Modelling the Thermal Decompositions of Wood and Leaves Under a Nitrogen Atmosphere

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The thermal decomposition of six different samples of wood and leaves in nitrogen has been studied by using dynamic thermogravimetry. In the experiments two main weight loss processes took place and the total weight loss at 500°C was over 95% in all six cases. By means of the Doyle method, the two processes were found to fit most closely the plot for the second-order equation in the form \( \frac{dx}{dt} = k(1-x)^2 \), and each of the weight loss processes was found to be controlled, respectively, by two dominant reactions as the temperature increases. It was inferred that competing reactions occur during the overall temperature interval for all the six samples. By comparing the activation energies using this model with those by the method of Moll et al., and by comparing the experimental and theoretical thermogravimetric curves, the ‘second-order’ model was tested to be able to predict the weight loss processes of the samples with very good accuracy. It can be concluded that the ‘second-order’ kinetic model acts much better than the conventionally adopted first-order model. © 1998 John Wiley & Sons, Ltd.

INTRODUCTION

The combustion of wood or leaves involves two separate and distinct processes: (i) the thermal decomposition of a solid phase into volatile products, both combustible and non-combustible, and combustible non-volatiles, i.e., charcoal, and (ii) gas-phase oxidation reactions of volatiles near and above the surface. The thermal decomposition of the solid phase has long been recognized as an important problem in fire research. Consider a piece of wood or leaf initially at ambient temperature. Suppose one now subjects the exposed surface to a prescribed heat flux. At first the solid heats up by pure transient conduction. Then, when the surface layer becomes sufficiently hot, it pyrolyzes (decomposes) into gaseous volatiles and residual char. Along with the volatiles becoming hotter and hotter, ignition will take place. Subsequently, the gas-phase combustion feeds heat to the solid which, in response, generates more fuel volatiles. The volatiles, in turn, sustain the combustion. In one sense, understanding the thermal decomposition process can be looked on as the key to model the subsequent ignition.

Early papers attempted to understand the decomposition of wood by the study of the thermal degradation of cellulose, which is the main component of any ordinary plant. Schwenker and Beck succeeded in isolating 37 products from the degradation of cellulose, and it is understandable that the detailed mechanism of the thermal degradation is not clear. For apparent cellulose pyrolysis kinetics, it is now well known that under conditions which minimize vapour–solid interactions and heat transfer intrusions, the TGA and DTG curves associated with the pyrolysis of a wide variety of pure cellulose samples can be well fitted by an irreversible, single-step, first-order rate equation. For the pyrolysis of whole biomass (such as wood and leaves), however, Antal and Varhegyi concluded that minute amounts of mineral matter naturally present in whole biomass samples strongly catalyze the decomposition of the cellulose component in unpredictable ways, and until now little was known concerning the whole biomass pyrolysis kinetics. Chen and Li pointed out that when isolated from wood or leaves, cellulose shows different characteristics of decomposition. Kung studied theoretically the pyrolysis of a wood slab, one side of which is heated and the other side insulated and impervious. The model provided a quantitative representation of many interacting physical processes during the decomposition. However, no kinetic model with good accuracy was implied in the paper, and a single first-order rate equation was assumed to obtain some specific solutions. Zeriouh and Belkbir studied the thermal decomposition of a Moroccan wood by means of dynamic and isothermal experiments, carried out under a nitrogen atmosphere. At low heating rates, weight loss measurements on pyrolysis samples of the wood indicated that decomposition occurs in discrete stages of hemicellulose evolution, cellulose decomposition and lignin degradation. Jiang investigated the thermal decomposition of some biomass fuels. The paper also indicated that the pyrolysis contains the stages of cellulose decomposition and lignin degradation, just as Zeriouh and Belkbir’s paper stated. However, a single first-order rate equation was still adopted to model the kinetics and its accuracy was not justified in the paper.

Many other kinetic studies of wood decomposition have been undertaken. However, it appears that all of them have assumed single first-order rate reaction. No previous work has been found in the literature dealing with the kinetics of the decomposition of leaves. By contrast, being aimed at the decompositions of both

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wood and leaves, the present study seeks for a more suitable kinetic explanation of the process in terms of the general interpretations of solid-state reactions by dynamic thermogravimetry. The model presented reflects competing reactions in the decomposition process and can be applied to predict the weight loss against the temperature with satisfactory accuracy.

EXPERIMENTAL

The six samples used in the investigation were respectively leaves of masson pine, leaves of tea tree, leaves of oil-tea tree, nanmu wood, paulownia wood, and willow wood collected from Qimen forest zone in Anhui province of China (Table 1). These samples were ground and the fraction passing 20 mesh was used for analysis. Thermal decomposition was observed in terms of the overall weight loss by using a WRT-3 thermobalance made by Shanghai Balance Instrument Plant. An atmosphere of dry nitrogen, oxygen-free, was passed into the furnace at a flow rate of 80 ml/min. This flow rate had previously been shown to be sufficient to prevent back-diffusion of air into the furnace. The sample was contained in a wide-mouth porcelain crucible lightly packed so as not to impede loss of volatile products. Dynamic thermogravimetry, which works much better than conventional isothermal studies, was adopted in this study. In the thermogravimetric experiments the furnace was lowered around the sample, the balance mechanism was released, and after allowing 20 min for the nitrogen to purge the system, the temperature was increased to 500 °C at a rate of 15 °C/min. The buoyancy effect was considered to be negligible, which was justified by some other researchers, e.g., Simons.

RESULTS AND DISCUSSION

Characteristics of the thermogravimetric curves

In nitrogen all six samples were found to give the same general shape for the plot of weight loss against temperature. The thermogravimetric (TG) and differential thermogravimetric (DTG) curves are shown in Figs 1 and 2. The first weight loss occurred in the temperature range 40–110 °C, constituting an approximately 10% weight loss for all the six samples. This loss of weight was attributed to the evaporation of sorbed water. Following the water evaporation, a second weight loss began slowly and accelerated rapidly in the temperature range 290–310 °C. A third weight loss followed the second one and reached an overall weight loss of more than 95%. The residue is charcoal. Compared with Zeriouh and

<table>
<thead>
<tr>
<th>Material name</th>
<th>Botanical name</th>
<th>Initial weight (mg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Masson pine</td>
<td>Pinus massoniana</td>
<td>9.20</td>
</tr>
<tr>
<td>Tea tree</td>
<td>Camellia sinensis</td>
<td>7.51</td>
</tr>
<tr>
<td>Oil-tea tree</td>
<td>Camellia oleifera</td>
<td>5.73</td>
</tr>
<tr>
<td>Nanmu</td>
<td>Phoebe nanmu</td>
<td>8.16</td>
</tr>
<tr>
<td>Paulownia</td>
<td>Paulownia tomentosa</td>
<td>8.98</td>
</tr>
<tr>
<td>Willow</td>
<td>Salix babylonica</td>
<td>6.19</td>
</tr>
</tbody>
</table>

Figure 1. Weight losses of the thermal decomposition of three leaf samples: (a) leaves of masson pine; (b) leaves of tea tree; (c) leaves of oil-tea tree.

Figure 2. Weight losses of the thermal decomposition of three wood samples: (a) nanmu wood; (b) paulownia wood; (c) willow wood.
Belkbir’s results, it can be inferred that the second weight loss mainly corresponds to the decomposition of cellulose, while the decomposition of lignin leads to the third weight loss process. It can be concluded that the decompositions of wood and leaves have qualitatively common behaviour.

Kinetic model of the weight loss process

The thermal decomposition of wood or leaves can be expressed by the stoichiometric equation

$$A \text{ (solid)} \rightarrow B \text{ (solid)} + C \text{ (gas)}$$

The rate expression for the disappearance of the original sample is

$$\frac{dz}{dt} = kf(z)$$

where $z$ is the fraction reacted in time $t$, and $k$ is the empirical rate constant, which depends on the absolute temperature $T$ according to the Arrhenius equation

$$k = A \exp(-E/RT)$$

where $A$, $E$, and $R$ are constants known as the pre-exponential factor (or frequency factor), the activation energy, and the gas constant, respectively. The specific form of $f(z)$ depends on the reaction mechanism. Many kinds of mechanisms have been found to control different kinetic weight loss processes, and the corresponding kinetic equations were frequently stated in the literature, e.g. References 11 and 12.

In the dynamic experiments the temperature of the system increases linearly, i.e. the rate of heating was kept constant. Combining Eqns (1) and (2) gives

$$\frac{dz}{f(z)} = \frac{A}{\beta} \exp(-E/RT)\,dT$$

where $\beta$ is the constant rate of heating.

A new function $g(z)$ is defined by

$$g(z) = \int_0^z \frac{dz}{f(z)}$$

and Doyle$^{13}$ showed that the following form can be obtained:

$$\ln\left[\frac{g(z)}{T^2}\right] = \ln\frac{AR}{\beta E} - \frac{E}{RT}$$

For a reaction which satisfies that $f(z) = (1 - z)^n$, where $n$ is the order of reaction, Coats and Redfern$^{14}$ derived the following dynamic equation:

$$\ln\left[\frac{g(z)}{T^2}\right] = \ln\frac{AR}{\beta E} \left[1 - \frac{2RT}{E}\right] - \frac{E}{RT}$$

where

$$g(z) = \begin{cases} -\ln(1-z), & n = 1 \\ \frac{1 - (1-z)^{1-n}}{1-n}, & n \neq 1 \end{cases}$$

A plot of $\ln[g(z)/T^2]$ against $1/T$ should result in a straight line of slope $-E/R$ for the correct reaction mechanism, and constitutes the foundation of the method to find the suitable model of wood or leaves pyrolysis. As soon as the form of $g(z)$ is obtained, the apparent activation energy $E$ and the frequency factor $A$ can be calculated from the straight line in the light of Eqns (5) or (6).

The thermal decomposition of wood or leaves is in essence an extremely complex process. No rigorous horizontal portion or plateau exists on the obtained thermogravimetric curves, which indicated that overlapping reactions occur over the total temperature interval. For this reason, it is difficult to locate on the TG curve an unambiguous point where one reaction ends and the other starts. In other words, it is not easy to estimate the fraction reacted $z$, which can be expressed as

$$z = \frac{w_i - w_f}{w_i - w_f}$$

where $w_i$ is the initial sample weight percentage, $w_f$ the final weight percentage of the char, and $w_f$ the weight percentage at absolute temperature $T$. In our research the temperature corresponding to the minimum in the DTG curve was regarded as the point of inflection between two weight loss processes. Wendlandt$^{15}$ interpreted this viewpoint in detail.

Because the final weight for each pyrolysis process was approached asymptotically, it was impracticable to run each experiment to its endpoint. The value of $w_f$ for the third weight loss process of each run (which was controlled by the decomposition of lignin) was determined by constructing a graph of weight against reciprocal absolute temperature $T$. As $T$ became large, i.e. $1/T$ became small, the graph became linear; and it was possible to extrapolate to $1/T = 0$ in order to estimate $w_f$. The validity of this procedure was justified by Dollimore and Holt in their study of thermal degradation of cellulose.$^{16}$

Using the experimental thermogravimetric curves, many specific kinetic equations corresponding to different forms of $g(z)$ were substituted into Eqn (5), respectively, to see if any one of them gave good linearity for Eqn (5). In the calculations, values of $z$ were calculated at convenient intervals of temperature and for every attempted form of $g(z)$ the corresponding values of $\ln[g(z)/T^2]$ were calculated. The plots are significant (Table 2). The broken line indicated that for every weight loss process, a broken line rather than a completely straight one was obtained. The linear regressions have acceptable correlation coefficients $r^2$, confirming that the plots are significant (Table 2). The broken line indicated that for every weight loss process (either the decomposition of cellulose or that of lignin), the apparent activation energy, or in other words, the apparent primary reaction controlling the process undergoes a sudden change in a very small range of temperature. This can result if there are two competing reactions.
with different activation energies. However, it can be inferred that the two controlling reactions have little possibility to constitute purely parallel reactions in the form such as

\[ A \text{ (solid)} \rightarrow B \text{ (solid)} + C \text{ (gas)} \]
\[ A \text{ (solid)} \rightarrow D \text{ (solid)} + E \text{ (gas)} \]

because in such parallel reactions, a broken line can be obtained only if the rate constant of the rate-determining reaction keeps much bigger than that of the other reaction. However, using Eqn (2), the rate constants of the two reactions can be calculated, respectively, at any point of temperature, and they are found not to differ too much.

A consecutive reaction can be inferred to be the possible mechanism which results in the broken line. The first reaction step produces intermediates, which follows to begin the second one. With the first step going on, the quantity of the original sample decreases and that of the intermediates increases. Gradually, the rate-determining step changes from the first one to the second one.

It should be pointed out, however, that the above clarified rate-determining reactions are accompanied by many other less important reactions (in the sense that they contribute little to the loss of weight) at all times. In essence, the thermal decomposition of wood or leaves is of extreme complexity. It is understandable that the detailed mechanisms of the reactions involved in the process are not clear. The present study, however, implies that some primary reactions play a leading role in the overall weight loss processes apparently.

The apparent activation energies and frequency factors were obtained from the straight lines in Figs 3 and 4. The results are listed in Table 2.

**Figure 3.** Plots of ln \([a/(1-a) T^2]\) against the reciprocal of \(T\) for the three leaf samples: (a) leaves of masson pine; (b) leaves of tea tree; (c) leaves of oil-tea tree.

**Figure 4.** Plots of ln \([a/(1-a) T^2]\) against the reciprocal of \(T\) for the three wood samples: (a) nanmu wood; (b) paulownia wood; (c) willow wood.

**Verification of the ‘Second-order’ kinetic model**

Comparison of the activation energies obtained by the ‘second order’ model with the values by the method of Moll et al. For the general reaction which satisfies that \(f(a) = (1 - a)^n\), Moll et al.\(^{17}\) derived a method to estimate the activation energy without knowing the reaction order, which is the main advantage over the method of Doyle, because for the latter kinetic parameters can be obtained only if the order \(n\) is known. In view of this difference between the two independent methods, the obtained activation energies by the method of Moll et al. can be compared with the corresponding values in Table 2, and their coincidence can test and verify the correctness of the ‘second order’ kinetic model from one side.

Combining Eqns (1) and (2), and using the relation \(f(a) = (1 - a)^n\), the reaction rate can be expressed as follows:

\[
d\alpha/dT = A \beta e^{-E/RT}(1 - a)^n \tag{9}\]

Moll et al. assumed that two points of temperature \(T_1\) and \(T_2\) can be selected on the TG curve such that the corresponding values of \((1 - x_1)^n\) and \((1 - x_2)^n\) have difference little enough to be negligible, where the activation energy \(E\) can then be estimated by the following expression:

\[
E = R \frac{\ln[(dx_2/dT)/(dx_1/dT)]}{1/T_1 - 1/T_2} \tag{10}\]

where the values of \([dx_1/dT]/(dx_2/dT)\) can be obtained directly from the DTG curve.
Table 2. Kinetic parameters for thermal decomposition of six samples by the ‘second-order’ model

<table>
<thead>
<tr>
<th>Material</th>
<th>Weight loss process and rate-determining reaction</th>
<th>Temperature range (°C)</th>
<th>Activation energy (kJ/mol)</th>
<th>Frequency factor A (min⁻¹)</th>
<th>ρ²</th>
</tr>
</thead>
<tbody>
<tr>
<td>LMP</td>
<td>DC(a)</td>
<td>150-284</td>
<td>78.82</td>
<td>8.089 × 10⁶</td>
<td>0.9955</td>
</tr>
<tr>
<td></td>
<td>DC(b)</td>
<td>284-354</td>
<td>160.63</td>
<td>7.357 × 10¹⁴</td>
<td>0.9926</td>
</tr>
<tr>
<td></td>
<td>DL(a)</td>
<td>354-419</td>
<td>226.63</td>
<td>2.685 × 10¹⁷</td>
<td>0.9884</td>
</tr>
<tr>
<td></td>
<td>DL(b)</td>
<td>419-500</td>
<td>169.20</td>
<td>1.073 × 10¹³</td>
<td>0.9913</td>
</tr>
<tr>
<td>LTT</td>
<td>DC(a)</td>
<td>149-302</td>
<td>65.78</td>
<td>4.768 × 10⁶</td>
<td>0.9938</td>
</tr>
<tr>
<td></td>
<td>DC(b)</td>
<td>302-372</td>
<td>117.92</td>
<td>5.256 × 10¹⁰</td>
<td>0.9968</td>
</tr>
<tr>
<td></td>
<td>DL(a)</td>
<td>372-466</td>
<td>110.45</td>
<td>2.313 × 10⁷</td>
<td>0.9872</td>
</tr>
<tr>
<td></td>
<td>DL(b)</td>
<td>466-527</td>
<td>286.18</td>
<td>1.630 × 10²⁰</td>
<td>0.9971</td>
</tr>
<tr>
<td>LOT</td>
<td>DC(a)</td>
<td>154-275</td>
<td>63.68</td>
<td>1.953 × 10⁶</td>
<td>0.9914</td>
</tr>
<tr>
<td></td>
<td>DC(b)</td>
<td>275-378</td>
<td>111.54</td>
<td>1.178 × 10¹⁰</td>
<td>0.9951</td>
</tr>
<tr>
<td></td>
<td>DL(a)</td>
<td>378-435</td>
<td>188.70</td>
<td>4.472 × 10¹³</td>
<td>0.9962</td>
</tr>
<tr>
<td></td>
<td>DL(b)</td>
<td>435-490</td>
<td>406.80</td>
<td>1.269 × 10³⁰</td>
<td>0.9964</td>
</tr>
<tr>
<td>NW</td>
<td>DC(a)</td>
<td>162-295</td>
<td>116.57</td>
<td>3.320 × 10¹⁰</td>
<td>0.9945</td>
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<tr>
<td></td>
<td>DC(b)</td>
<td>295-350</td>
<td>290.53</td>
<td>1.191 × 10²⁶</td>
<td>0.9880</td>
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<tr>
<td></td>
<td>DL(a)</td>
<td>350-455</td>
<td>109.37</td>
<td>3.543 × 10⁷</td>
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<td></td>
<td>DL(b)</td>
<td>455-514</td>
<td>320.37</td>
<td>1.382 × 10²³</td>
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<tr>
<td>PW</td>
<td>DC(a)</td>
<td>182-298</td>
<td>149.00</td>
<td>4.584 × 10¹³</td>
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<tr>
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<td>DC(b)</td>
<td>298-358</td>
<td>215.21</td>
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<td>DL(a)</td>
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<td>287.32</td>
<td>2.337 × 10²²</td>
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<tr>
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<td>DL(b)</td>
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<td>645.17</td>
<td>4.451 × 10²⁰</td>
<td>0.9462</td>
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<td>WW</td>
<td>DC(a)</td>
<td>173-322</td>
<td>118.73</td>
<td>1.522 × 10¹⁰</td>
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</tr>
<tr>
<td></td>
<td>DC(b)</td>
<td>322-385</td>
<td>296.93</td>
<td>1.515 × 10²⁶</td>
<td>0.9888</td>
</tr>
<tr>
<td></td>
<td>DL(a)</td>
<td>385-426</td>
<td>226.56</td>
<td>4.877 × 10¹⁶</td>
<td>0.9818</td>
</tr>
<tr>
<td></td>
<td>DL(b)</td>
<td>426-495</td>
<td>711.36</td>
<td>2.643 × 10³³</td>
<td>0.9984</td>
</tr>
</tbody>
</table>

¹LMP: the leaves of masson pine; LTT: the leaves of tea tree; LOT: the leaves of oil-tea tree; NW: nanmu wood; PW: paulownia wood; WW: willow wood.
²DC represents the weight loss process controlled by the decomposition of cellulose and DL the weight loss process controlled by the decomposition of lignin. The small letters in parenthesis represent the apparent rate-determining reactions involved in the corresponding weight loss processes.

Table 3. Comparison of the activation energies by the ‘second-order’ model and the corresponding values by the method of Moll et al.

<table>
<thead>
<tr>
<th>Material</th>
<th>T₁(°C)</th>
<th>T₂(°C)</th>
<th>(dα₁/dT)(dα₂/dT)</th>
<th>Activation energy by the ‘second order’ model (kJ/mol)</th>
<th>Activation energy by the method of Moll et al. (kJ/mol)</th>
</tr>
</thead>
<tbody>
<tr>
<td>LMP</td>
<td>222.3</td>
<td>227.5</td>
<td>1.20</td>
<td>78.82</td>
<td>71.61</td>
</tr>
<tr>
<td>LTT</td>
<td>218.5</td>
<td>224.5</td>
<td>1.36</td>
<td>117.92</td>
<td>111.98</td>
</tr>
<tr>
<td>LOT</td>
<td>187.75</td>
<td>193.5</td>
<td>1.23</td>
<td>63.68</td>
<td>64.28</td>
</tr>
<tr>
<td>NW</td>
<td>241.0</td>
<td>246.4</td>
<td>1.31</td>
<td>116.57</td>
<td>112.67</td>
</tr>
<tr>
<td>PW</td>
<td>240.0</td>
<td>243.9</td>
<td>1.33</td>
<td>149.00</td>
<td>158.27</td>
</tr>
<tr>
<td>WW</td>
<td>243.8</td>
<td>249.6</td>
<td>1.31</td>
<td>118.73</td>
<td>102.32</td>
</tr>
</tbody>
</table>

¹LMP: the leaves of masson pine; LTT: the leaves of tea tree; LOT: the leaves of oil-tea tree; NW: nanmu wood; PW: paulownia wood; WW: willow wood.

Table 3 shows the comparison of the activation energies obtained by the two independent methods, which gives good quantitative agreement. The points of temperature were carefully selected to satisfy the conditions of the method of Moll et al. as much as possible. However, in fact, the condition that the values of (1−α₁)ⁿ and (1−α₂)ⁿ differ little can never be satisfied very well, which might be the reason for the discrepancies.

Comparison of the experimental and theoretical TG curves

By comparing the theoretical and experimental TG curves, the ‘second order’ kinetic model can be further tested and verified. Every theoretical TG curve is made up of four curve segments which correspond to the primary reactions controlling the weight loss processes in four ranges of temperature respectively. The equation of the theoretical curve can be derived as follows: For \( f(x) = (1 - x)^2 \), Eqn (4) gives

\[ g(x) = (1 - x)^{-1} - 1 \]  \( (11) \)

Rearrangement of Eqn (5) yields

\[ g(x) = \frac{AR^2}{\beta E} \exp(-E/RT) \]  \( (12) \)
Combining Eqn (11), (12) and (7), the sample weight percentage at temperature \( T \) can be expressed as follows:

\[
w_T = w_i - \frac{w_i - w_f}{1 + \frac{E}{RT}}
\]

The theoretical TG curve can then be obtained by substituting the parameters \( E \) and \( A \) into the above expression and linking up the four curve segments of \( w_T \) against \( T \) in four separate ranges of temperatures.

Figure 5 presents the comparison of the experimental and theoretical TG curves for the leaf sample of masson pine. For clarity, the experimental and theoretical curves and their superimposed plot are presented respectively.

The excellent agreement of the theoretical curves with the experimental ones in Fig. 5 provided an excellent evidence of the accuracy of the ‘second-order’ kinetic model. Similar results were found for the other five samples.

It should be pointed out that the comparison above does not just reverse the dynamic analysis. The dynamic analysis process only uses a few points in the TG curves. That is to say, from only a few number of points we get the dynamic equations of the decomposition, the dynamic parameters such as the activation energies and pre-exponential factors being obtained. After that, we use the dynamic equations, i.e. the information, from only a few number of points in TG curves to plot the total process of the decomposition. Therefore, the fit of the experimental and theoretical TG curves can justify the correctness of the second-order model.

CONCLUSION

The simple second-order kinetic equation has been found to be able to interpret the results of the dynamic thermogravimetry of six wood and leaf samples within excellent accuracy. Dominant, competing, Arrhenius type reactions were found to exist in every weight loss process. The activation energies by the method of Moll et al. are compared with the corresponding values by the ‘second order’ kinetic model. Also the theoretical TG curves by the ‘second-order’ model are compared with the experimental TG curves. These comparisons all result in good agreements, which fully justifies the ‘second-order’ kinetic model. It can be inferred from this study that for the extremely complex process of the thermal decomposition of wood or leaves, the weight loss can be apparently regarded as being controlled by several primary reactions in the overall range of temperature. Wood and leaves have qualitatively the same characteristics of thermal decomposition.

The chemical kinetics of pyrolysis is of great importance in the ignition process. For dense fuels that pyrolyze the value of the ignition temperature is controlled by the chemical kinetics of pyrolysis. Until now many studies have been made on the ignition and burning process of wood or leaves, e.g. Kung. How to integrate the present study with the numerous ignition models which involve many interacting physical processes during the ignition is the aim of future work.

Acknowledgements

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REFERENCES