Thermogravimetric and kinetic analysis of energy crop Jerusalem artichoke using the distributed activation energy model

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Abstract Jerusalem artichoke has great potential as future feedstock for bioenergy production because of its high tuber yield (up to 90 t ha^{-1}), appropriate biomass characteristics, low input demand, and positive environmental impact. The pyrolytic and kinetic characteristics of Jerusalem artichoke tubers were analyzed at heating rates of 5, 10, 20 and 30 °C min^{-1}. TG and DTG curves in an inert (nitrogen) atmosphere suggested that there were three distinct stages of mass loss and the major loss occurs between about 190–380 °C. Heating rate brought a lateral shift toward right in the temperature. And, it not only affects the temperature at which the highest mass loss rate reached, but also affect the maximum rate of mass loss. The distributed activation energy model (DAEM) was used to study the pyrolysis kinetics and provided reasonable fits to the experimental data. The activation energy (E) of tubers ranged from 146.40 to 232.45 kJ mol^{-1}, and the frequency factor (A) changed greatly corresponding to E values at different mass conversion.

Keywords Biomass · Jerusalem artichoke · Pyrolysis · Thermogravimetric · Distributed activation energy model

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Introduction

Biomass has been recognized as a potential renewable energy source and a substitute for the declining supply of fossil fuel resources [1]. As an energy group, Jerusalem artichoke obtained growing interests because of its high tuber yield (up to 90 t h^{-1}), appropriate biomass characteristics, low input demand and positive environmental impact [2]. JA can grow well in non-fertile land and is resistant to frost and plant diseases. Planting of this drought-resisting crop can also contribute to the improvement of soil and water conservation in desertified areas [3]. To date, JA has predominantly been cultivated in North America, Northern Europe, China, Korea, Australia, and New Zealand. In several provinces of China, such as Shandong, Qinghai, Shanxi, Heilongjiang, Jiangsu, Sichuan, Ningxia, and Xinjiang, the plant has been extensively cultivated to improve the salt-alkaline soils, oil-polluted soils, and coal-mining soils [4]. The large amount of available Jerusalem artichoke makes it an ideal candidate as the future feedstock for bioenergy production.

The thermal decomposition reactions play a crucial role during several of the biomass utilization processes. Thermogravimetric analysis (TG) is a high-precision method for the study of pyrolysis under well-defined conditions in the kinetic regime. Kinetic data from thermogravimetry analysis are not only useful for understanding the thermal degradation processes and mechanisms, but also can be used as input parameters for a thermal degradation reaction system. The extensive literature has been published on the experiments of great range of biomass, such as woods [5], agriculture residues [6], and municipal solid wastes [7]. However, to the author's knowledge, there is little information available about the pyrolysis kinetics of Jerusalem artichoke tubers.

Numerous models have been used for the analysis of thermal decomposition processes. A simplified model, known as distributed activation energy model (DAEM) is originally developed by Vand in 1943. The model assumes that many irreversible first-order parallel reactions that have different rate parameters occur simultaneously. Since then, it has been applied to analyze the complex reactions in thermal degradation of coal and activated carbon [8, 9]. Recently, the nth-order DAEM is proposed considering the influence of temperature on the frequency factor, which gives an excellent fit to the pyrolytic mass loss curves of several types of biomass without the assumption that all reactions share the same frequency factor (A) [10, 11]. It is also found that DAEM is more accurate than some pseudomechanistic model, especially when the decomposition is carried out under inert atmosphere [10, 12]. This approach led to favorable results and allowed predictions outside the experimental conditions of the experiments used in the parameter determination [13]. The objectives of this study were to investigate the pyrolysis behavior of Jerusalem artichoke tubers at different heating rates and obtain the distributed activation energies E, frequency factor A and coefficients corresponding to different mass conversions.

Methods

Materials

Jerusalem artichoke was collected from a saline land of Yantai, Shandong province, China. The tuber was oven-dried at 60 °C for 4 days, and then grounded with a Mini-Mill (Bilang Instrument Lim. Com., Shanghai, China) to pass through a 125 μm sieve.

Proximate and ultimate analysis

The moisture analysis was conducted according to ASTM E871–82 (2006). The ash content was determined according to ASTM E1755–01 (2007). The volatile matter content was analyzed according to ASTM E872–82 (2006). The fixed carbon was expressed as the 100 %-ash content-volatile matter-moisture content. The C, H, O, N, and S contents in the samples were measured using a vario microcube elemental analyzer. All measurements were replicated three times.

Thermogravimetric analysis

Jerusalem artichoke powders were analyzed by a Mettler Toledo TGA/DSC1 STARe thermo analyzer. The mass of sample for each test was in the range of 7–10 mg, and it was spread uniformly on the bottom of the alumina crucible of thermal analyzer. The pyrolysis experiments were performed at heating rates of 5, 10, 20, and 30 °C min^{-1} in a dynamic high purity nitrogen flow of 50 mL min^{-1}. The temperature of the furnace was programed to rise from room temperature to 1,000 °C, and the sampling time was set to 1 s per point.

Kinetic analysis using DAEM

Distributed activation energy model (DAEM) has been widely used in analyzing complex reaction system [12, 14]. In this study, it is assumed that the whole thermal conversion process of Jerusalem artichoke under inert atmosphere is composed of a set of irreversible single (first-order) reactions occurring successively. The model is expressed as:

$$1 - \frac{V}{V_\infty} = \int_0^\infty \Phi(E,T) f(E) \mathrm{d}E \qquad (1)$$

where

$$\Phi(E,T) = \exp\left(-\frac{A}{\beta} \int_{T_0}^T \mathrm{e}^{-E/RT} \mathrm{d}T \right) \qquad (2)$$

and

$$V = X_0 - X; \quad V_\infty = X_0 - X_\infty \qquad (3)$$

$f(E)$ is the normalized distribution curve of the activation energy, representing the variation of the activation energies of those first-order irreversible reactions. Through a numerical estimation by Miura [15], the $\Phi(E,T)$ function varies steeply with E at a given temperature T for the heating rate β. A step function U at an activation energy E_S for $\Phi(E,T)$ is postulated to give an approximation for Eq. (2) as:

$$\Phi(E,T) = U(E - E_S) \qquad (4)$$

And, the Eq. (1) could be simplified to:

$$1 - \frac{V}{V_\infty} = \int_{E_S}^\infty f(E) \mathrm{d}E \qquad (5)$$

This condition is found to hold approximately when E_S was chosen at $\Phi(E,T) = 0.58$ for many combinations of A and $f(E)$ from the preliminary examinations, giving the expression for determining A corresponding to activation energy E:

$$\frac{0.545\beta E}{ART^2} = \mathrm{e}^{-E/RT} \qquad (6)$$

together with the following approximate equation for:

$$\Phi(E,T) \cong \exp\left(-\frac{ART^2}{\beta E} \mathrm{e}^{-E/RT} \right) \qquad (7)$$

Table 1 Properties of the Jerusalem artichoke tuber and other biomass

Species	Biomass	Proximate analysis/% w/w				Reference
		Moisture	Volatile	Fixed carbon	Ash	
Terrestrial plant	Jerusalem artichoke tuber	2.8	74.6	18.0	4.6	This work
	Mimosa	1.6	71.1	23.6	3.7	[2]
	Rapeseed	8.4	70.0	15.8	5.8	[28]
	Arundo donax	8.2	68.4	18.4	5.0	[29]
	Miscanthus giganthus	10.0	78.8	9.5	2.7	
	Pine	4.2	79.0	16.6	0.2	[12]
	Birch	3.8	83.5	12.4	0.3	
Aquatic plant	*Pophyra yezoensis*	9.2	36.8	22.1	31.3	[16]
	Plocamium telfairiae Harv	11.7	30.6	24.3	33.2	
	Corallina pilulifera	10.5	32.2	18.4	38.6	
	Dunaliella tertiolecta	5.0	54.5	27.0	13.5	[17]
	Laminaria japonica	18.3	41.6	13.3	26.8	[23]
	Sargassum pallidum	10.3	44.6	8.3	36.4	

Fig. 1 Typical TG-DTG curves of Jerusalem artichoke at heating rate of 10 °C min^{-1}
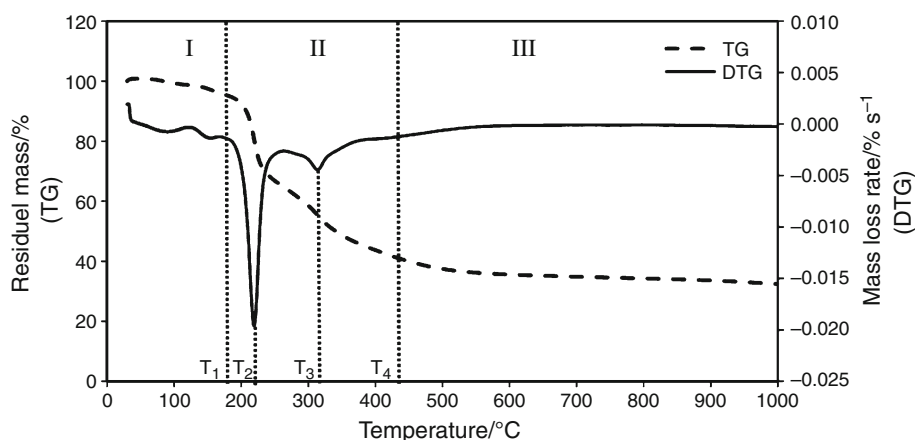
This treatment of approximation is given mathematically at the temperature T where the ith reaction occurs for a constant heating rate β by Miura and Maki (1998):

$$\frac{\mathrm{d}(V/V_\infty)}{\mathrm{d}T} \cong \frac{\mathrm{d}(V_i/V_\infty)}{\mathrm{d}T}$$
$$= \frac{A_i}{\beta}\exp(-E_i/RT)(V_{i\infty}/V_\infty - V_i/V_\infty) \quad (8)$$

It shows that the overall reaction rate could be represented approximately by the rate of ith reaction at the temperature T while only ith reaction is occurring, where V_i and $V_{i\infty}$, respectively, are the amount of volatiles evolved and the effective volatile for the ith reaction at the temperature T. Consequently, the whole reaction system of biomass decomposition could be approximated by a set of singe (first-order) reactions, occurring at different temperatures corresponding to the successive mass losses or solid conversions for a fixed heating rate.

Equation (8) could be integrated for a constant heating rate as:

$$1 - V_i/V_{i\infty} = \exp\left(-\frac{A_i}{\beta}\int_{T_0}^{T}\exp(-E_i/RT)\right)$$
$$\cong \exp\left(-\frac{A_i R T^2}{\beta E_i}\mathrm{e}^{-E_i/RT}\right) \quad (9)$$

The Eq. (9) could be rewritten as:

$$\ln\left(\frac{\beta}{T^2}\right) = \ln\left(\frac{AR}{E}\right) - \ln[-\ln(1 - V_i/V_{i\infty})] - \frac{E}{R}\frac{1}{T} \quad (10)$$

Since $1 - V_i/V_{i\infty} = \Phi(E, T) \cong 0.58$ was set for deriving the Eqs. (6), (10) could be simplified by

Table 2 Characteristics temperature associated with the pyrolysis process

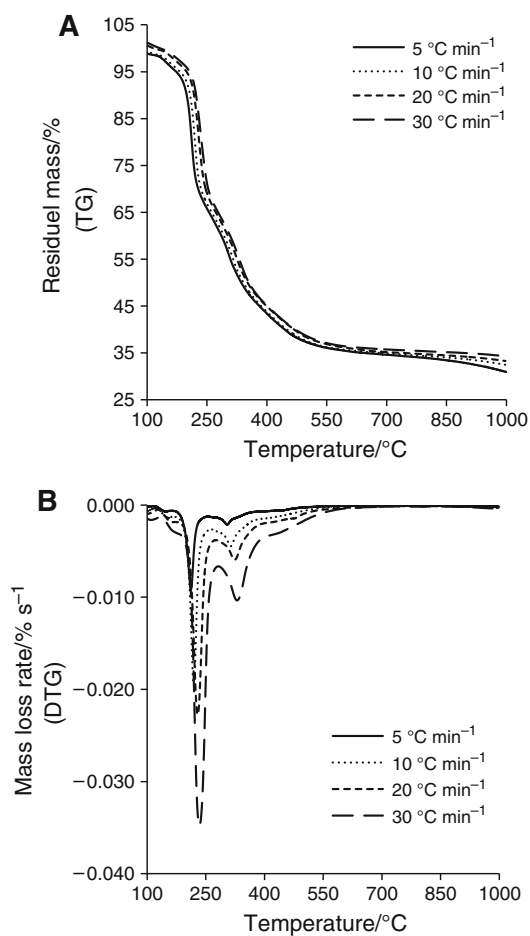| Heating rate/°C min^{-1}	T_1/°C	RM/%[a]	T_2/°C	RM/%[a]	T_3/°C	RM/%[a]	T_4/°C	RM/%[a]
5	184	93.86	212	80.27	305	55.90	349	48.26
10	189	94.37	219	80.84	316	54.95	370	46.48
20	195	95.42	229	81.41	324	55.49	388	45.99
30	198	95.98	233	82.33	333	54.52	398	45.24

[a] RM/% is the residual mass

Fig. 2 TG and DTG curves of Jerusalem artichoke at different heating rate against temperature ((**a**) TG curves, (**b**) DTG curves)

$$\ln\left(\frac{\beta}{T^2}\right) \ = \ \ln\left(\frac{AR}{E}\right) \ + \ 0.6075 \ - \ \frac{E}{R}\frac{1}{T} \qquad (11)$$

Equation (11) develops a linear relationship between $\ln(\beta/T^2)$ and $(1/T)$ with the slope of $(-E/R)$. Consequently, the activation energy for the reaction can be determined by the slope of the linear-fitting curve from the correlated experimental data, while the pre-exponential factor A is estimated from the intercept.
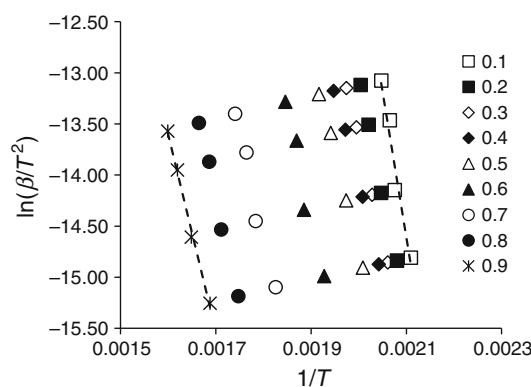

Fig. 3 Plots for determining activation energy at different conversion rate of stage II

Results and discussion

Proximate and ultimate analysis

In order to correlate the respective composition to the thermal behavior of the biomass materials under characterization, proximate and ultimate analysis were done. Proximate analysis showed that the moisture, ash, volatiles and fixed carbon contents of Jerusalem artichoke were 2.83, 4.56, 74.62 and 17.99 %, respectively. The proximate compositions of Jerusalem artichoke tuber were found to be similar with other terrestrial plants (Table 1), with lower moisture and ash content than other aquatic plants. Ultimate analysis showed that tuber consisted of moderately high carbon (40.85 %) and oxygen (32.16 %) content but low amounts of hydrogen (7.22%), nitrogen (1.70 %), and sulfur (0.09 %).

Characteristics of the thermal degradation process

A typical TG and DTG curves (Fig. 1) at heating rate of 10 °C min^{-1} indicated that the pyrolytic process was made up of three stages. Stage I occurred as the temperature increased from ambient to T_1, while stage II goes from temperature T_1 to T_4. Stage III occurred as the temperature

Table 3 The activation energies obtained at different conversion rate

m/m_0	E/kJ mol^{-1}	A/s^{-1}	R
0.1	232.45	7.97×10^{23}	0.9759
0.2	184.47	1.29×10^{18}	0.9983
0.3	160.41	1.67×10^{15}	0.9998
0.4	146.40	3.12×10^{13}	0.9990
0.5	152.01	6.76×10^{13}	0.9989
0.6	175.22	3.60×10^{15}	0.9807
0.7	166.97	5.42×10^{13}	0.9875
0.8	170.36	2.16×10^{13}	0.9969
0.9	156.42	2.93×10^{11}	0.9978
Average	171.63		

increased from T_4 to 700 °C. The temperature characteristics are shown in Table 2.

During stage I, the small change in loss of the mass is attributed to the loss of water and light volatile compounds in the biomass samples. Stage II is characterized by a major mass loss, and this loss mainly occurred between 190 and 380 °C. TG (Fig. 2a) and DTG (Fig. 2b) curves of the tuber samples exhibited two mass loss steps, with a similar pattern like three red algae [16]. Two peaks were observed in DTG curves, with the peak temperature at T_2 and T_3, and the rate of mass loss reached maximum at T_2. In this stage, most of the organic materials were gradually released, resulting in a large of mass loss (more than 65 % of total volatiles) and formation of the main pyrolytic products. In stage III, the carbonaceous matters decomposed at a slow rate.

Table 2 shows that the beginning of the decomposition occurs at about 190 °C, with a lower temperature than terrestrial biomass with a high content of cellulose (straws and grasses) [2] or lignin (woody biomass) [5] at the same

heating rate. Moreover, the temperature range of pyrolysis and the total mass of volatile matter for Jerusalem artichoke were all similar with those obtained from red [16] and marine algae samples [17].

Heating rate had an effect on the temperature range of the pyrolysis stages (Table 2). The TG curves showed that they shifted toward the right with the heating rate increased. This phenomenon was similar to a previous description of various biomass feedstocks [18–20], and it is typical to the non-isothermal pyrolysis process. It can be seen from Fig. 2b that the maximum rate of decomposition tends to increase at higher heating rate because there is more thermal energy to facilitate better heat transfer between the surroundings and the insides of the samples. The temperature corresponding to the maximum mass loss was all increased, and these temperatures were 212, 219, 229, and 233 °C at heating rate of 5, 10, 20, and 30 °C min^{-1}, respectively. In addition, as the heating rate increased, the volatile matter yields decreased in certain content for the Jerusalem artichoke samples. And, the volatile matter yield were 69.08, 67.54, 66.73, and 66.01 % for heating rate of 5, 10, 20, and 30 °C min^{-1}, respectively. Huang et al. [21] also proposed that higher heating rates provided less time for reactions to form more volatile products.

Kinetic analysis of the pyrolysis process

Figure 3 showed the representative plots for the main stage of mass loss (i.e., stage II), all plots had fairly high linear correlation coefficients greater than 0.97 (Table 3). The activation energies calculated by DAEM method were listed in Table 3. It can be seen that the activation energy revealed fluctuation at different conversion rate m/m_0. And, E values changed from 146.40 to 232.45 kJ mol^{-1}. The values of

Table 4 The activation energy of terrestrial and aquatic plant

Species	Biomass	E/kJ mol^{-1a}	Average	Reference
Terrestrial plant	Jerusalem artichoke	152.0–232.5	171.6	This work
	Corn stalk	163.0–225.6	203.0	[30]
	Sawdust	209.8–246.0	230.8	
	Birch	178.0–216.0	–	[12]
	Pine	191.0–250.0	–	
	Phragmites australis	90.7–592.1	291.8	[18]
Aquatic plant	*Dunaliella tertiolecta*	131.7–152.7	145.7	[17]
	Pophyra yezoensis	118.7–176.1	154.1	[16]
	Plocamium telfairiae Harv	153.0–320.8	244.7	
	Corallina pilulifera	191.9–291.2	250.7	
	Laminaria japonica	173.2–225.7	209.1	[23]
	Sargassum pallidum	151.2–302.6	203.5	

^{a} The E values quoted in this table were all calculated by DAEM or KAS method and all experiments were carried out under nitrogen atmosphere

A (from 2.93×10^{11} to 7.97×10^{23} s^{-1}) changed greatly with E values at different mass conversion. Because DAEM gives an excellent fit to the pyrolytic mass loss curves, it is plausible that the thermal decomposition of Jerusalem artichoke under inert atmosphere probably undergoes a set of single (first-order) reactions, which could be expressed by the DAEM. It also needs to be noted that DAEM reflects the E distribution of the whole pyrolysis process in terms of consecutive mass conversion in spite of heating rate.

The relationships of the conversion rate and the activation energy suggested that the activation energy decreased with conversion rate within the conversion rate range of 0.1–0.4, and increased when the conversion rate ranged from 0.4 to 0.6, then shifted little when the conversion rate ranged from 0.7 to 0.9. The activation energies exhibited the highest values at the conversion rate of 0.1 and the activation energies varied with the "W"- shape during the pyrolysis process. It is clearly that the activation energy of biomass pyrolysis is widely distributed, and each conversion rate has individual corresponding activation energy during the biomass pyrolysis (Table 3).

Biomass pyrolysis is complex processes due to differences in the chemical composition of components within the biomass material. Hence, no single kinetic model can explain universally the mechanism of thermal decomposition in all types of biomass. E values vary greatly with the kinetic approaches employed in pyrolysis analysis. Such as, Flynn–Wall–Ozawa develops a linear representation of lnβ versus $1/T$ with the slope of $(-1.0516\ E/R)$ [17]. Kissinger–Akahira–Sunose (KAS) [22] develops a linear relationship between ln(β/T^2) and $(1/T)$ with the slope of $(-E/R)$ [17, 23], which indicates that the E values evaluated by KAS are same with the values determined by DAEM (Table 3).

The activation energy of terrestrial and aquatic plant was summarized in Table 4. All the experiments were carried out under nitrogen atmosphere and E values were calculated by DAEM or KAS method. The activation energy of Jerusalem artichoke showed lower average E value than other terrestrial plants, some reports suggested that it may because the chemical composition of individual specie plays a fundamental role in the kinetics determination [2, 24]. However, relation between chemical composition and activation energy was not conclusive. Other possible explanation may be associated with the presence and absence of mineral content in various biomass materials [25–27]. The effects of mineral content in biomass ash need further investigation.

Conclusions

The proximate compositions of Jerusalem artichoke tuber were found to be similar with other terrestrial plants, with lower moisture and ash content than other aquatic plants.

And, the pyrolysis process of Jerusalem artichoke was composed of three stages and the major decomposition occurs between about 190 and 380 °C. Heating rate brought a lateral shift toward right in the temperature. And, it not only affected the temperature at which the highest mass loss rate reached, but also affected the maximum rate of mass loss. DAEM was used in kinetic analysis and provided reasonable fits to the experimental data. The activation energy of tubers ranged from 146.40 to 232.45 kJ mol^{-1}, and the frequency factor (A) values changed greatly corresponding to E values at different mass conversion.

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