

THERMOGRAVIMETRIC ANALYSIS OF THERMAL AND KINETIC BEHAVIOR OF *ACACIA MANGIUM* WOOD

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(Received December 2014)

Abstract. Thermogravimetric analysis was used to study the thermal behavior of *Acacia mangium* wood under inert atmosphere at heating rates ranging from 5 to 15°C min⁻¹, from room temperature to 800°C. Weight losses of *A. mangium* wood in inert atmosphere were found to occur in three stages. These three states are generally associated with the decomposition of the three main components of the lignocellulosic materials. Most decomposition occurred in the range of 300-400°C (80% weight approximately). The kinetic parameters of the process were evaluated using the independent parallel reaction model, attributed to the three main components of lignocellulosic materials: hemicelluloses, cellulose, and lignin. The values of activation energy, preexponential factor, and contribution factor were similar to those reported in other studies for this type of biomass. The model proposed predicted an acceptable correlation between the experimental and the calculated curve to the decomposition of *A. mangium*, with an error of less than 3% of deviation in the temperature range studied.

Keywords: Biomass, kinetic, pyrolysis, thermogravimetric analysis, *Acacia mangium*.

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INTRODUCTION

The forest products industry generates residues in the form of some of the most socially and environmentally beneficial biomass resources. Energy crops, harvested solely for their energy content, present additional fuel resources with total biomass energy potential. As long as forest products industries grow new plants at a rate greater than or equal to the harvest rate and thereby produce residues and energy crops in a sustained manner, no increase in atmospheric CO₂ is associated with their use as a fuel (McKendry 2002).

Biomass is currently a promising renewable energy and is potentially neutral in relation to global warming. Plant materials can be used with thermochemical conversion processes aimed at the production of fuel gases, chemicals, and energy (Senelwa and Sims 1999).

In its strategy for 2020, Cuba's forest policy takes into account the use of tools for new forest plantations, including using fast-growing species for energy purposes on 500 kha, allowing the area to raise forest cover from 21% to 29%, which means moving from 2.4 to 3.08 Mha of forest (Herrero 2010).

The development of tropical tree plantations has been on the rise in recent decades, presumably to meet the ever-increasing demands for timber and firewood. A wide spectrum of tree species, usually described as multipurpose trees (MPT), is often involved in such programs. Important attributes of MPT include rapid juvenile growth, efficient dry matter production in terms of water and nutrient inputs, crown characteristics to maximize interception of solar radiation, and ease of regeneration by coppicing (Fege 1981).

Among the fast-growing species is *Acacia mangium*, introduced in Cuba, which may be a promising species for energy production. This species has been planted extensively in the humid tropics of southeast Asia because of its fast growth in a wide range of sites, including degraded land, and acceptable quality of wood for pulp and paper (Turnbull et al 1988). This

plant would be a possible alternative to wood for short rotation forestry.

During recent decades, extensive research has been carried out in the field of biomass pyrolysis. Thermal decomposition of the major constituents (cellulose, hemicellulose, and lignin) has been extensively studied (Szabó et al 1996; Caballero et al 1997; Meszaros et al 2004; Abreu Naranjo et al 2012). The weight loss was generally attributed to moisture loss, hemicelluloses and cellulose decomposition, and lignin decomposition during the slow pyrolysis of lignocellulose materials in inert atmosphere (Jeguirim and Trouvé 2009). Other investigations on thermal decomposition of the basic constituents of wood have reported that there were temperature intervals at which the decomposition of the single constituent was dominant (Gronli et al 2002). According to results, thermal decomposition of these chief components took place in the following temperature intervals: at $T < 230^{\circ}\text{C}$, the decomposition of the hemicelluloses dominated; at $230^{\circ}\text{C} < T < 260^{\circ}\text{C}$, the hemicelluloses as well as the cellulose were decomposed; at $230^{\circ}\text{C} < T < 290^{\circ}\text{C}$, the dominant decomposition corresponded to the cellulose; and at $T > 290^{\circ}\text{C}$, the cellulose, as well as the lignin, was decomposed. However, the decomposition range can vary according to the nature of material, experimental heating rate, and other factors (Meszaros et al 2004; Branca et al 2005).

Thermal decomposition reactions play a crucial role during several of the biomass utilization processes. Thermogravimetric analysis (TGA) is a high-precision method for the study of pyrolysis under well-defined conditions in the kinetic regime. It has been widely used to characterize forest residues. It can provide information about the partial processes and reaction kinetics. The biomass fuels and raw materials contain a wide variety of pyrolysate species (Bridgwater 2003; Czernik and Bridgwater 2004).

Acacia mangium has great potential as an energy crop because it is a fast-growing species that does not require annual reseeded and agricultural inputs. It can grow in poor soils and low fertility.

However, for the development of technically and economically feasible systems for the conversion of *A. mangium* by thermochemical processes, it is fundamental that knowledge of the kinetic behavior be obtained. In general, the design of pyrolysis units depends on the residence times required for complete conversion of biomass particle feed. The residence time is, in its turn, the result of heat, mass, and momentum transfer and solid degradation rate in the reacting environment. Of course, this can be predicted only if the chemical kinetics are known (Di Blasi and Lanzetta 1997).

The purpose of this study was to investigate the thermal behavior of *A. mangium*, including a kinetic approach by means of thermogravimetric measurements and determination of kinetic parameters.

MATERIALS AND METHODS

Equipment and Procedures

The samples were obtained from a short rotation forestry plantation in Pinar del Río, Cuba. The samples originated from young *A. mangium* and were milled to an average size of 2.36-0.04 mm in accordance with ASTM (2007b).

Proximate and ultimate analyses were carried out to characterize the samples of *A. mangium*. These were done by the methods described by the appropriate standards (Table 1).

Lignin (Klason lignin) and cellulose contents were determined according to Kürshener and Höffer methods (Browning 1967). Thermogravimetric analyses were carried out with a thermo balance (TGA-TA Instruments 2050, New

Castle, DE). These analyses were carried out at heating rates of 5, 10, and 15°C min⁻¹ under argon flux of 100 mL min⁻¹ from room temperature to about 800°C. Mass of the initial samples was approximately 8 mg. A small mass was used to decrease the heat and mass transfer effects, in the kinetic control (Branca et al 2002).

Kinetic Model for Pyrolysis

Determining the kinetics of biomass thermal decomposition is difficult because of the large number of reactions involved. Therefore, simplified kinetic models are generally applied. In this study, the pyrolysis process is described by a three independent parallel reaction model. This is considered the most realistic approach in the case of lignocellulosic materials (Orfão et al 1999; Caballero and Conesa 2005). According to this model, the decomposition of biomass is associated with its chief pseudo-components, hemicellulose, cellulose, and lignin (Caballero et al 1997; Várhegyi et al 1997; Orfão et al 1999; Gronli et al 2002). In this way, the following scheme was assumed to model the kinetic parameters:



where F_i represents different fractions or pseudo-components of the solid material, V_i are volatile and condensable gases released in the corresponding reactions ($i = 1-3$), and S_i is charcoal formed from the decomposition of each F_i ($i = 1-3$).

With respect to an n th order kinetic decomposition, the equation for the pyrolysis can be stated as follows:

$$-\frac{dw_{s_i}}{dt} = k_i w_{s_i}^{n_i} \quad (2)$$

or

$$\frac{dV_i}{dt} = k_i (V_{i\infty} - V_i)^{n_i} \quad (3)$$

Weight loss can be expressed as the ratio $\frac{w_f}{w_o}$ (weight fraction).

Table 1. Standards used for determining the parameter in the proximate and ultimate analyses of *Acacia mangium* samples.

Standard	Parameter
ASTM-E1757-01 (2007b)	Preparation of biomass
ASTM-E777 (2006a)	Carbon and hydrogen
ASTM-E778 (2006b)	Nitrogen
ASTM-E871-82 (2006c)	Moisture
ASTM-D1102-84 (2007a)	Ash
ASTM-E872-82 (2006d)	Volatile matter

To the kinetic constant, the Arrhenius equation is applied as follows:

$$K_i = K_{i0} \exp\left(-\frac{E_i}{RT}\right) \quad i = 1, 2, 3 \quad (4)$$

Replacing 4 into 2 and expressed as a function of temperature gives the following:

$$\frac{dw_i}{dT} = -\frac{K_{i0}}{\beta} \exp^{-\frac{E_i}{RT}} w_i^{n_i} \quad (5)$$

Integrating,

$$\begin{aligned} f(W, T) &= W_i(T_o + \Delta T) \\ &= W_i(T_o) - \Delta T \frac{K_{i0}}{\beta} \exp^{-\frac{E_i}{RT}} w_i^{n_i} \end{aligned} \quad (6)$$

The kinetic model was simultaneously adjusted using data at the three heating rates, which were studied under dynamic conditions to decrease compensation in the estimation of kinetic parameters. The calculated values were obtained from Eq 6, by applying an integral method or numerical method, specifically the integration method Runge–Kutta 4th order (Caballero and Conesa 2005). The time intervals considered were small for the error to be negligible.

The Solver function in Excel was used to minimize the difference between experimental and calculated curves. The minimized objective function is expressed as

$$O.F = \sum_j \sum_i \left[\frac{\left(\left(\frac{dw}{dt} \right)_{\text{exp}} - \left(\frac{dw}{dt} \right)_{\text{cal}} \right)}{\max \left(\frac{dw}{dx} \right)_{j \text{exp}}} \right]^2 \quad (7)$$

where i represents the experimental data in time t to the heating rate j and the values of $\left(\frac{dw}{dt} \right)_{\text{exp}}$ and $\left(\frac{dw}{dt} \right)_{\text{cal}}$ are the derivatives with respect to experimental and calculated weight loss, respectively. The denominator in Eq 7 is the maximum experimental value of each heating rate and is considered to minimize the differences in the optimization process (Gullón et al 2004; Moltó et al 2009).

The deviation between the experimental and calculated curve is defined in correspondence to previous studies (Gronli et al 2002; Branca et al 2005; Abreu Naranjo et al 2012).

$$\text{fit}(\%) = \frac{\sqrt{S/N}}{\max \left(\frac{dw}{dt} \right)_{\text{exp}}} \times 100 \quad (8)$$

$$S = \sum_{j=1, N} \left[\left(\frac{dw}{dt} \right)_{\text{exp}} - \left(\frac{dw}{dt} \right)_{\text{cal}} \right]^2 \quad (9)$$

where N is number of experimental points or measures.

RESULTS AND DISCUSSION

Chemical–Physical Characterization of *A. mangium* Wood

The volatile matter, fixed carbon, and ash content values obtained from the elementary analysis for the studied species (Table 2) were similar to those reported for *Dichrostachys cinerea* (77.3%, 19.4%, and 3.4%), *Leucaena leucocephala* (79.9%, 17.7%, and 2.4%), and *Lysiloma latisiliquum* (77.8%, 20.7%, and 1.5%), although that of ash content was lower in *A. mangium* compared with the three species cited (Suárez et al 2000; Abreu Naranjo et al 2010).

Nevertheless, a marked similarity was found with the values reported by Gronli et al (2002) to the redwood sample, with volatile matter and fixed carbon contents of 82.3% and 17.5%, respectively, but not the same result for the ash content of 0.2%, which is lower than the obtained for the *A. mangium* samples. Also,

Table 2. Proximate and ultimate analyses of *Acacia mangium*.

Ultimate analyses	Mass (%)	Proximate analyses	Mass (%)
C	45.45 ± 0.24	Moisture	3.78 ± 0.16
H	6.30 ± 0.08	Ash	0.97 ± 0.05
N	0.23 ± 0.01	Volatiles	82.43 ± 0.38
O ^a	48.26 ± 0.34	Fixed Carbon	17.57 ± 0.40

Table 3. Chemical composition of the main components of the *Acacia mangium* wood.

Polysaccharides (%)	Cellulose	44.05 ± 0.3
	Xylans	14.05 ± 0.07
	Galactans	0.55 ± 0.07
	Mannans	1.30 ± 0.14
	Arabinans	0.25 ± 0.07
Lignins (%)	Insoluble	28.25 ± 0.21
	Soluble	1.45 ± 0.07
	Total	29.70 ± 0.14
Extractable substances = ([alcohol:toluene 1:2], [alcohol] y [hot water])		4.45 ± 0.07

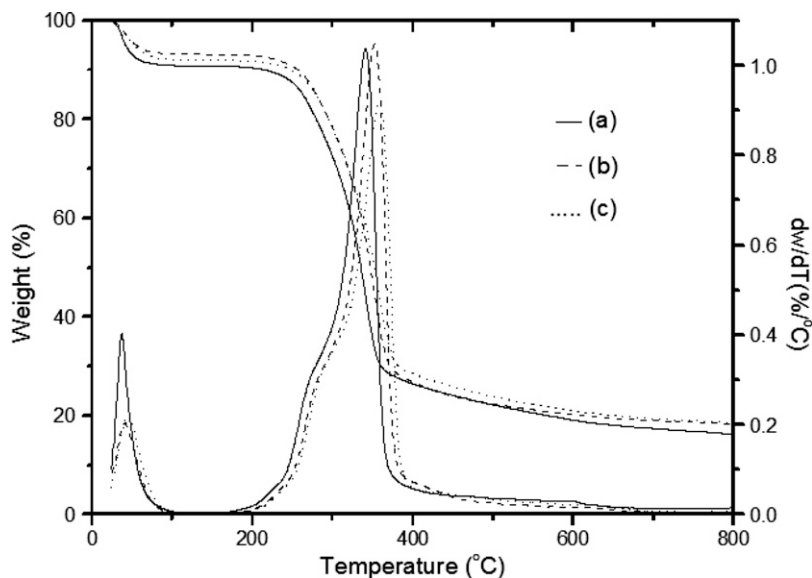
the wood composition varied depending on the species, and even within the same species, it can vary according to habitat, age, soil, and location in the tree (trunk, branches, and roots) (Pereira 1988). Proximate and ultimate analyses and the chemical composition values of the main components are summarized in Table 2.

With respect to the polysaccharide content, cellulose yielded the highest percentage, followed by xylans (which has been used as a representative of hemicellulosic materials) (Bilbao et al 1989). A smaller proportion correspond to arabinans (a pentose that occurs as one of the sugar units in some hemicelluloses), which con-

stitute 15-35% of the weight of most woods. They include several components such as xylose and mannose (which are the prevalent monomers for hardwoods and softwoods, respectively), glucose, galactose, arabinose, and other polysaccharides (Di Blasi and Lanzetta 1997). Similar values have been reported by other researchers for hardwoods (Carballo et al [2004] in *Corymbia citriodora* and Orea et al [2004] in *Eucalyptus saligna* and *Eucalyptus pellita*). The lignin content was also within the ranges reported by these researchers for hardwood (Table 3).

Thermogravimetric Analyses of *A. mangium* Sawdust

Figure 1 shows the mass loss and the derivative of mass loss curves obtained during the pyrolysis of *A. mangium* under inert atmosphere at a heating rate of 5-15°C min⁻¹. According to Fig 1, the pyrolysis curve of *A. mangium* follows the usual shape for lignocellulose materials (Orfão et al 1999). As for biomass, its thermal degradation can be divided into three stages: moisture drying, main devolatilization, and continuous slight devolatilization. Initial weight loss of the *A. mangium* sample was caused by a

Figure 1. Thermal analysis of *Acacia mangium* heated (a) 5°C min⁻¹, (b) 10°C min⁻¹, and (c) 15°C min⁻¹.

loss of moisture starting at about 30°C and continuing up to about 130°C. Similar results were obtained by Angelini et al (2009) and Jeguirim and Trouvé (2009). The *A. mangium* samples started to decompose and release volatiles at about 200°C. The thermogravimetric curves of the sample clearly showed changes in the slope of the curves. This behavior of dynamic decomposition for this type of material is principally attributed to the three main pseudo-components of the lignocellulosic material (hemicellulose, cellulose, and lignin) as explained in previous studies (Orfão et al 1999; Manyà et al 2002). Most decomposition occurred in the range of 300-400°C (approximately 80 wt %). The first peak was attributed to the decomposition of hemicellulose and the second to that of cellulose. Lignin is characterized by a wide range of decomposition throughout the process, because of the great complexity of the molecule in relation to the other constituents of the biomass (Meszaros et al 2004; Di Blasi 2008). Also, lignin decomposition started, spanned a wide temperature interval, and was overlapped by decomposition of the other components. But lignin decomposition is essentially a predominant step at higher temperatures at which it is characterized by its low devolatilization rates. This was explained in previous studies (Gronli et al 2002; Meszaros et al 2004).

The process of biomass decomposition occurs in three basic parts, which is in line with previous results for this kind of lignocellulosic material (Gronli et al 2002; Meszaros et al 2004; Jeguirim and Trouvé 2009) (Fig 1).

Several studies for modeling and determining kinetic parameters for various lignocellulosic materials were published in the last decade. They were generally based on a kinetic mechanism

of three parallel reactions associated with the three main pseudo-components, hemicellulose, cellulose, and lignin (Orfão et al 1999; Gronli et al 2002; Meszaros et al 2004; Di Blasi 2008; Mahanta et al 2009). The inclusion of various heating rates in the kinetic analysis of biomass devolatilization is important from both theoretical and practical points of view. The evaluation of multiple curves was proposed by Caballero and Conesa (2005) to avoid the compensation effect of the parameters.

The kinetic parameters are summarized in Table 4. Values of activation energy for the three fractions were less than the ranges reported in the literature: 80-116 kJ mol⁻¹ for hemicellulose, 180-286 kJ mol⁻¹ for cellulose, and 18-65 kJ mol⁻¹ for the lignin (Gronli et al 2002; Meszaros et al 2004; Mahanta et al 2009). The contribution factor of each component as well as the correlation factor are summarized in Table 5.

The values of the component contribution calculated as a percentage of the total mass fraction agree with the ranges reported in the literature: 20-30% for hemicelluloses and 28-38% to 10-15% for cellulose and lignin for this type of lignocellulosic material (Di Blasi 2008). The proportions of these constituents and the presence of inorganic ions can cause the characteristics of decomposition to vary from one species to another, even within the same species (Gronli et al 2002; Meszaros et al 2004; Várhegyi et al 2004). Another influencing factor is the heating rate at which the experiments were carried out (Branca et al 2005). Figure 2 shows the experimental and calculated curves of the biomass decomposition at 10°C min⁻¹. It also shows the evolution of the curves for each of the separate fractions by model and kinetic parameters estimated in the study. The simulated evolution for

Table 4. Pyrolysis kinetic parameters of *Acacia mangium*.

Fraction	k ₀ /min ⁻¹	E/kJ mol ⁻¹
Hemicellulose	4.70 × 10 ⁵	79.04
Cellulose	1.02 × 10 ¹⁵	181.59
Lignin	14.55	30.80

Table 5. Parameters estimated for the main components at the heating rates studied.

Heating rate/°C min ⁻¹	Contribution factor of each component			
	Hemicellulose	Cellulose	Lignin	Fit (%)
5	0.18	0.42	0.23	2.31
10	0.25	0.41	0.14	2.30
15	0.27	0.36	0.17	2.31

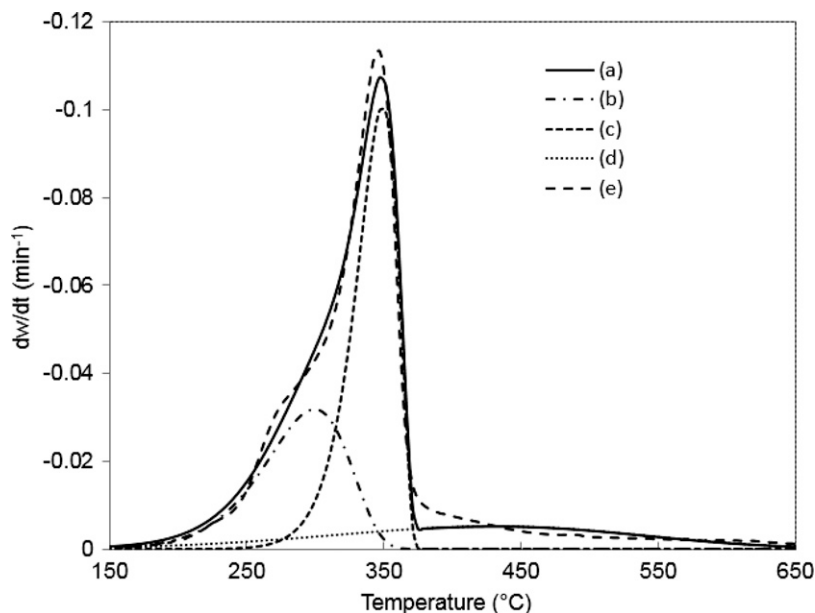


Figure 2. Experimental and calculated derivate thermogravimetry curves of *Acacia mangium* at $10^{\circ}\text{C min}^{-1}$ (a) calculated, (b) hemicellulose, (c) cellulose, (d) lignin, and (e) experimental.

the hemicellulose, cellulose, and lignin agreed with studies for these components separately (Varhegyi et al 1989; Antal and Varhegyi 1995). A good correlation between the experimental curves and those simulated by the model is shown in Fig 2.

CONCLUSIONS

The thermograms show that the pyrolysis process of *A. mangium* highlights three areas of decay, mainly associated with its three main components: hemicellulose, cellulose, and lignin.

The proposed model predicts the curves of biomass pyrolysis of *A. mangium* with a deviation of less than 3% between calculated and experimental curves at the heating rates studied. This confirms that the process of pyrolysis of lignocellulosic materials can be explained by a kinetic mechanism of three independent parallel reactions. The values of the calculated kinetic parameters, (activation energy, preexponential factor, and contribution factor for each of the pseudo-components (hemicellulose, cellulose, and lignin)) are consistent with those reported

by other researchers for this type of lignocellulose material.

ACKNOWLEDGMENTS

This research was supported by the Research Project of the Cuban and Brazilian Ministries of Higher Education CAPES/CGCI n° 002/2009. The authors are grateful to the Laboratório de Celulose e Papelda Universidade Federal de Viçosa.

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