Kinetic Study

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Thermal pyrolysis of Thai Napier grass, cassava stalk and rice straw were studied thermogravimetrically, which was followed by a kinetic analysis assuming multiple kinetic schemes and a distributed activation energy model (DAEM) with reaction order $n = 1$ and $n \neq 1$. The results showed that the DAEM described well the experimental data. The assumption of multiple kinetic schemes appeared more reasonable than that of similar studies reported in the literature assuming a single kinetic scheme. Consequently, the kinetic parameters extracted from the present study are more realistic and within reasonable ranges. Overall, the activation energies are within 31.6–223.53 kJ mol⁻¹. The reaction orders are within 1.02–1.32 for the assumption of $n \neq 1$.

1. Introduction

According to a report of the National Science Technology and Innovation Policy Office of Thailand, the total of agricultural residues in Thailand in 2013 was equivalent to 9.20 Mtoe, of which cassava stalk accounted for 0.50 Mtoe. On the other hand, approx. 8-14 Mt of rice straw becomes byproduct after paddy harvesting annually (Suramaythangkoo and Gheewala 2008). Lately, the Thai government has promoted a project of electricity production from energy crops with an emphasis on Napier grass and biogas production from Napier grass by anaerobic digestion (AD). However, AD is not capable of digesting the lignin component of grasses, which normally account for one thirds of the energy content of energy crops. The digestate can be used as fertilizer or solid fuel for heat and power generation via thermochemical conversion processes. In general, the digestate, Napier grass, cassava stalk and rice straw can be converted to thermal energy through combustion, of which the devolatilization (pyrolysis) is a fundamental step (Biagini et al. 2008). Therefore, it is important to study the thermal behaviour of these biomass materials during devolatilization. However, past studies on non-catalytic pyrolysis of Napier grass (Phuakpunk et al. 2020) and cassava stalk (Zhang et al. 2020; Jia et al. 2014) available in the literature were very limited. In addition, these studies assumed single lump kinetic schemes, which are less relevant for pyrolysis of complex lignocellulosic materials (Tran et al. 2014). For these reasons, the present study was carried out for better understandings of pyrolysis behaviour and kinetics of Napier grass and cassava stalk. Rice straw was included in this study for comparison, co-considering the seasonal variation of biomass and the consistence need of an energy alternate. The pyrolysis was studied by means of thermo-gravimetric analysis (TGA), associated with a kinetic modelling assuming multiple kinetic schemes followed with a non-linear regression analysis.

2. Material and experimental methods

Three biomass samples of Napier grass, cassava stalk and rice straw from Thailand were collected for this work. The samples were carefully washed with distilled water to eliminate contaminants. After that, the samples were dried at 378 K for 24 h, followed by grinding and sieving to obtain sample powders with particles sizes smaller than 125 μm . The use of these particle sizes is to ensure a kinetic control regime for the TGA experiments. The obtained powder was stored in plastic bags and kept at room temperature.

Table 1. Characterization of three biomass samples

Sample	Proximate analysis (wt%)				Ultimate analysis (wt%)			
	M	VM	FC	Ash	C	H	N	O
Cassava stalk	3.12	80.77	10.36	5.75	38.60	7.22	1.00	53.18
Napier grass	3.56	64.45	14.54	17.45	35.50	6.10	1.80	56.60
Rice Straw	3.20	69.23	9.36	18.21	34.33	5.96	0.96	58.75

M = Moisture; VM= volatile matter; FC= fixed carbon.

Fuel properties of the biomass materials under investigation are shown in Table 1, which include the data of ultimate analysis and proximate analysis. The content of carbon, hydrogen and nitrogen was determined by an elemental analyzer LECO Truspec CHN, whereas the oxygen content was calculated by difference. The proximate analysis was determined by using a thermogravimetric analyzer (NETZSCH STA 449F3). The proximate analysis procedure is as follows: first the samples were heated from room temperature to 383 K at a heating rate of $10^\circ\text{C min}^{-1}$ with a hold-up time of 10 min to obtain the weight loss associated with the moisture content; Subsequently, the temperature was ramped at $20^\circ\text{C min}^{-1}$ to 1173 K, at which the samples were held for 10 min to determine the weight loss associated with the volatile matters. Oxygen was then introduced into the furnace chamber to oxidize the formed charcoals. The weight loss during this period was attributed to the fixed carbon. The remaining material after combustion was regarded as the ash content (Jeguirim et al. 2014). Another thermogravimetric analyzer Pyris 1 (TGA), Perkin Elmer, was used to study the non-isothermal pyrolysis of the biomass samples in Nitrogen. For each TGA run, approximately 6-8 mg of a dried sample was loaded into a crucible located in the furnace of the TGA. Before heating, the furnace was purged with Nitrogen for 20 min to eliminate any possible air content. The furnace was then heated from room temperature to 873 K at a constant heating rate of $10^\circ\text{C min}^{-1}$ and a Nitrogen gas flow rate of 80 ml min^{-1} .

2.1 Kinetic modelling assuming distributed activation energy model

Thermal pyrolysis of biomass may be defined as the process of thermal treatment of the material at elevated temperatures in an inert environment. It is a common practice to describe the process by the global scheme of Eq. 1 (Tran et al. 2014).



The fundamental kinetic equation for the above scheme is represented by Eq. (2)

$$\frac{d\alpha}{dt} = A \cdot e^{-\frac{E_a}{RT}} \cdot f(\alpha) \quad (2)$$

where $f(\alpha)$ is the conversion function of conversion degree, α , which is determined as the mass fraction of the decomposed solid (mass loss) or released volatiles of samples. The conversion degree α is defined by Eq. (3)

$$\alpha = \frac{m_o - m_t}{m_o - m_f} \quad (3)$$

where m_o , m_t , and m_f are the initial mass, final mass, and the mass of the sample at time t , respectively. The conversion function $f(\alpha)$ is dependent on the reaction mechanism and its different forms can be found in the literature (Criado et al. 1989; Vlaev et al. 2003)

Various methods for estimating the kinetic parameters have been proposed and can be classified into three main groups based on the solution approaches: 1) simple algebraic models; 2) analytical model; and 3) integral model. The details of many different models have been intensively reviewed by several researchers (Di Blasi 1993; Moghtaderi 2006). Among others, the distributed activation energy model (DAEM) is believed to be the most accurate model. This model assumes that thermal pyrolysis consists of numerous independent parallel reactions with their own activation energy and pre-exponential coefficient. If it is simplified that all reactions share the same pre-exponential coefficient and the number of reactions is large enough so that the activation energy is then represented by a continuous distribution function $f(E)$ (Anthony and Howard 1976). Several forms of $f(E)$ can be applied to DAEM model including Gaussian, Gamma, Weibull and Logistic distribution (Cai and Liu 2008; Cai et al. 2011; De Caprariis et al. 2012). Among these, Gaussian function (Eq. 4) is most widely used following the popularization (Anthony et al. 1975).

$$f(E) = \frac{1}{\sigma\sqrt{2\pi}} \exp\left[-\frac{(E - E_0)^2}{2\sigma^2}\right] \quad (4)$$

where E_0 and σ are the mean activation energy and its standard deviation, respectively.

For the single DAEM model with the reaction order that equals to unity, $n = 1$ (Eq. 4a), or differs from unity, $n \neq 1$ (Eq. 4b), the conversion degree can be described as below (Anthony and Howard 1976):

$$\int_0^\infty \exp\left(-\int_{T_0}^T \frac{A}{\beta} \exp\left(-\frac{E}{RT}\right) dT\right) f(E) dE \quad (4a)$$

$$\alpha = -\int_0^\infty \left(-\frac{1-n}{\beta} \int_{T_0}^T \frac{A}{\beta} \exp\left(-\frac{E}{RT}\right) dT\right)^{\frac{1}{1-n}} f(E) dE \quad (4b)$$

In general, lignocellulosic biomass materials are mainly composed of three components including hemicellulose, cellulose and lignin. The chemical structure of hemicellulose is more complicated than that of cellulose and it undergoes the thermal degradation in the temperature range of 473- 623 K (Jensen et al. 1998; Shen et al. 2010). However, the degradation of cellulose occurs mainly in the range between 588 and 673 K (Yang et al. 2007). In addition, lignin is thermally the most stable and decomposes within a broad temperature range (Raveendran et al. 1996). It is therefore too simplistic to describe the thermal decomposition of lignocellulosic biomass material by a single DAEM model. Recently, the multi DAEM model has been adapted by researchers to represent three main components including hemicellulose, cellulose and lignin (Várhegyi et al. 2011; Cai et al. 2013; Tran et al. 2016). The reaction rate is then described by Eq. (5) wherein c_i is the contribution factor of i component.

$$\frac{d\alpha}{dt} = \sum_1^3 c_i \frac{d\alpha_i}{dt} \quad i = 1,2,3 \quad (5)$$

In addition to that, Napier grass contains considerable amount of protein and lipid, 27% and 14.8%, respectively (Dongmeza et al. 2009; García-Galván et al. 2012). In other words, Napier grass consists of five main components including protein and lipid apart from holocellulose and lignin. Therefore, it is reasonable to assume a multi DAEM model consisting of five pseudo-components for the pyrolysis of Napier grass. A similar approach done by Bui et al. was successful to reflect the pyrolysis kinetic of microalgae residues (Bui et al. 2016). Following this assumption, the pyrolysis can be kinetically represented by Eq. (6).

$$\frac{d\alpha}{dt} = \sum_1^5 c_i \frac{d\alpha_i}{dt} \quad i = 1,2,3,4,5 \quad (6)$$

In Eq. (5) and Eq. (6), the conversion degree of every pseudo-component follows Eq. (4a, b) and the overall conversion rate equals to the summation of partial conversion rates.

2.2 Numerical method

The non-linear least square method was applied to the kinetic modelling and simulation of the assumed models. The objective function to minimize is described in Eq. (7).

$$S = \sum_{i=1}^n \left[\left(\frac{d\alpha_i}{dt} \right)_{exp} - \left(\frac{d\alpha_i}{dt} \right)_{model} \right]^2 \quad (7)$$

where $\left(\frac{d\alpha_i}{dt}\right)_{exp}$ and $\left(\frac{d\alpha_i}{dt}\right)_{model}$ stand for the experimental and modelled conversion rate, N is the number of experimental points. The fit quality is appraised by Eq. (8) (Branca and Di Blasi 2003)

$$Fit (\%) = \left(1 - \frac{\sqrt{S}}{\left[\left(\frac{d\alpha_i}{dt} \right)_{exp} \right]_{max}} \right) \cdot 100\% \quad (8)$$

3. Results and discussion

Results from the kinetic analysis are presented in Figures 1, 2 and 3 for both cases of $n = 1$ and $n \neq 1$, whereas the extracted kinetic parameters are tabulated in Table 2. Note that the Napier grass curves and data are obtained from simulation assuming five pseudo-components, while only three pseudo-components were assumed for the cassava stalk and rice straw data. As can be visually inspected, the simulated curves fit well the experimental data. This is confirmed by the high fit qualities of around 99% in Table 2. The simulations with $n \neq 1$ allow small improvement in fit quality. For instance, it is just 0.22% increment in the case of Napier grass. Other investigations also confirmed that the reaction orders higher than one allowed better description of experimental data (Manyà et al. 2003; Bach, Tran et al. 2015). Nonetheless, other researchers argued that the pyrolysis process of lignocellulosic biomass materials can be represented by first order reaction, $n = 1$ (Broström,

Nordin et al. 2012). Indeed, for the case $n \neq 1$, the reaction order was not far away from unity for the present study.

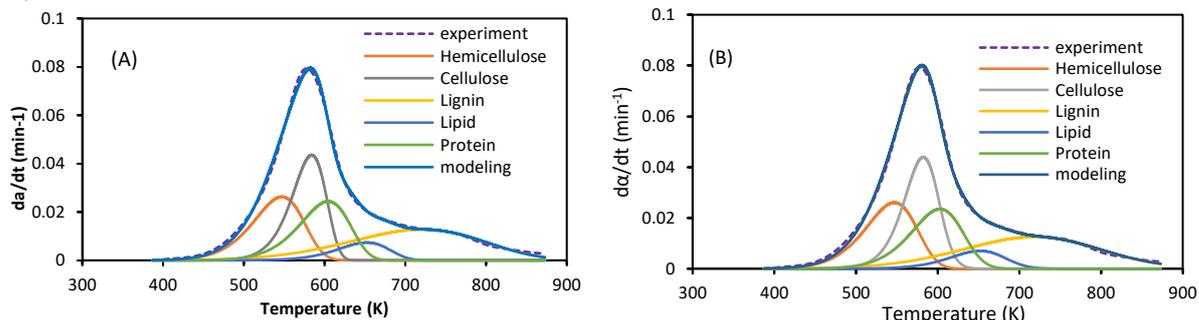


Figure 1: DAEM simulation for Napier grass with (A) $n=1$; (B) $n \neq 1$

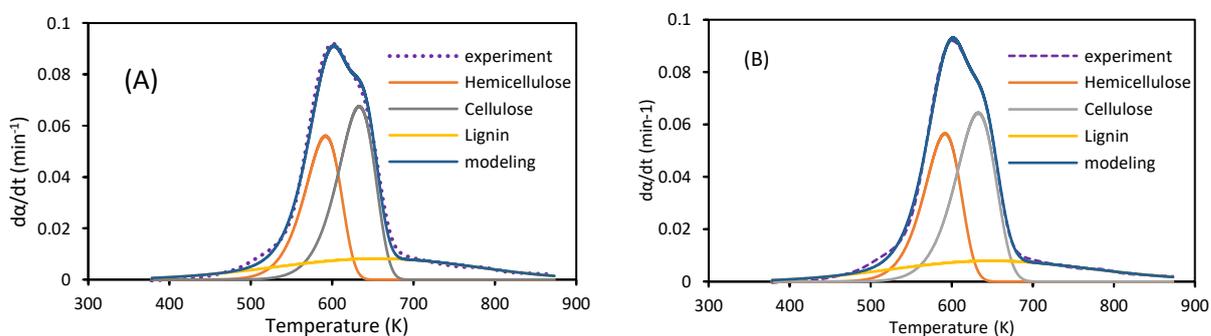


Figure 2: DAEM simulation for Cassava stalk with (A) $n=1$; (B) $n \neq 1$

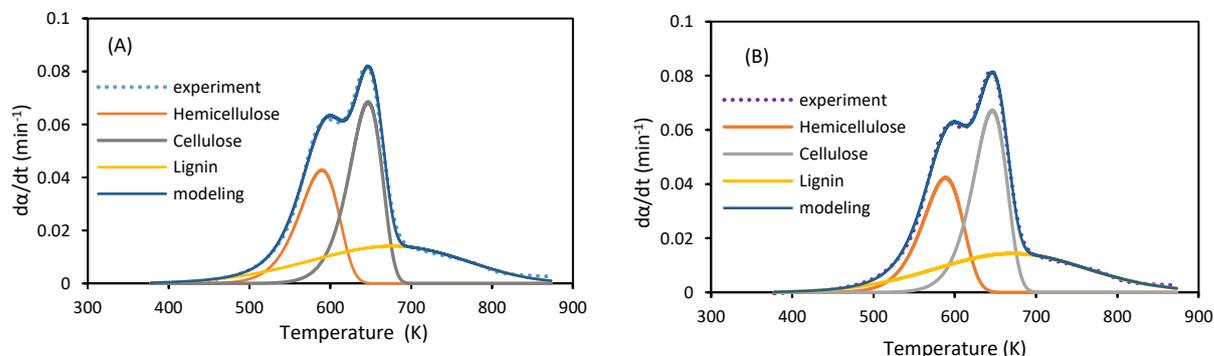


Figure 3: DAEM simulation for Rice Straw with (A) $n=1$; (B) $n \neq 1$

The calculated kinetic parameters shown in Table 2 are consistent with other studies (Grønli et al. 2002; Manyà et al. 2003). It is observed that the value of standard deviation for hemicellulose was highest among the others whereas that of lignin was smallest. It means that the activation energy of hemicellulose varies within a wider range than those for other pseudo-components. The mean activation energy of hemicellulose from Cassava stalk was about 167 kJ mol^{-1} , consistent with the literature of $154\text{-}165 \text{ kJ mol}^{-1}$ (Teng and Wei 1998). Nevertheless, it was claimed that the decomposition of hemicellulose is described by activation energy in the range of $80\text{-}90 \text{ kJ mol}^{-1}$ (Orfão et al. 1999) or $105\text{-}111 \text{ kJ mol}^{-1}$ (Várhegyi et al. 1997). Among the three samples, hemicellulose of the cassava stalk sample exhibited the highest activation energy, being within the range of $166.61\text{-}167.85 \text{ kJ mol}^{-1}$, followed by $149.21\text{-}151.27 \text{ kJ mol}^{-1}$ for the rice straw and $98.04\text{-}100.64 \text{ kJ mol}^{-1}$ for the Napier grass. However, cellulose of the rice straw sample had the highest activation energy, being within the range of $233.37\text{-}233.53 \text{ kJ mol}^{-1}$, followed by $176.21\text{-}187.02 \text{ kJ mol}^{-1}$ for the cassava stalk and $171.05\text{-}184.81 \text{ kJ mol}^{-1}$ for the Napier grass. When it comes to the activation energy of lignin component, the Napier grass sample showed the highest value within $61.10\text{-}64.41 \text{ kJ mol}^{-1}$, followed by $41.57\text{-}46.08 \text{ kJ mol}^{-1}$ for the rice straw and $31.60\text{-}33.24 \text{ kJ mol}^{-1}$ for the cassava stalk. In addition, the activation energy of lipid and protein component

of the Napier grass is within 147.14-148.10 kJ mol⁻¹ and 123.08-124.22 kJ mol⁻¹, respectively. The predicted content of these two components is 11% and 19%, respectively.

Table 2. Extracted kinetic data

Sample		$n = 1$					$n \neq 1$					
		A min ⁻¹	E_o kJmol ⁻¹	σ kJmol ⁻¹	c	Fit %	A min ⁻¹	E_o kJmol ⁻¹	σ kJmol ⁻¹	c	n	Fit %
Napier grass	H	7.52E+06	100.64	18.04	0.20	98.70	4.91E+06	98.04	17.58	0.20	1.02	98.93
	C	2.78E+11	171.05	30.66	0.24		2.79E+12	184.81	33.13	0.24	1.20	
	L	2.75E+02	61.10	10.95	0.26		4.47E+02	64.41	11.55	0.26	1.16	
	L _d	1.15E+08	147.14	22.34	0.11		1.57E+08	148.01	22.96	0.11	1.32	
	P	4.75E+07	123.08	22.06	0.19		5.94E+07	124.22	22.26	0.19	1.07	
Cassava stalk	H	9.00E+10	166.61	29.86	0.32	98.41	1.11E+11	167.85	30.09	0.33	1.05	99.00
	C	2.39E+11	187.02	32.57	0.44		5.66E+10	176.21	31.24	0.43	1.06	
	L	5.88E+00	31.60	5.68	0.24		7.98E+00	33.24	5.97	0.24	1.18	
Rice	H	5.14E+09	149.21	26.32	0.29	98.85	7.33E+09	151.27	26.65	0.29	1.04	99.15
Straw	C	1.53E+13	223.37	36.95	0.48		1.60E+13	223.53	36.98	0.48	1.04	
	L	4.16E+01	41.57	9.13	0.23		9.63E+01	46.08	10.24	0.23	1.20	

In another study, it was reported that the activation energy of Napier grass pyrolysis within 87.8 to 289.2 kJ mol⁻¹ for the conversion up to 70% (Phuakpunk et al. 2020). However, for the higher conversion, 80% and 90%, the reported values were 494.3 kJ mol⁻¹ and 887.2 kJ mol⁻¹, respectively. These values are unrealistic for lignocellulosic materials (Anca-Couce et al. 2020). From the present study, the activation energies for Napier grass are ranging from 61.10 to 184.81 kJ mol⁻¹ for the higher conversion, 80% and 90%, which are more realistic.

4. Conclusions

Thermal pyrolysis of the three types of Thai biomass including Napier grass, cassava stalk and rice straw was studied by means of a thermogravimetric analyzer for bioenergy application in Thailand. The collected TG data was employed for kinetic analyses assuming the multiple DAEM (five pseudo-components for the Napier grass, but only three for the cassava stalk and rice straw), with reaction order $n = 1$ and $n \neq 1$. The results showed that the multi DAEM model described well the experimental data with higher fitting quality. The assumption of multiple kinetic schemes appeared more reasonable than that of similar studies reported in the literature assuming a single kinetic scheme. Consequently, the kinetic parameters extracted from the present study are more realistic and within reasonable ranges.

Overall, the activation energies are within 31.6–223.53 kJ mol⁻¹. The activation energy of lignin is within 31.60–64.41 kJ mol⁻¹, followed by 98.40–167.85 kJ mol⁻¹ and 171.05–223.53 kJ mol⁻¹ for hemicellulose and cellulose, respectively. The reaction orders are within 1.02–1.32 for the assumption of $n \neq 1$. In addition, the activation energy of lipid and protein component of the Napier grass is within 147.14-148.10 kJ mol⁻¹ and 123.08-124.22 kJ mol⁻¹, respectively. The predicted content of these two components is 11% and 19%, respectively, which are also in good agreement with the literature. These data can be used as input data for modelling of energy systems as well as reactor and process design (Kempegowda et al. 2012).

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