

Research Article

Thermal Degradation Characteristics and Kinetics of Oxy Combustion of Corn Residues

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Thermogravimetric analysis was used to investigate oxy combustion of corncob and stover. The biomass samples were heated from ambient temperature to 900°C at different heating rates of 10, 30, and 50 K/min. Both biomass samples showed similar weight loss patterns with three zones, corresponding to dehydration, devolatilization, and char combustion, but displayed different degradation temperatures. Increasing heating rate was found to shift the degradation patterns to higher temperatures. Decomposition rates of cob and stover may have been influenced by their lignocellulosic composition. The kinetic parameters of the thermal degradation process were also determined and compared using the Flynn-Wall-Ozawa and Kissinger-Akahira-Sunose methods. Both methods were found to give similar values and patterns of activation energy against conversion fraction. The average values were found to be in similar magnitude to those reported in the literature, around 170 and 148 kJ/mol for cob and stover, respectively.

1. Introduction

Corn is an important agricultural crop in the world and the north of Thailand. It is widely planted for food production and animal feeding. After harvesting, there is a large amount of corn residues. Cob and stover (stalk and leaves) are the main residues. About 0.15 kg of cobs and 0.72 kg of stover are generated for every 1 kg of dry corn grains produced [1]. For Thailand, this resulted in the generation of about 0.73 and 3.50 million tons of corncob and stover in 2013 [2]. The residues are not normally utilized but left as waste or burned openly in the fields [3]. This open burning seriously pollutes the atmosphere, causes smog problem, affects human health, and also results in more CO₂ emission. These residues have great potential and should be harnessed as valuable feedstock for energy production.

The utility industry burning coal is the main sector responsible for major CO₂ emissions [4], which cause climate change and global warming. Utilization of biomass in coal fired power plants has gained great interest because of biomass's carbon neutrality and ability to lower SO_x emissions. Biomass cofiring and oxygen combustion can have

double effect in cutting down carbon emission from coal power plants.

Oxy combustion is an innovative combustion technology for burning fuel using pure oxygen instead of air. In this technique, because there is no nitrogen, NO_x is greatly reduced. The flue gas consists mainly of CO₂ and water vapor which can make it simpler for subsequent CO₂ capture and storage [5]. Combustion of biomass in highly oxidative atmosphere has not yet been widely investigated. Better understanding on its thermal degradation characteristics and burning kinetics is still needed. Thermogravimetric analysis is considered as a useful technique to access the thermal behavior and decomposition kinetics of biomass. It can determine thermal stability and fraction of volatile components of solid material by monitoring the change of weight that occurs as a sample is heated. There are many research works studied on potential biomass materials using thermogravimetric analysis [6–10]. It is well known that different decomposition atmospheres affect degradation behavior greatly. So far, most reported studies about the kinetics of biomass decomposition focused mainly on pyrolysis and combustion under nitrogen [11–14] and air atmospheres [15–18]. Only a few published works

TABLE I: Properties of corncob and stover on a dry basis.

	Proximate analysis			Ultimate analysis				Biochemical analysis			References
	Volatile	Fixed carbon	Ash	C	H	O	N	Cellulose	Hemicellulose	Lignin	
Corncob	80.2	16.7	3.1	49	6	44.7	0.3	—	—	—	[22]
	82.2	16.9	0.9	45.5	6.2	47	1.3	—	—	—	[23]
	—	—	—	47.4	5.9	38.1	0.7	30	38	3	[24]
Corn stover	74.9	17	8.2	47.4	5	38.1	0.8	—	—	—	[6]
	78.7	17.6	3.7	—	—	—	—	51.2	30.7	14.4	[25]
	—	—	—	46.6	5	40.1	0.8	48	29	6	[24]

were related to biomass combustion under oxygen [19, 20]. Investigations on oxy combustion of corn residues are still very rare.

The aim of this research is to investigate the combustion behavior of corncob and stover under oxygen environment. Nonisothermal thermogravimetric analysis is used to evaluate thermal characteristics and kinetics of corn residues under highly oxidative condition. The findings should provide useful data for future development of efficient biomass combustion applications.

2. Materials and Methods

2.1. Sample Preparation. Samples of corncob and stover were collected from agriculture land in Nan, Thailand. They were naturally dried, crushed to small particle size, and sieved with 120 meshes. The samples of biomass material were subsequently kept in a ziplock plastic bag and stored at room temperature until they were needed for experiment. Typical properties of the samples are shown in Table 1.

2.2. Thermal Degradation Experiment. The experiments were performed using a Mettler Toledo thermogravimetric analyzer model TGA/SDTA 851e at 0.1 μg resolution. The initial weight of 10 mg was placed in an alumina crucible. It was heated from room temperature to the final temperature of 900°C with continuously purged high purity research grade oxygen at a flow rate of 20 mL/min. The heating rates were performed at 10, 30, and 50 K/min. The instrument continuously monitored and recorded weight loss data and temperature every second. Experiments were repeated at least two times for each condition.

2.3. Kinetic Analysis. Kinetic analysis was carried out to determine apparent activation energy for the associated thermal degradation. The analytical techniques can be divided into two classes: model-fitting and model-free (isoconversional) kinetics. The model-fitting method is based on a single heating rate, which is a disadvantage because the activation energy varies with the heating rate, due to mass and heat transfer effects. The isoconversional method is preferred by researchers because, with the use of multiple heating rates, it is sufficiently flexible to allow for a change in mechanism during reactions and mass transfer limitations [21]. Therefore,

in this work the model-free kinetic methods by Flynn-Wall-Ozawa (FWO) and Kissinger-Akahira-Sunose (KAS) were adopted.

The degree of weight loss was shown as conversion, α , as follows:

$$\alpha = \frac{w_0 - w}{w_0 - w_f}, \quad (1)$$

where w_0 , w , and w_f are initial mass of sample, mass at any time, and final mass at the end of reaction, respectively. The kinetic equation of reactivity is generally described as

$$\frac{d\alpha}{dt} = kf(\alpha), \quad (2)$$

where $f(\alpha)$ is the reaction function and the reaction rate constant, k , is a temperature function defined by Arrhenius equation as

$$k = A \exp\left(\frac{-E}{RT}\right), \quad (3)$$

where A is the exponential factor, E is the apparent activation energy, and R is the molar gas constant, 8.314 J/mol·K. Combining (2) and (3) and rearranging with heating rate $\beta = d\alpha/dT$, nonisothermal kinetics can be written as

$$\frac{d\alpha}{dT} = \frac{A}{\beta} \exp\left(\frac{-E}{RT}\right) f(\alpha). \quad (4)$$

Integrated function of (4) is represented as

$$g(\alpha) = \int_0^\alpha \frac{d(\alpha)}{f(\alpha)} = \frac{A}{\beta} \int_{T_0}^T \exp\left(\frac{-E}{RT}\right) dT. \quad (5)$$

With the FWO [26] method using Doyle's approximation for the temperature integral, the following equation can be obtained:

$$\log \beta = \log\left(\frac{AE}{Rg(\alpha)}\right) - 2.315 - 0.4567\left(\frac{E}{RT}\right). \quad (6)$$

At a fixed value of α , $\log \beta$ was plotted against $1/T$ at different heating rates. A straight line should be obtained with slope equal to $-0.4567(E/R)$. The activation energy can be calculated. The KAS [26] is based on the Coats-Redfern approximation of the temperature integral; the equation is

$$\ln\left(\frac{\beta}{T^2}\right) = \ln\left(\frac{AR}{Eg(\alpha)}\right) - \frac{E}{RT}. \quad (7)$$

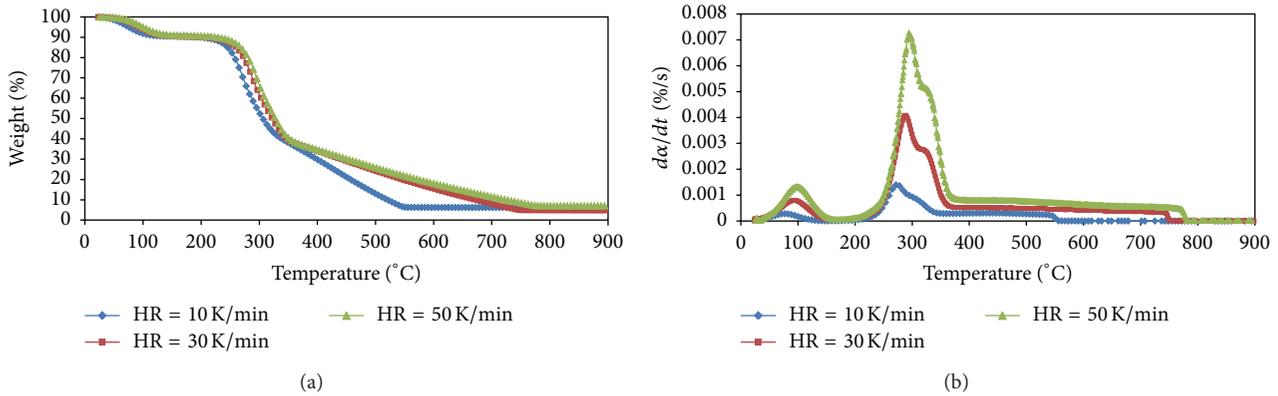


FIGURE 1: (a) TG and (b) DTG curves of corncob.

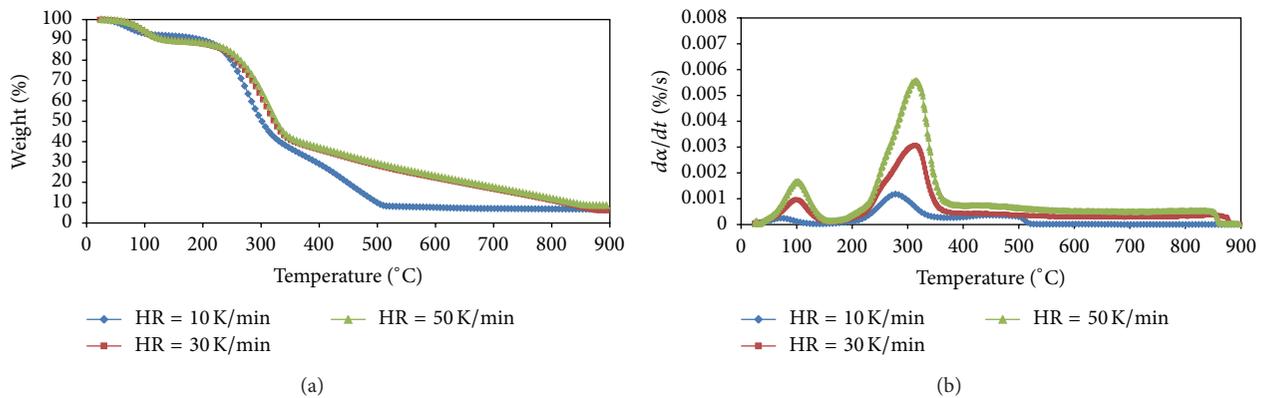


FIGURE 2: (a) TG and (b) DTG curves of corn stover.

Determination of the activation energy is similar to the previous technique; by plotting $\ln(\beta/T^2)$ against $1/T$, a straight line should be obtained with a slope of E/R and the activation energy can be calculated.

3. Results and Discussion

3.1. Thermal Degradation Characteristics. In this work, features of the thermal degradation characteristics were shown as the change of mass loss with temperature (TG) and mass loss rate (DTG) profiles. Nonisothermal thermogravimetric analysis of corncob was carried out at different heating rates of 10, 30, and 50 K/min in an oxygen atmosphere and the results are plotted in Figure 1. Thermogravimetric tests of corn stover were performed under similar conditions and the data plotted in Figure 2. It can be observed that the TG curves of both samples were similar, implying that the same reactions occurred at all heating rates. However, the temperatures corresponding to reactions were different and were shifted to a higher value with the higher heating rate. According to both TG results, increasing in temperature from ambient temperature to 900°C resulted in continuous weight loss and became constant at around 6–8% weight. The final product was ash. The differential TG curves gave three clear thermal degradation steps. This result was in agreement with previous

researchers [19]. The first step of mass loss occurred in the temperature up to around 150°C corresponding to release of moisture content in the samples. Afterward, there was a small change in weight and then rapid weight loss occurred again in the second stage from approximately 200°C to around 400°C, which was attributed to the evolution of the volatile compounds generated during decomposition of cellulose and hemicelluloses. The final stage occurred, corresponding to the combustion of the remaining lignin and char residue from the previous reactivity.

The temperature values at each stage and those characteristics in which the reactions took place are presented in Table 2. The second and third stages showed major weight loss, about 55 and 30% of the total weight loss. The start and final temperatures of the devolatilization zone of corncob were in the range of 190–340, 194–372, and 204–378°C. The peak temperatures were 271, 289, and 295°C for heating rates of 10, 30, and 50 K/min, respectively. The corn stover devolatilization temperature was in the ranges 167–368, 177–390, and 177–391°C with the peak temperature of 275, 313, and 315°C at the same condition. The values obtained for the peak temperature during the devolatilization step of the thermal degradation were in similar magnitude to those obtained by Mansaray and Ghaly [19] for rice husk at a heating rate of 20 K/min. Moliner et al. [27] also obtained peak temperatures

TABLE 2: Thermal degradation characteristics of corn residues under oxygen atmosphere.

Stage	Heating rate = 10 K/min					Heating rate = 30 K/min					Heating rate = 50 K/min					
	T_{range} (°C)	%wt	T_{peak} (°C)	$d\alpha/dt_{\text{peak}}$ (%/s)	α_{peak}	T_{range} (°C)	%wt	T_{peak} (°C)	$d\alpha/dt_{\text{peak}}$ (%/s)	α_{peak}	T_{range} (°C)	%wt	T_{peak} (°C)	$d\alpha/dt_{\text{peak}}$ (%/s)	α_{peak}	
Corn cob	1st	25	100	74	0.29×10^{-3}	5.05	25	100	92	0.8×10^{-3}	5.22	25	100	98	1.32×10^{-3}	5.06
		139	90.6				163	90.32				163	90.85			
	2nd	190	90.18	271	1.42×10^{-3}	31.23	194	90.08	289	4.1×10^{-3}	31.57	204	90.54	295	7.2×10^{-3}	31.95
		340	39.75				372	36.76				378	36.59			
	3rd	340	39.75	—			372	36.76	—			378	36.59			
		550	6.38				747	4.92				776	7.1			
Corn stover	1st	25	100	69	0.26×10^{-3}	3.84	25	100	98	0.9×10^{-3}	6.05	25	100	101	1.66×10^{-3}	6.18
		132	92.21				157	89.22				161	89.4			
	2nd	167	91.5	275	1.17×10^{-3}	37.24	177	88.83	313	3.1×10^{-3}	47.96	177	89.14	315	5.58×10^{-3}	47.32
		368	34.91				390	36.89				391	37.81			
	3rd	368	34.91	—			390	36.89	—			391	37.81	—		
		514	8.41				878	6.27				858	8.86			

of 271, 273, 288, 292, and 297°C at heating rates of 2, 5, 10, 15, and 20 K/min, respectively.

It can be seen that the peak devolatilization temperature of stover was higher than cob but the mass loss rate was smaller. The thermal degradation characteristic was affected by the chemical composition of biomass. Difference in their constituent components resulted in different thermal behavior. According to the biochemical composition, both kinds of feedstock samples contained small lignin content, but the fractions of cellulose and hemicelluloses were relatively distinct. This indicated that different behaviors were expected in the devolatilization process. The thermal decomposition of the individual components of lignocellulosic materials was studied by several researchers [28–30] under different atmospheres. These results indicated that decomposition of hemicellulose starts first, followed by the cellulose and lignin. The lower peak temperature of cob can be explained by its higher hemicelluloses and lower cellulose content, compared with stover. Lignin decomposes within a wider range of temperatures and is considered to be the significant contributor for the production of char residue. There is similar lignin content in the two kinds of feedstock; therefore, the same amount of char residue was realized.

The degradation rate of the char combustion process in the third stage was lower than the devolatilization of the second step. The degradation rates of cob and stover were similar. Degradation of stover appeared to have a higher starting temperature and ends at a higher final temperature than cob. At a relatively high heating rate, the combustion of these char occurred within a much wider temperature range. A constant combustion rate was also observed. This observation generally agreed with the results reported by other researchers [16].

The decomposition process was shifted to a higher temperature zone as the heating rate increased. The peak degradation temperature during the first weight loss stage of cob and stover was increased from 74 and 69 to 98 and 101°C, respectively. For the second mass loss stage, it was increased

from 271 and 275 to 295 and 315°C, respectively. The translation of the TG curves to higher temperature at higher heating rates was due to the reduction of residence time which was not sufficient for heat transfer to permeate into the center of the particle. Thus, the thermal decomposition process was delayed. To have the same weight loss, the degradation temperature was expected to be higher. Gai et al. [8] reported that this may have been due to the fact that, with the increase of heating rate, the time taken to reach the settled final temperature was less. Consequently, the heat transfer from the heating furnace within the thermogravimetric instrument to the tested sample may be limited, which may bring about a discrepancy between the recorded temperature and the actual sample temperature, agreed to by other researchers [16, 17, 31].

3.2. Kinetic Analysis. From TG analysis data, the kinetic parameters of overall weight loss were derived from the FWO and KAS methods. Figure 3 shows the evolution of mass loss as a function of temperature. Conversions of 0–15 and 85–100% were normally associated with the moisture release and the mineral matter decomposition. The conversion ranging between 20 and 80% was related to the volatile release and char combustion. Therefore, this conversion range was considered for the subsequent calculation, according to (6) and (7). Linear regression was plotted and shown in Figures 4 and 5. The apparent activation energy (E_a) can be calculated from the slopes, shown in Figure 6. Within the range of conversions considered, the values of activation energy obtained from both methods were similar. The FWO method appeared to give slightly higher E_a than those from the KAS method. This was due to the distinctive linear approximation to the temperature integral. It was observed that the apparent activation energy of both samples varied with conversion fractions, showing similar trends. They showed an initial ascending trend in the range of 30–60% conversion and decreasing trend thereafter. The activation energies of corncob were around 77–249 and 69–252 kJ/mol with average value of 172 and 171 kJ/mol for the FWO and

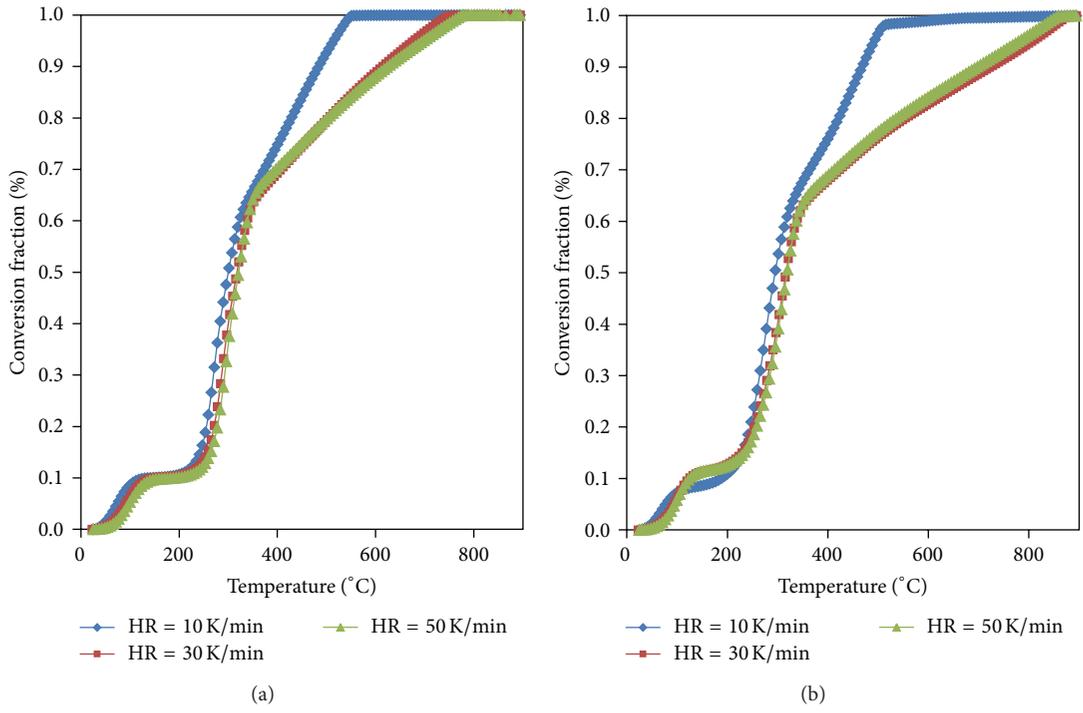


FIGURE 3: Conversion at any temperature: (a) cob, (b) stover.

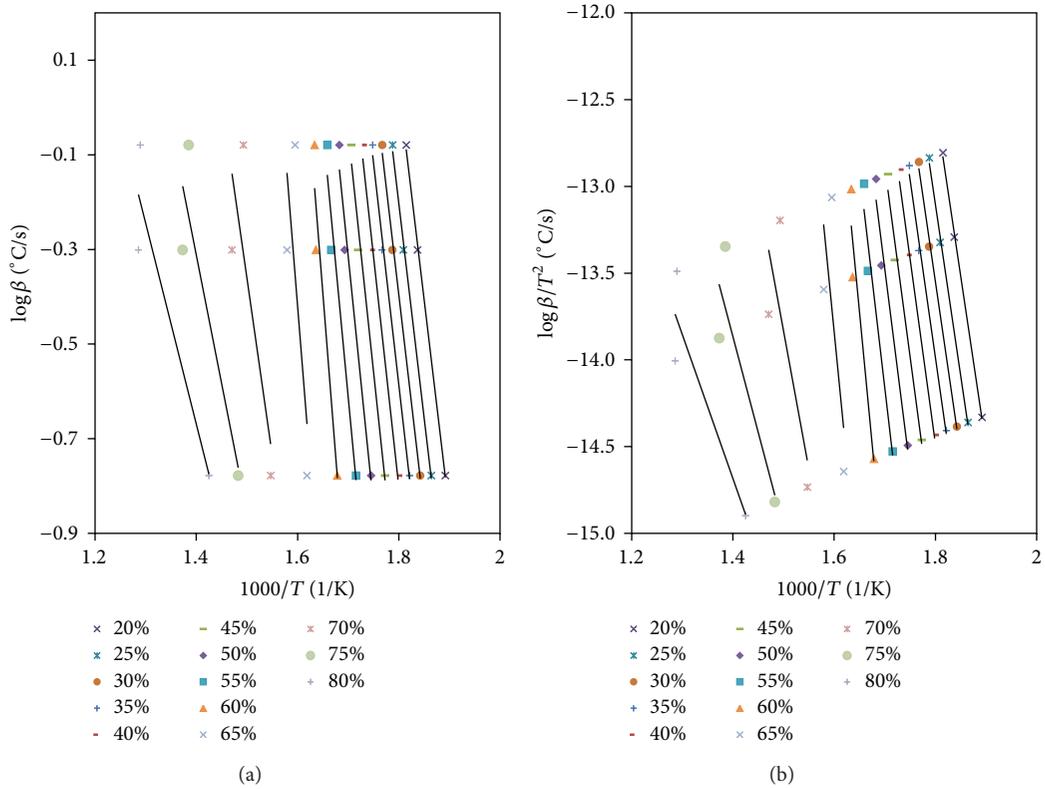


FIGURE 4: Linear plots for determination of the activation energy at different values conversion for corncob: (a) FWO method and (b) KAS method.

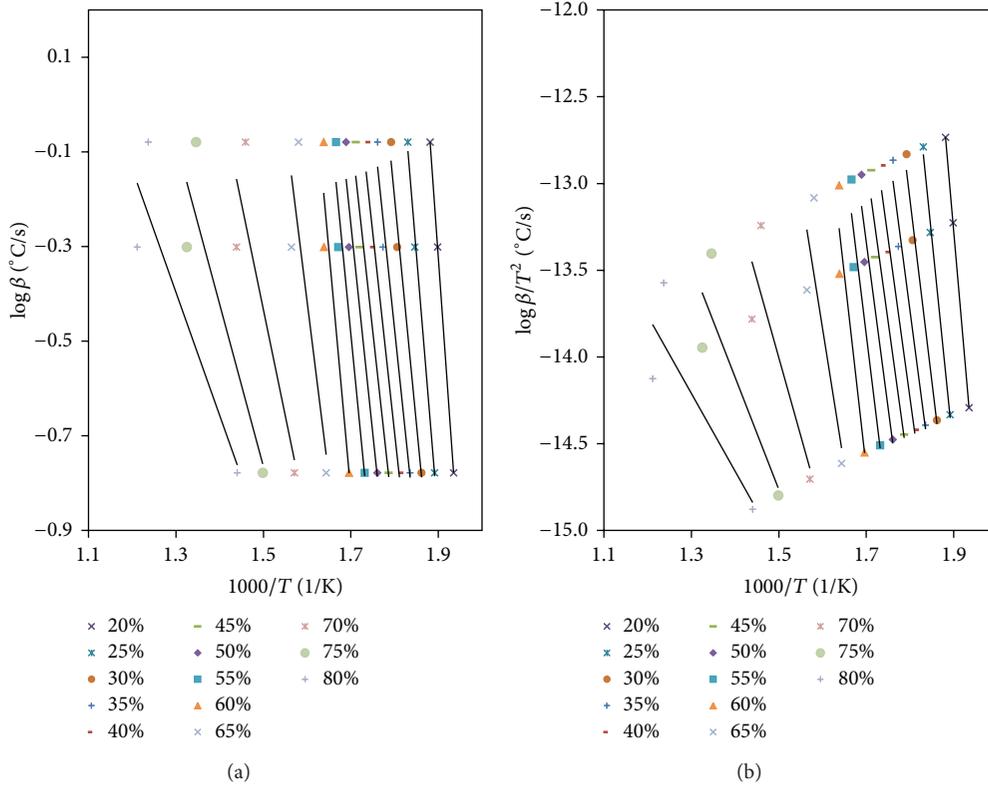


FIGURE 5: Linear plots for determination of the activation energy at different values of conversion for corn stover: (a) FWO method and (b) KAS method.

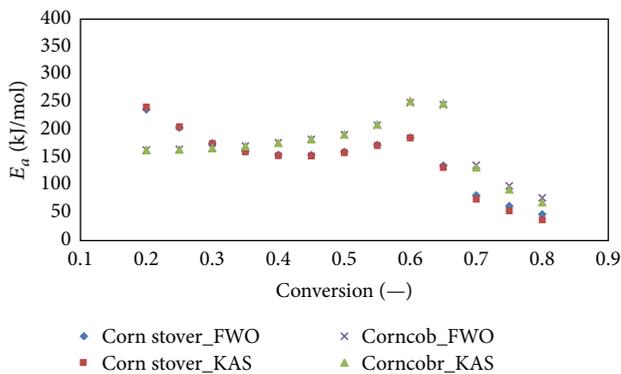


FIGURE 6: Variation of apparent activation energy of corn residues with conversion rate.

KAS methods, respectively. For corn stover, it was found to be around 47–237 and 37–241 kJ/mol with average value of 149 and 146 kJ/mol for the FWO and KAS methods. This means that the reaction mechanism varied considerably in an oxidative decomposition process. The activation energy was strongly dependent on conversion.

The characteristic of devolatilization occurred in conversions of around 20–60%. The average E_a values of cob and stover in this step were 186 and 178 kJ/mol. Cellulose and hemicelluloses were decomposed in this range. Huang et al. [32] suggested that the apparent activation energy

of cellulose was the highest, followed by hemicelluloses and lignin, respectively. The increase in activation energy at higher conversion may be attributed to ignition and oxidation of char residues. After 60% conversion range, char combustion took place. The average E_a values in this step were 139 and 82 kJ/mol for cob and stover, respectively. The lower kinetic values of E_a obtained in the oxidation reaction zone, compared to those obtained in the devolatilization reaction zone, may be due to the fact that lignin which had lower decomposition rate than cellulose and hemicelluloses components was condensed to char [19]. There may also have been a catalytic effect from inorganic elements of ash on the combustion of char [33]. Mansaray and Ghaly [19] and Harun et al. [20] also reported lower E_a in this zone for rice husk and bagasse, while the E_a for combustion of sawdust was higher than those from the devolatilization. Leroy et al. [34] suggested that for conversions between 10 and 30% characterized processes involve competing reactions such as dehydration and pyrolysis. Between 30 and 60%, pyrolysis and smoldering combustion were in competition. For 60–90%, it was the transition of process to diffusion regime.

4. Conclusions

Thermal degradation of corncob and stover under a highly oxidative environment was investigated using TGA at different heating rates of 10, 30, and 50 K/min. Kinetic parameter

in terms of the apparent activation energy was determined and compared using the FWO and KAS methods. Oxidative thermal degradation of both samples was found to occur in three mass loss stages: water evaporation, devolatilization, and char combustion. Increasing heating rate was observed to result in increasing mass loss rates, but the start of the thermal decomposition was delayed to higher temperatures. Different lignocellulosic composition of the feedstock may affect degradation behavior differently. Stover appeared to have higher hemicellulose and lower cellulose contents than cob and, hence, lower initial degradation temperatures. From the FWO and KAS methods, the activation energy was found to vary with the conversion fraction. The activation energy calculated by the FWO and KAS methods was similar. The average was about 172 and 149 kJ/mol for cob and stover, respectively.

Nomenclature

- A : Preexponential factor (s^{-1})
 E_a : Apparent activation energy (kJ/mol)
 f : Reaction function
 g : Integral function of conversion
 k : Reaction rate constant (s^{-1})
 R : Universal gas constant (8.314×10^{-3} kJ/mol·K)
 T : Temperature (K)
 T_0 : Initial temperature (K)
 w_0 : Initial mass of sample (kg)
 w : Mass at any time (kg)
 w_f : Final mass at the end of reaction (kg)
 α : Degree of conversion (—)
 β : Heating rate (K/min).

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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