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Experimental and numerical analysis of wood thermodegradation

Mass loss kinetics

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Abstract Torrefaction is a thermal treatment step in a temperature range of 210–240 °C, which aims to improve the dimensional stability and durability of wood. The mass loss kinetics for torrefaction of wood samples was studied using equipment specially conceived to measure mass losses during thermal treatment. Laboratory experiments were performed under nitrogen for heating rates of 0.1, 0.25, 1, and 2 °C min^{-1}. A mathematical model for the kinetics of the thermodegradation process was used and validated. Measurements of temperature distribution and anhydrous mass loss were performed on dry sample of poplar wood during torrefaction in an inert atmosphere for different temperatures. The mathematical formulation describing the simultaneous heat and mass transfers requires coupled nonlinear partial differential equations. These unsteady-state mathematical model equations were solved numerically by the commercial package FEMLAB for the temperature under different treatment conditions. A detailed discussion of the computational model and the solution algorithm is given below. Once the validity of different assumptions of the model had been analyzed, the experimental results were compared with those calculated by the model. Acceptable agreement was achieved.

Keywords Heat treatment · Modeling · Reaction kinetics · Thermodegradation · Wood

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Introduction

Nowadays, the use of wood as building material is promoted. The heat treatment of the wood by mild pyrolysis is used to improve some of the characteristics of the final wood product, such as, its durability and dimensional stability. This process substitutes the use of external chemical products, which enables the treated wood to remain as an environmentally friendly product. Its an effective method to improve biological durability of wood [1–3]. The heat treatment of the wood induces a chemical modification of the main wood constituents. The lignin polymer structure is modified [4–8], the ratio between the amorphous and the crystalline cellulose is also changed [9–11], hemicelluloses are strongly decomposed [10, 12, 13], and some precursor products of the charcoal appear [14]. These chemical modifications confer with the new wood properties, such as, the improved dimensional stability [15–17], the improved fungal resistance [18–20], some color changes [21–25], hydrophobic increase [19, 22, 25–29], and improved mechanical properties [30–32].

The concept of thermal treatment to stabilize the wood structure has lead to the development of several treatment processes in some European countries [1]. In the 70s, the research developed by the Ecole des Mines de Saint-Étienne (France) has worked out for the first time, an industrial process named «Retified wood®». More recently, in the 90s, the forest industry in Finland has developed a new process; Thermowood®. Some others technologies have been developed, such as, the Netherlands process «PLATO®» and the Menz Holz's process OHT (Oil Heat Treatment) in Germany. The wood torrefaction is a heat treatment of ligno-cellulosic material carried out at temperatures up to 300 °C. Torrefaction can be carried out under different gaseous atmospheres. Gaseous combustion products released during the thermal treatment are employed most of the time. Chemical reactions involved during torrefaction, as well as final properties of the material depend strongly on the treatment temperature and the duration of the process. During torrefaction, slow pyrolysis prevails: wood is thermally decomposed at a slow rate [33]. The anhydrous mass loss (AML) resulting from the wood decomposition is representative of the physical–chemical transformations of wood. The AML matches to the advancement of the torrefaction process.

According to the treatment intensity, the conferred properties of the wood vary strongly. For high values of the treatment temperature and time, the fungal resistance becomes total, the coloring is darkened, and the mechanical resistance is strongly distorted. Recent studies have shown the direct relation between the anhydrous mass loss (AML) due to thermodegradation and the conferred wood properties [20–34]. Hence, controlling the heat treatment process means to control precisely the AML of the material. It requires a coupled model, taking into account, heat transfer and mass transfer due to thermodegradation reactions inside wood sample.

The mechanism of wood thermodegradation shows the presence of several phases when the temperature increases. A phase of elimination of some volatile compounds takes place at temperatures lower than 190 °C. For temperature range of 190–280 °C, hemicelluloses are converted essentially into gases and acetic acid. This step corresponds to wood roasting.

At the temperature range about 250–300 °C, the lignin and cellulose decompose to give three products: gas (vaporization of volatile extractives and of bound water absorbed on the wood fibers, formation of volatile by-products like furfural or hydroxymethylfurfural, the depolymerization of lignin network through cleavage linkage or dehydration reactions lead to the formation of numerous low molecular weight phenolic compounds), tar, and char. This decomposition leads to an anhydrous mass loss, representative for wood chemical transformation. Properties of heat-treated wood depend on the mass losses [35]. Hence, controlling the quality of the heat-treated wood means to control precisely the mass loss during the treatment. Therefore, the knowledge of the kinetic schemes of the wood thermodegradation is required.

The aim of this study is to develop a tool allowing the prediction of the temperature profiles in the wood and the associated AML during the treatment. A two-stage semi-global kinetic model is used here to describe the wood thermodegradation. Numerous experiences of heat treatment at 240 °C under nitrogen were realized with poplar wood. The curves of the instantaneous mass loss and temperature were recorded. The experimental data were used to determine the model parameters. Finally, a comparison between the experimental data and the numerical results was realized.

Materials and methods

Wood sample

Heat treatment is carried out on wood panels of large sections, dried in the steam room at 105 °C, until their mass stabilizes around 269–280 g. The panel's dimensions are 25 × 11 × 2.5 cm^3, respectively, in the longitudinal, tangent, and radial directions. The wood specie used in the present study is the Poplar (*Popul*us *nigra*).

Heat treatment by conduction and measure of the evolution of the dynamic sample mass

Heat treatment has been carried out under nitrogen by conduction between two metallic heating plates placed on a precision balance (Fig. 1). The instantaneous mass and temperatures at the surface and in the middle of the sample are recorded. The heat treatment phase consists of three successive thermal stabilization areas (Fig. 2). The first one is kept at 105 °C to verify the mass stability (anhydrous mass). The second one (165 °C) is useful for the thermal homogenization of the panel. The last one corresponds to

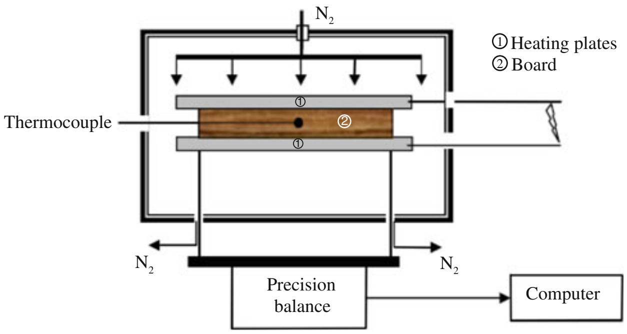

Fig. 1 Schematic equipment used for heat treatment.

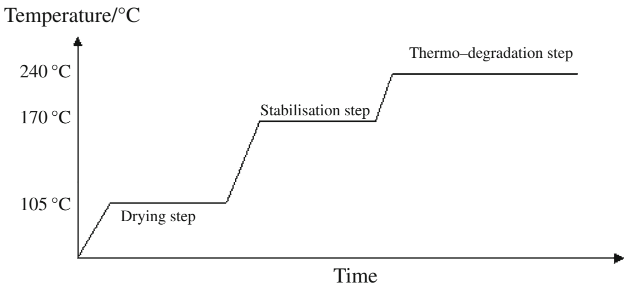

Fig. 2 Typical heat treatment schedule.

the thermodegradation operating temperature. Each test has been repeated three times to check the reproducibility.

Mathematical formulation

Modeling of the heat and mass transfers

Wood is a complex material whose thermodegradation involves a series of chemical reactions with coupled heat and mass transfer. This section is devoted to the mathematical formulation of the mentioned transfers in the wood porous media introducion, as well as the heat effects due to the reactions of polymer decomposition, as the formation of volatiles and non-degradable solid products. The following simple assumptions are used for the mathematical formulation:

- the wood sample has a rectangular form characterized by its length L, width l and thickness e, respectively in the longitudinal, tangent, and radial directions. The sample thickness is very weak compared with other dimension and the modeling can be represented in 1D.
- the convection transfer occurring because of the formation of volatiles inside the wood material during the heat treatment is neglected.
- The dimensions of the sample are considered invariable; the formation of fissures and cracks is neglected.
- The wood physical and thermal properties, respectively, the specific heat Cp, and the wood thermal conductivity λ are considered as a linear function of the temperature [36].

$$Cp = 1112 + 4.85\,(T - 273) \tag{1}$$

$$\lambda = 0.13 + 0.0003\,(T - 273) \tag{2}$$

The macroscopic conservation equation governing the heat transfer phenomenon is given by:

$$\rho\, Cp\, \frac{\partial T}{\partial t} = \frac{\partial}{\partial x}\left(\lambda \frac{\partial T}{\partial x}\right) + H_{\mathrm{p}} \frac{\partial \rho}{\partial t} \tag{3}$$

where $\rho = m(t)/V$ is the averaged anhydrous wood density, T is the local temperature, and H_{p} is reaction enthalpy. The instantaneous sample mass $m(t)$ is detailed in Eq. 11, the sample volume V is considered invariable.

The process of the wood heat treatment involves several reactions of polymer degradation and synthesis giving numerous reaction products [35, 37]. Consequently, mathematical modeling describing the wood thermodegradation is a simplified approximation of the real phenomena. However, the knowledge of kinetic schemes describing the thermodegradation process is required for the elaboration of predicting tools that allow the control of wood heat treatment.

Numerous mechanisms have been published to provide a rational explanation of the wood thermodegradation and the associated heat effects. Three main approaches could be distinguished: the first one considers the wood as a single homogeneous element [38–41], the second one takes into account the decomposition of the essential wood compounds: cellulose, hemicelluloses, and lignin [42], and the third one considers the existence of three fractions (pseudo-compounds) whose decomposition obeys different laws [43]. It is usually assumed that, the reaction kinetics follows an Arrhenius law with two parameters: activation energy and kinetic constant.

The mechanisms adopted in this study, is based on a two-stage, semi-global, multi-reaction kinetic model of wood mild pyrolysis, where the wood is subdivided into three pseudo-components A_1, A_2, *and* A_3, as it was previously proposed [43]. Each pseudo-component is characterized by a specific kinetic law of decomposition and its mass fraction, respectively α_1, α_2, and α_3, such as, $\alpha_3 = 1 - \alpha_1 + \alpha_2$. At a temperature level lower than 200 °C, the elimination of some volatile wood compounds occur. At the temperature range of 165–240 °C, the degradation of A_1 prevails, giving a gaseous product G_1. The thermodegradation of A_2 leads to the production of a solid fraction C_2 and a gas product G_2. The proposed kinetic scheme is given by the Eqs. 4–5.

$$A_1 \longrightarrow k_1 G_1 \tag{4}$$

$$A_2 \longrightarrow k_2 \gamma_2\, C_2 + (1 - \gamma_2) G_2 \tag{5}$$

The mass fractions of the non-degradable solid γ_2 depends on the temperature.

The assumption that, the kinetics of all the involved reactions is described by the first order laws, and the mass balance equations can be written, respectively as follows:

$$\frac{\mathrm{d}m_{A_1}}{\mathrm{d}t} = -k_1 m_{A_1} \tag{6}$$

$$\frac{\mathrm{d}m_{A_2}}{\mathrm{d}t} = -k_2 m_{A_2} \tag{7}$$

$$\frac{\mathrm{d}m_{C_2}}{\mathrm{d}t} = k_2\, \gamma_2\, m_{A_2} \tag{8}$$

where m_{A_1}, m_{A_2} and m_{C_2} are respectively the mass of the constituents A_1, A_2, and C_2; k_1 and k_2 are the reaction rate constants obeying the Arrhenius law such as:

Table 1 The kinetic parameters used in the numerical simulation for the poplar.

Species	α_1	α_2	Ea_1/kJ mol^{-1}	Ea_2/kJ mol^{-1}	k_{01}/s^{-1}	k_{01}/s^{-1}	γ	H_{p}/kJ mol^{-1}
Poplar	0.08	0.12	118.5	119	4.10^{7}	6.10^{8}	0.07	123

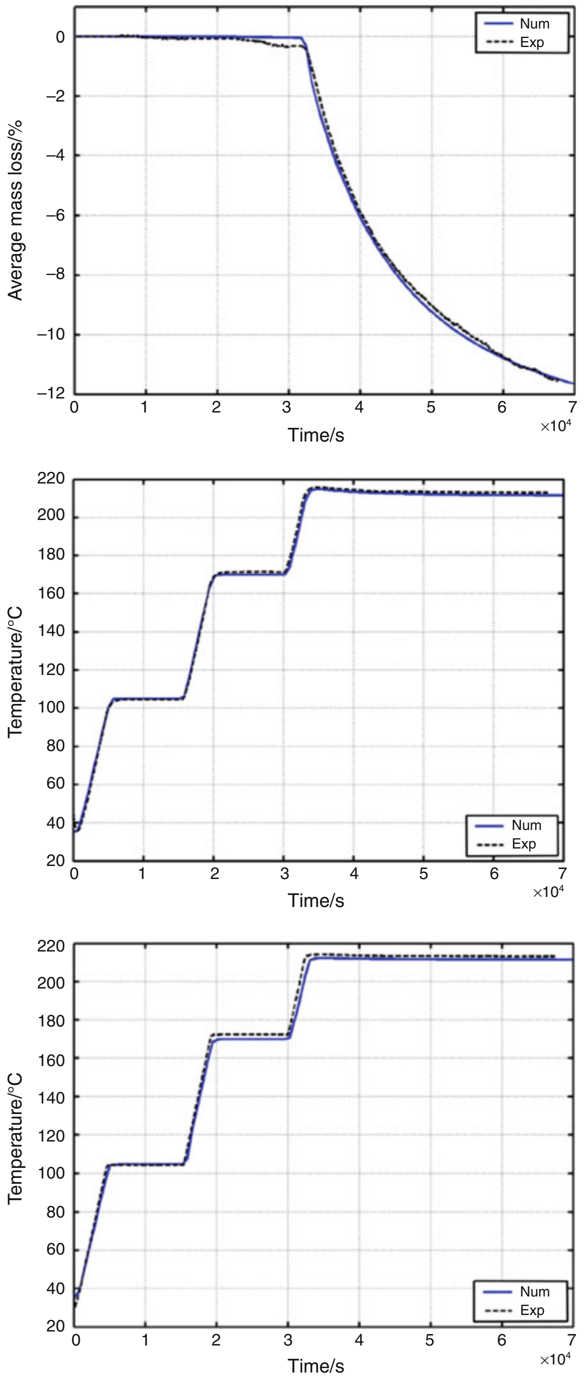

Fig. 3 Time evolution of average weight loss, surface temperature, and center temperature: Poplar (1 °C min^{-1}, $T_{\max} = 210$ °C).

$$k_1 = k_{01}\ \exp\left(-\frac{Ea_1}{RT}\right) \tag{9}$$

$$k_2 = k_{02}\ \exp\left(-\frac{Ea_2}{RT}\right) \tag{10}$$

where, k_{01} and k_{02} are the pre-exponential factors; Ea_1 and Ea_2 are the activation energies. The instantaneous total mass of the sample is equal to:

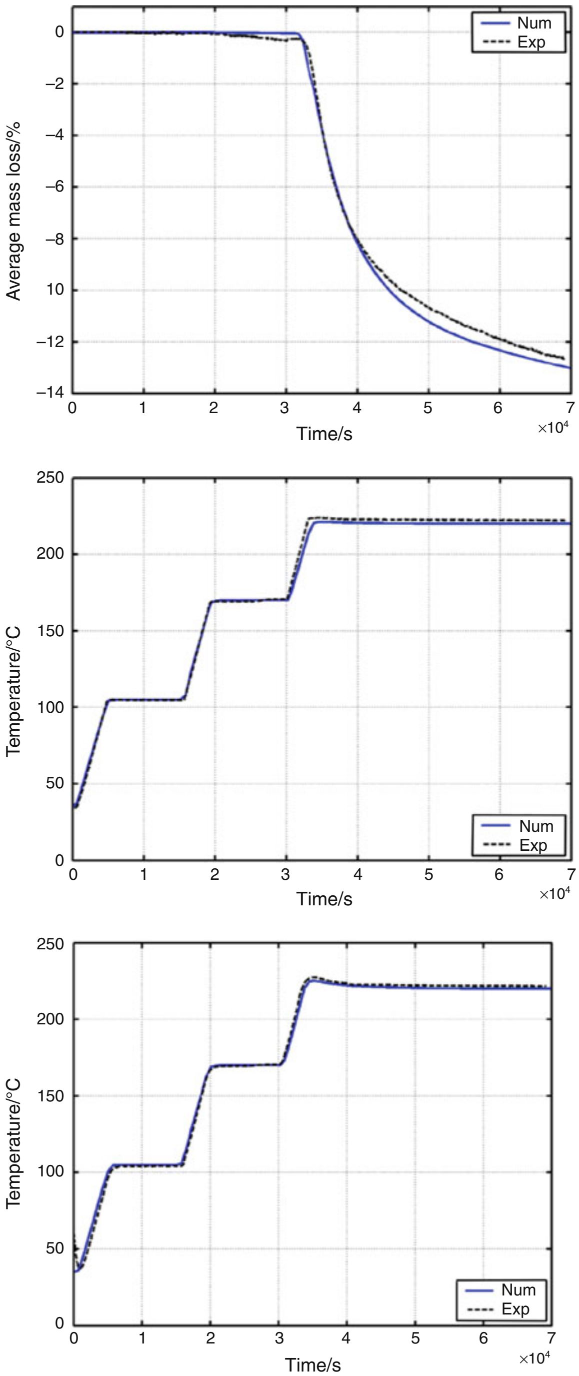

Fig. 4 Time evolution of average weight loss, surface temperature, and center temperature: Poplar (1 °C min^{-1}, $T_{\max} = 220$ °C).

$$m(t) = \mathrm{m}_{A_1}(t) + m_{A_2}(t) + m_{A_3}(t) + m_{C_2}(t) \tag{11}$$

Determination of the model’s parameters

The resolution of the Eqs. 3–10 requires the determination of 8 parameters: the pre-exponential factors k_{01}, k_{02}, and activation energies Ea_1, Ea_2, the mass fraction fractions α_1,

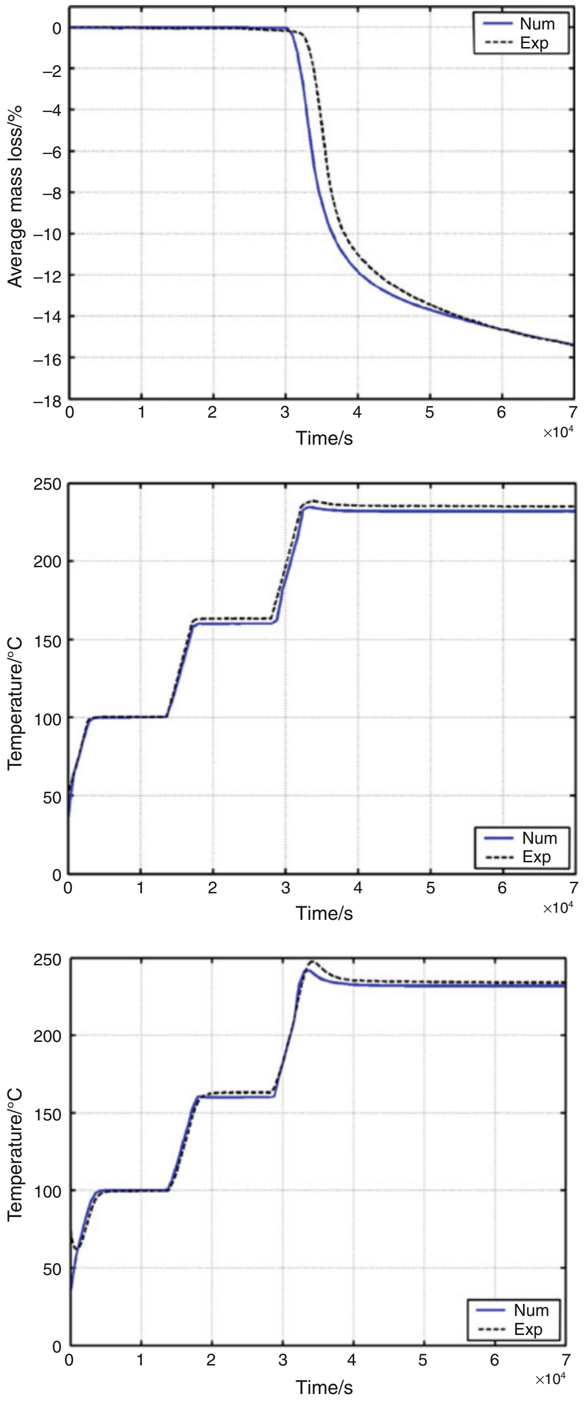

Fig. 5 Time evolution of average weight loss, surface temperature, and center temperature: Poplar (1 °C min^{-1}, $T_{\max} = 230$ °C).

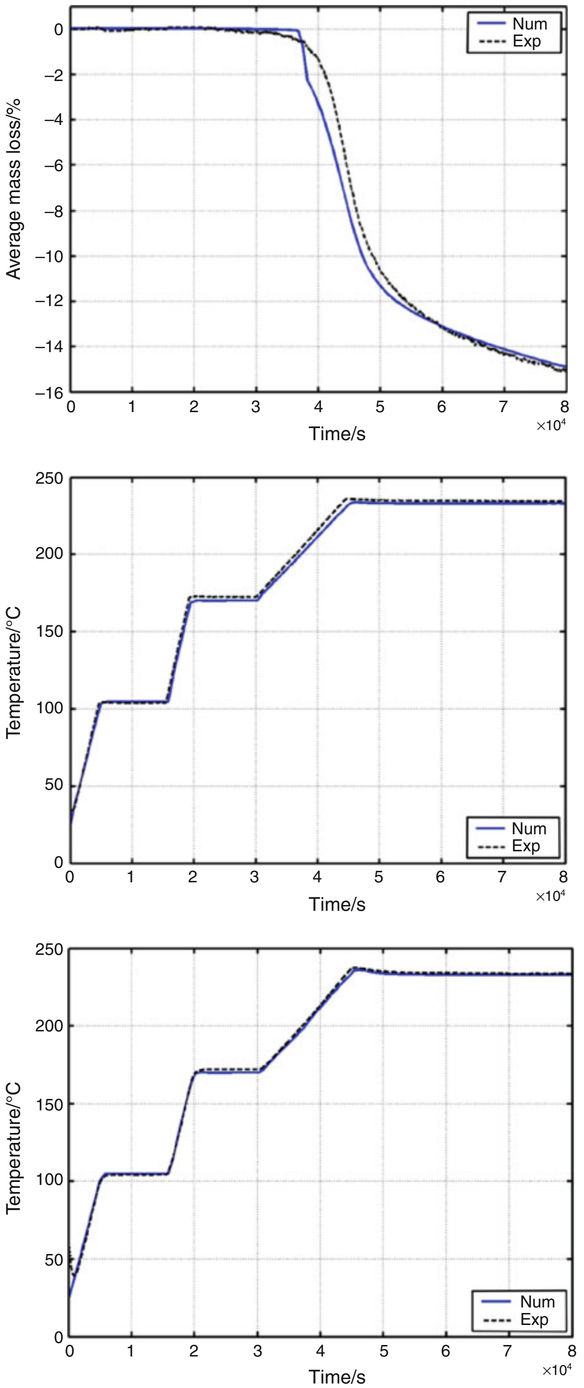

Fig. 6 Time evolution of average weight loss, surface temperature, and center temperature: Poplar (0.25 °C min^{-1}, $T_{\max} = 230$ °C).

α_2, and γ_2; the reaction enthalpy *Hp*. These parameters have been obtained by minimizing an error function between the experimental data and simulation results. The number of parameters to determine is large and the optimization procedure becomes difficult. Consequently, it is necessary to find a good method to obtain an efficient optimization. Because of the complexity of the model, the dependence between the parameters, and the existence of several local optima, the deterministic methods (conjugate gradient, Lagrange etc.) could not be used. A method based on a random selection of numbers and exploring the search space more efficiently is required. As example of such kind of methods, it can be cited to the Monte Carlo algorithm or genetic revolution [44–46], the hybrid algorithm, the simulated annealing algorithm, etc. The genetic revolution has been adopted in this study. The error function to minimize over both the thermal and mass measurements is given in the Eq. 12.

$$\mathrm{error}_{\mathrm{global}} = \frac{1}{n_1}\sum_{i=1}^{n_1}\frac{\left|T_{\exp}(i) - T_{\mathrm{calculated}}(i)\right|}{\left(T_{\exp}(i) + T_{\mathrm{calculated}}(i)\right)} + \frac{1}{n_2}\sum_{j=1}^{n_2}\frac{\left|AML_{\exp}(j) - AML_{\mathrm{calculated}}(j)\right|}{\left(AML_{\exp}(j) + AML_{\mathrm{calculated}}(j)\right)} \quad (12)$$

where n_1 is the number of data points of the temperature, $T_{\exp}(i)$ is the experimental temperature, $T_{\mathrm{calculated}}(i)$ is the calculated temperature on the i-th grid point, n_2 is the number of data points of the mass loss $\mathrm{AML}_{\exp}(j)$ and $\mathrm{AML}_{\mathrm{calculated}}(j)$ are respectively the experimental and the calculated mass loss due to the thermodegradation on the j-th grid point. The anhydrous mass lost is defined as follows:

$$\mathrm{AWL} = (m_0 - m(t))/m_0 \quad (13)$$

m_0 is the anhydrous wood mass before heat treatment.

The experiments are carried out in such a way, allowing the determination of the model parameters over two stages. At the first, with the isothermal area at 165 °C, the reaction given by the Eq. 5 can be neglected, because of its very slow rate. It has also been considered that the reaction enthalpy of the first reaction is negligible. The three parameters relative to the first reaction (Eq. 4) are then determined: α_1, k_{01}, and Ea_1. The second isothermal area at 240 °C, corresponding to the heat treatment process, allows the determination of the five other parameters: α_2, γ_2, k_{02}, Ea_2, and Hp. A random algorithm has been used, and the minimum and maximum values of each parameter have been initialised according to the values available in the literature [39, 42, 43, 47].

The governing equations are solved numerically with the commercial package, FEMLAB [48]. This is designed to simulate systems of coupled, non-linear, and time dependent partial differential equations (PDE) in one-, two-, or three- dimensions. The mesh convergence was verified with refined mesh sizes. Time step of 360s, 1866 nodes, and 8080 elements mesh size were considered to be appropriate.

The kinetic parameters obtained using the method described are in Table 1.

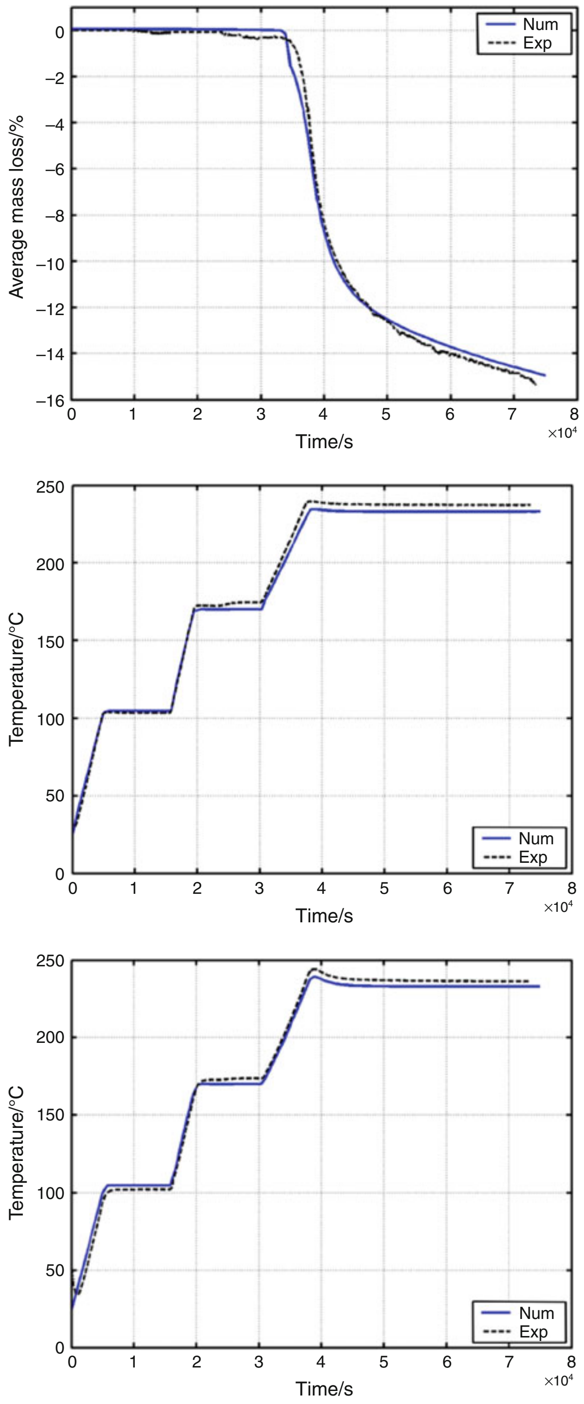

Fig. 7 Time evolution of average weight loss, surface temperature, and center temperature: Poplar (0.5 °C min^{-1}, $T_{\max} = 230$ °C).

Results and discussion

Effect of maximum temperature

This section is devoted to the investigation of the effect of temperature of the heat treatment on the anhydrous mass loss, the parameter, chosen as the controlling parameter for the heat treatment process. Three experiments have been carried out using poplar wood during 20 h under heat treatment at temperatures 210, 220, and 230 °C, respectively. The heat treatment *d* rate has been fixed to 1 °C min^{-1}. Experimental and numerical results are shown on Figs. 3, 4, and 5. It can be observed that the increase of

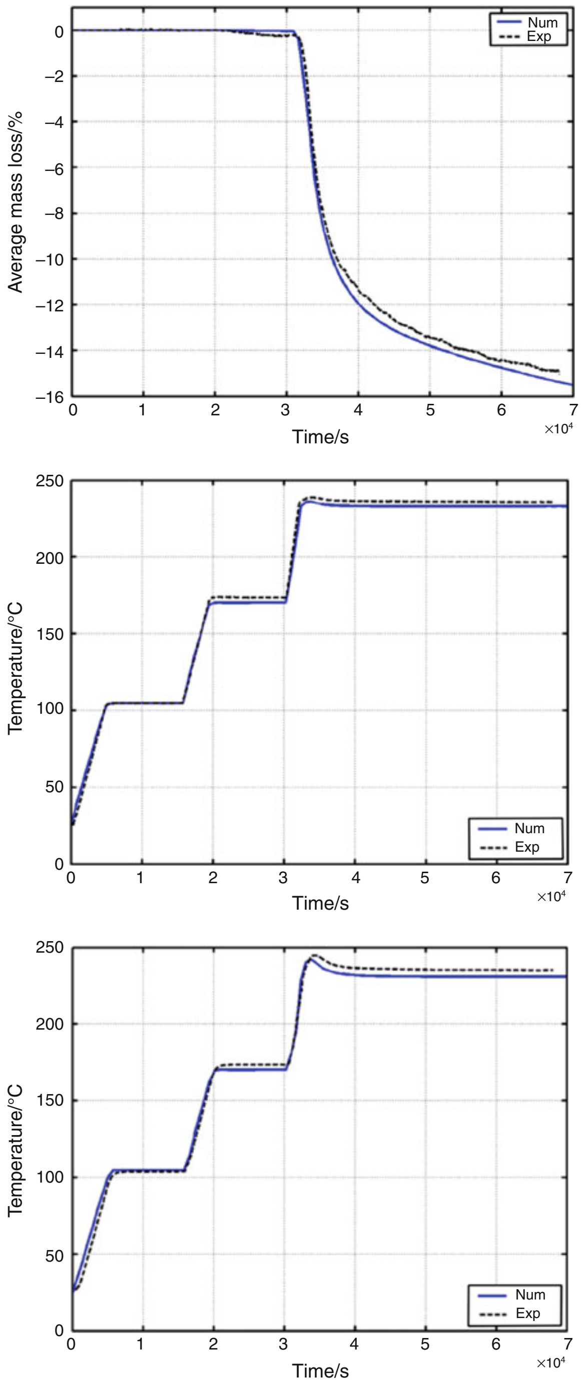

Fig. 8 Time evolution of average weight loss, surface temperature, and center temperature: Poplar (2 °C min^{-1}, $T_{\max} = 230$ °C).

the treatment temperature enhances the AML. A good agreement is observed between the experience and the numerical simulation.

Effect of heating rate

In this paragraph, the study has been performed on the poplar wood, under treatment temperature of 230 °C and for different heating rate of 0.25, 0.5, 1, and 2 °C min^{-1}, respectively. Results are shown on Figs. 5, 6, 7, and 8.

Conclusions

The kinetics for the pyrolysis of wood was investigated. Based on the laboratory experiments performed with the described device, a mathematical model for the kinetics of the pyrolysis process was proposed and validated. This model incorporates the reaction pathway that was developed by Grioui et al. [43], for the description of the transformation of wood subject to heat. This model allows the computation of internal profiles of temperature and average mass loss. The Femlab [48] software was used to solve the system of partial differential equations. This study includes a validation section, that shows the ability of the model to accurately predict the internal profiles of temperature, and mass loss in the experimental configuration. Comparison of the simulated temperature profiles and average mass loss with experimentally measured values showed reasonable agreement.

Further studies may allow more accurate modeling of wood torrefaction mainly, by taking into account the thermodegradation of lignin and accurate thermo physical properties. This model can be used in the design of industrial pyrolysis installations.

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