

## THERMAL UPGRADING OF SUSTAINABLE WOODY MATERIAL: EXPERIMENTAL AND NUMERICAL TORREFACTION ASSESSMENT

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**ABSTRACT:** Torrefaction is a promising technology to produce woody sustainable materials and biochar for combustion and gasification. To aid in the process and reactor design concept, numerical models are applied to predict processes parameters giving treatment time estimation, solid and volatile yields, and calorific values of the solid fuels. In this work, a hardwood (*Eucalyptus grandis*) was investigated by using thermogravimetric analysis (TGA), as well as elemental analysis, which can reveal the thermal events and identify degradation products as a function of treatment time and temperature. A numerical prediction based on a two-step kinetics model was employed to predict solid yield, elemental composition, higher heating values (HHV), and its enhancement factor during the treatment. The further analyzed prediction 3D surfaces and profiles allows clear identification of the torrefaction severity. The predicted HHV's showed enhancement factors up to 10.2% for severe torrefaction. The obtained kinetic rates for *Eucalyptus grandis*, as well as the numerical results, can provide useful information for industrial operation and reactor projects.

**Keywords:** biomass, torrefaction, thermochemical conversion, upgrading, modeling

## 1 INTRODUCTION

The demand for environmentally acceptable replacements for fossil fuels has been increasing the use of renewable energy sources [1]. Eucalyptus woody biomass has gained attention in Brazilian cellulose and energy industries due to fast-growing, productive, and easily flexible tree species [2–6]. However, its raw solid wood presents some inherent drawbacks such as low energy density, hydrophilic nature, elevated moisture, and highly variable composition and properties [7].

Biomass pretreatment, like torrefaction (mild pyrolysis), is reported to be a feasible thermochemical conversion route to overcome some of the raw biomass drawbacks [7]. Torrefaction is a thermochemical process generally operating at low temperatures (200–300 °C) in the absence of oxygen for biomass thermal upgrading for solid fuel production [7–9].

Nowadays, numbers of experimental [10–15] and numerical studies [16–25] have been conducted to characterize and comprehensively examine the biomass torrefaction process. The two-step consecutive reactions kinetic model [26] was widely employed to torrefaction modeling conducting isothermal numerical predictions [22].

Past studies have employed the two-step model to obtain the ultimate composition dynamics throughout the torrefaction treatment [16,25,27,28]. However, experimental indirect method issues related to volatiles collection and analysis, as well as the lack of observed points in the regression method, are not feasible for industrial applications.

Therefore, this study aims to perform an experimental and numerical thermal upgrading assessment for *Eucalyptus grandis*. For that, a kinetic model [23] was applied to obtain the kinetic parameters. The ultimate analysis of feedstock and final product, with the calculated kinetics based on [22], allowed to obtain the C, H, and O dynamics evolution during the process. The 3D surfaces and numerically predicted profiles for the evaluated properties permitted the torrefaction assessment providing valuable perceptions into the biofuel upgrading.

## 2 MATERIAL AND METHODS

### 2.1 Material

The biomass used was *Eucalyptus grandis* due to its large planting in Brazil [2]. A tree was extracted from the University of Brasília property for wood species controlled growing [15]. The biomass samples were dried in an oven at 104 °C until mass stabilization. Then, the samples were grounded and sieved (60 mesh). The proximate and ultimate analyses, as well as energy content values for the raw material, are shown in Table I.

**Table I:** Elemental and calorific analyses of raw samples.

Raw material	<i>Eucalyptus grandis</i>
<b>Elemental analysis <sup>a</sup></b>	
C	44.28
H	5.65
N	0.22
O <sup>b</sup>	49.85
<b>Chemical formula</b>	CH <sub>1.52</sub> O <sub>0.85</sub> N <sub>0.004</sub>
<b>HHV (MJ kg<sup>-1</sup>)</b>	18.08

<sup>a</sup> Dry-ash-free, <sup>b</sup> O (by difference) (wt%) = 100–C–H–N

### 2.2 TGA apparatus and procedure

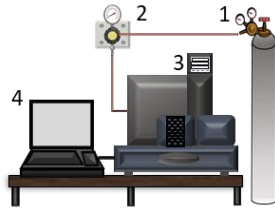
The torrefaction apparatus displayed in Fig. 01 consists of a nitrogen steel cylinder, a rotameter, a reaction unit (SDT Q600 TA), and a computer to system control and data processing. Nitrogen was controlled by the rotameter at a flow rate of 50 mL.min<sup>-1</sup> and was used to provide an inert atmosphere. The thermal behavior of the samples (15 mg) were evaluated by the calculated solid yield ( $S_Y$ ) over time, according to Eq. (01) [22,29–31], providing the instantaneous mass variation (TGA).

$$S_Y^{(T)}(t) = \frac{m_i(t)}{m_0} \times 100 \quad (1)$$

where  $m_0$  is the dried mass before torrefaction;  $m_i$  is the solid mass during torrefaction,  $t$  is the residence time and  $T$  the experiment temperature. The treatment parameters are listed in Table II.

**Table II:** Torrefaction parameters.

	Duration	Heating rate	Final temperature
Drying	30 min	20 °C.min <sup>-1</sup>	104 °C
Torrefaction	60 min	5 °C.min <sup>-1</sup>	210 °C
			230 °C
			250 °C
			270 °C
			290 °C

**Figure 1:** Experimental system. 1) N<sub>2</sub> cylinder, 2) Gas control rotameter, 3) SDT Q600 TA, 4) Computer.

### 2.3 Chemical analysis

The elemental analysis was conducted in a Perkin Elmer EA 2400 series II elemental analyzer according to the ASTM E777 and E778 to detect the mass percentages of *C*, *H*, *N* for raw and torrefied biomass [15]. The oxygen content *O* was calculated by difference [15].

### 2.4 Thermodegradation kinetics

A previous study developed a three-stage approach kinetics model [23] for biomass torrefaction kinetic prediction. This model was employed in this study. The model, originally proposed by [26], uses a first-order mechanism composed by a two-step consecutive reactions and four reaction rates constants  $k_i$  (min<sup>-1</sup>,  $i = B, C, V_1, V_2$ ) defined by the Arrhenius law [23]. In this approach, the torrefaction products are lumped into five pseudo-components: solid (feedstock *A*, intermediate solid *B* and residue *C*) and volatiles  $V_1$  and  $V_2$ , as Fig. 02 shows in [26]. The time cumulative solid yield is described by the sum of masses of *A*, *B*, and *C*, while the total mass of volatiles is described by the sum of  $V_1$  and  $V_2$  [26].

The solid ( $k_B$ ,  $k_C$ ) and volatile ( $k_{V1}$ ,  $k_{V2}$ ) reaction rates are calculated (based on the pre-exponential factors  $k_{0,i}$  and activation energies  $E_{a,i}$ ) with Eq. (03) in [23] by fitting numerical profiles to the experimental solid yield  $S_Y^{(T)}(t)$  using a fmincon minimization function in Matlab® [23].

### 2.5 Composition and HHV prediction

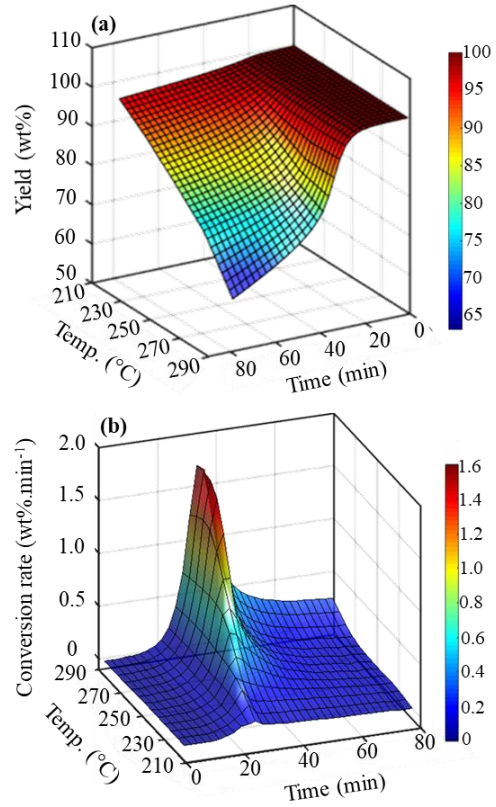
The solid composition model developed in a past study [22] was employed. The simple and accurate numerical prediction allows the estimation of solid carbon (*C*), hydrogen (*H*) and oxygen (*O*) evolution based on the kinetics and the initial (raw biomass) and final (torrefied product) elemental analysis [15,22]. The solver uses the minimization solver *Nelder-Mead* (Matlab® software) [15,22].

The HHV dynamics in time was calculated based on the past study [17] and the empirical correlation proposed by [32] and presented in Eq. (16) in [22], where the *C*, *H*, and *O* are the mass percentages of carbon, hydrogen, oxygen on a dry-ash free basis.

## 3 RESULTS AND DISCUSSIONS

### 3.1 Experimental wood torrefaction

The normalized (after drying process) solid yield and DTG 3D surfaces are presented in Figure 02 (a) and (b) to evaluate the thermal degradation.

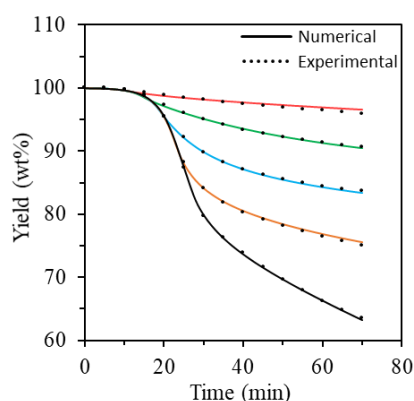
**Figure 02:** TG (a) and DTG (b) 3D surfaces.

The torrefaction treatment severity strongly affected wood thermodegradation and the normalized mass loss values were 3.39 (210 °C), 9.55 (230 °C), 16.57 (250 °C), 24.55 (270 °C) and 36.72 wt.% (290 °C), in agreement with [33]. The treatments until 235 °C has a slight degradation (maximum of 12.2 wt.% at 235 °C). Throughout mild to severe torrefaction, mass degradation varies from 12.2 to 40 wt.%.

The solid yield derivative surface is plotted in Fig. 02(b). Solid conversion starts around 180 °C (18 min), agreeing with reported results that showed the thermal degradation starting temperatures of 180–200 °C [34,35]. Temperature treatments of 210, 230, and 250 °C had a maximum decomposition of 0.17, 0.33, and 0.86 wt.%.min<sup>-1</sup>, respectively. Severe treatments (270 and 290 °C) results varied between 1.72 and 1.80 wt.%.min<sup>-1</sup>

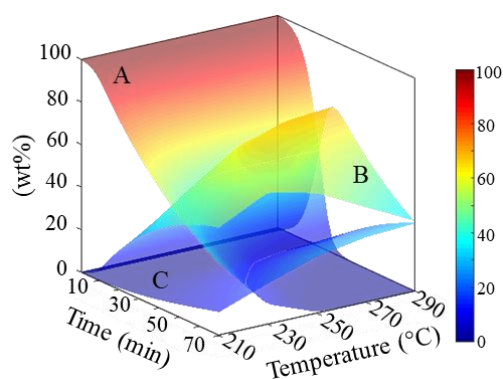
### 3.2 Eucalyptus torrefaction kinetic modeling

The simulation results for numerically predicted solid yield are displayed in Fig. 03. The obtained kinetic parameters are disposed of in Table III. For a better convergence time, mass yields evolutions are presented after the 160 °C treatment temperature [23]. Fig. 03 depicts that the predicted curves are in good agreement with the experimental results. A good fitting has been achieved at the beginning and the end of the treatment process using the thermal sensitivity three-stage approach [23].



**Figure 3:** Comparison between experimental (markers) and numerical (lines) solid yields obtained with the three-stage approach (a).

Analyzing the obtained kinetic rates in Table III, it is possible to observe that, for Eucalyptus torrefaction, similarly to the pure Xylan, Willow, Poplar, and Fir [23,26], the ranking of reaction rates from largest to smallest is  $k_1 > k_{v1}$  in the first step and  $k_2 > k_{v2}$  during second step as pointed out by [23,25,26,36]. The 3D surfaces of the solid (A, B, and C), and volatile ( $V_1$  and  $V_2$ ) pseudo-components evolution predicted with obtained kinetic rates are displayed in Fig. 04 and 05, respectively.



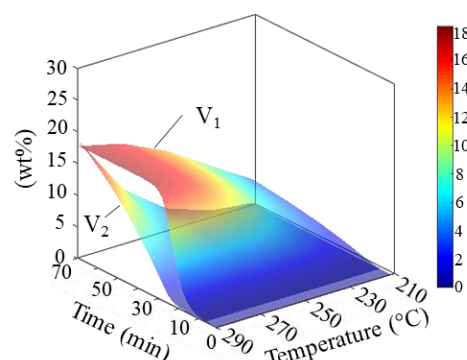
**Figure 4:** 3D surfaces of (a) Feedstock A, (b) intermediate product B, and (c) residue C during the thermal upgrading.

Until 250 °C treatment, there was a remaining extent of A in the final product. For treatments temperatures higher than 250 °C A is entirely transformed into B and volatile  $V_1$  at the end of treatment. For temperatures higher than 275 °C, A is entirely consumed with 38 min residence time. For treatments between 210 and 250 °C, corresponds to 50–80 wt.% of torrefied wood agreeing with [22,28]. For temperatures higher than 275°C, the main extent of torrefied wood changed into residue C, corresponding to 20–40 wt.% of torrefied product.

**Table III:** Calculated kinetic parameters.

Reaction step	Kinetics parameter	
First step reaction	$k_{0,i}$ (min <sup>-1</sup> )	$E_{a,i}$ (J.mol <sup>-1</sup> )
$A \rightarrow B$	2.76E+07	8.53E+04
$A \rightarrow V_1$	7.00E+11	1.37E+05
Second step reaction		
$B \rightarrow C$	2.31E+00	2.43E+04
$B \rightarrow V_2$	1.06E+09	1.19E+05

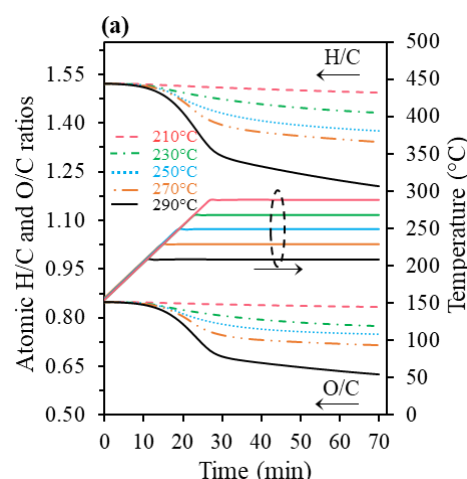
The 3D surfaces of volatiles releasing are displayed in Fig. 05, showing a markedly growing with increasing treatment temperature. The experimental mass loss reduction is numerically established through the  $V_1$  and  $V_2$  volatile productions [23]. The solid yield decrease is mainly due to the production of  $V_1$  for light and mild severity treatments. Concerning  $V_1$ , the volatile releasing becomes steady after 38 and 50 min for 290 °C and 275 °C, respectively. The importance of  $V_2$  increase after 235 °C temperature and becomes more important than  $V_1$  after 290 °C treatment.



**Figure 5:** 3D surfaces of volatiles (a)  $V_1$  and (b)  $V_2$  during the thermal upgrading.

### 3.3 Solid composition

The calculated kinetics and ultimate experimental analysis allowed the construction of O/C and H/C atomic ratio profiles displayed in Fig. 6.



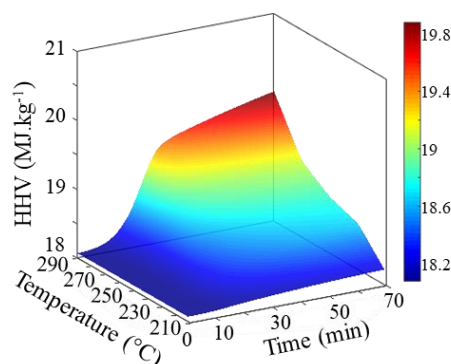
**Figure 6:** Predicted profiles of H/C and O/C ratios during the thermal upgrading.

The elemental composition information is conducive to evaluating the heat treatment intensity of wood materials and the treatment performance [37,38]. The calculated atomic H/C and O/C ratios of the raw and torrefied wood reported a decrease after undergoing torrefaction due to higher hydrogen and oxygen releasing and carbon retained in solid. Higher carbon, lower hydrogen, and lower oxygen contents were obtained for higher temperature treatments, as expected [22].

### 3.4 HHV prediction

In this study, the 3D surface of HHVs was also

obtained from the calculated ultimate composition allowing to evaluate the potential of torrefied wood as a solid fuel [17]. The assessment shows a smaller variation for lower temperatures, and more pronounced after 20 min and 250 °C temperatures. The HHV were 18.18 (210 °C), 18.66 (230 °C), 18.82 (250 °C), 19.11 (270 °C) 19.91 (290 °C) MJ.kg<sup>-1</sup>.



**Figure 10:** The 3D surface of HHV and during the thermal upgrading.

#### 4 CONCLUSIONS

Eucalyptus hardwood torrefaction between 210–290 °C has been successfully evaluated by prediction profiles and 3D surfaces. The employed three-step kinetic approach was able to obtain excellent results for the complete range treatment range. The evolution of the solid proportions (*A*, *B*, and *C*) result agrees with literature data suggesting that for treatment temperatures higher than 235 °C, feedstock *A* is already entirely converted, and the intermediate (*B*) starts to be consumed. For lower temperatures *V*<sub>1</sub> represents 85% of the produced volatiles. The importance of *V*<sub>2</sub> increases after 230 °C temperature treatment. The atomic H/C and O/C ratios were decreasing throughout torrefaction treatment. The enhancement factors of HHVs were in a range of 1.006 and 1.102 for treatments between 210 and 290 °C. Overall, the experimental and numerical assessment shows that most of the essential information for a torrefaction process can be predicted, and process optimization can be carried out with some additional information.

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