Determination of Time-to-ignition and Flaming Duration of Biomasses in a Muffle Furnace

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Abstract
Measuring the energy characteristics of solid biofuels can help to determine the most suitable species for combustion. The objective of this study is to propose a new methodology for determining the ignition time and flaming duration in lignocellulosic biomass. A muffle furnace was used, instead of an epiradiator, to measure the variables. The optimal oven temperature was defined according to the average time-to-ignition of biomasses in the literature. Ten biomasses were analyzed to obtain their high heating value, volatile matter, fixed carbon content, ash content, time-to-ignition, and flaming duration. The results showed a high correlation between the biomass volatile content, time-to-ignition, and flaming duration. In the literature, it is described that high levels of volatile materials accelerate the ignition of the material. Thus, the association between the volatile matter and the variables analyzed justifies the use of the muffle furnace methodology. Furthermore, biomasses with high levels of volatile matter have longer flaming durations than other solid biofuels.

Introduction
Biomass can be used as a solid fuel. To assess the potential of solid biofuels, different energy variables can be measured through proximate analysis. This is a simple, low-cost, and widely used methodology [1, 2] in which dry biofuel is divided into three content fractions: ash, volatile matter, and fixed carbon [3–6].

The volatile matter of biomass consists of incinerable gases (hydrogen, light hydrocarbons, and tar) and non-incinerable gases (water and carbon dioxide) [7]. Incinerable gases increase reactivity and the rate of combustion, thus preventing high temperatures from being reached [8, 9]. In non-pretreated biofuels, such as firewood and lignocellulosic waste, volatile materials can constitute 65–85% of the material.

The flammability of a solid fuel is one of the main variables influencing the combustion process [10]. Flammability is related to the biofuel's ability to ignite, characteristics that maintain biofuel burning, speed at which biofuels burn, and amount of biofuel burned [11].

According to Kovalsky et al. [12], parameters such as the ignition time (TTI) and flaming duration (FD) provide the characteristics of the flammability of biofuels. The procedure to determine these variables requires an epiradiator, which radiates heat to the tested samples [13–15]. Muffle furnace equipment, commonly found in laboratories, also releases heat and can be used for the same procedure. Gill and Moore [16] tested a muffle furnace at 400°C to measure the TTI; however, the study was not published.

Thus, the objective of this study was to test a muffle furnace to validate a new methodology for determining the TTI and FD of lignocellulosic materials.

Material And Methods
Collection of materials
The study was conducted in the Biomass and Bioenergy Laboratory at the Federal University of São Carlos, Sorocaba campus, at the geographical coordinates of 23° 34'52,72”S and 47°31’36,02” W.

Lignocellulosic residues were collected from *Pinus elliottii* (sawdust), *Tectona grandis* (sawdust), *Eucalyptus urophylla, E. grandis* (branch), *Saccharum officinarum* (thatch remnants), *Bambusa vulgaris* (culm), *Attalea speciosa* (remnants of the mesocarp), *Ficus elastica* (sawdust), *Cocos nucifera* (remnants of the mesocarp), *Apuleia leiocarpa* (sawdust), and *Dinizia excelsa* (sawdust). The samples were dried for 24 h at 103 °C and ground in a Wiley knife mill (Marconi, MA340). Subsequently, the material was passed through an 18-mesh sieve [17]. Figure 1 presents the methodology.

**Time-to-ignition and flaming duration**

The time-to-ignition (TTI) and flaming duration (FD) were adapted from Silva et al. [10] and Kovalsyki et al. [12]. The equipment used was a muffle furnace (Bravac M2/3). Initially, a test was conducted to define the optimal muffle furnace temperature (600 °C, 700 °C, 750 °C, and 800 °C) using one type of biomass (*T. grandis*). For the time analysis (TTI and FD), one gram of biomass was placed in a 25 ml-crucible positioned in the center of the muffle furnace, with the door open and at the adjusted temperature of the calibration. The stopwatch was started at the instant when the sample was placed in the muffle furnace. The TTI (s) was determined at the time of the emergence of the flame. After the flame appeared, the crucible was positioned on the edge of the muffle furnace. The time between the appearance and the absence of the flame was called FD (s). Ten repetitions were performed for each sample.

**Proximate analysis**

Samples from each biomass were separated. One gram of each sample, with a particle size greater than an 18-mesh sieve, was placed in a calcined crucible with a lid. The sample was placed in a 900°C muffle furnace for 7 min [17]. After the material was cooled in a desiccator, it was possible to calculate the volatile matter according to below:

\[
VM(\%) = \left[ \frac{(m_i - m_f)}{m_i} \right] \times 100
\]

Where \(m_i\) is the initial dry mass, \(m_f\) is the final mass after 7 min, and VM is the volatile matter.

To calculate the ash content, one gram of each sample (granulometry greater than 18 mesh) was placed in a calcined crucible. The sample was placed in a muffle furnace. The heating (20°C.min-1) was from 27°C to 250°C, and the temperature then remained at 250°C for another hour. After the elapsed time, the muffle furnace temperature was raised to 550°C. Upon reaching this new temperature, the sample remained in the oven for another two hours. The test was considered complete when the sample reached a constant mass [18]. The ash content (A) was calculated as follows:

\[
A(\%) = \left( \frac{m_f}{m_i} \right) \times 100
\]
Where $m_i$ is the initial dry mass, $m_f$ is the final mass, and $A$ is the ash content.

To calculate the fixed carbon content (FC) was calculated as follows:

$$\text{FC}(\%) = 100 - (VM + A)$$

**Higher heating value**

The tests of higher heating value (HHV) were carried out with dry biomass and with a particle size greater than 18 mesh. The HHV of Biomass was estimated by the calorimetric pump (IKA C 200). The appliance uses 2 L of water per analysis. The water temperature was adjusted in a range from 18 to 23º C. The biomass was pressurized to 30 bar oxygen in a calorimeter vessel. These conditions attest to the requirements of the standard ASTM D5865 [2].

**Results And Discussion**

Table 1 presents the results of the TTIs at respective temperatures.

| Temp. (°C) | TTI (s) | Model (°C): | $R^2$ | Temp. ignition (°C) |
|------------|---------|-------------|-------|---------------------|
| 600        | 96.0    | $\text{TEMP} = 120.82\ln(TI) - 197.86$ | 0.989 | 353.60              |
| 700        | 62.0    | $\text{TEMP} = 4.06(TI) + 119.06$ | 0.991 | 371.32              |
| 750        | 39.0    | $\text{TEMP} = 5.86(TI) + 108.5$ | 0.996 | 337.04              |
| 800        | 34.5    | $\text{TEMP} = 8.18(TI) + 119.3$ | 0.991 | 401.51              |

* Coefficient of Determination ($R^2$); Time-to-ignition (TTI); Temperature (Temp.)

The TTI of 39 s (750°C) for the biomass sample was the closest to the TTI values obtained by Silva et al. [10] and Kovalskyi et al. [12], which were 41 s on average. Therefore, in this study, a temperature of 750°C was established for testing. It was also the model that best presented the linear correlation coefficient (Table 1, Fig. 1).

Protásio et al. [19], during a thermogravimetric analysis, found the first peak of thermal degradation at a combustion temperature of 329°C for babassu bark, whereas for *Pinus densiflora*, the first peak was found at 340°C. These values are close to the 750°C (337°C) model.

It is noteworthy that the spontaneous combustion of wood occurs at temperatures close to 280°C [20]. However, at the moment when biomass depolymerization occurs, exothermic reactions are still insufficient to observe the appearance of a flame. Flames appeared at temperatures above 337°C.

Table 2 shows the results of burning and the higher heating value for different biomass species.
**Table 2**
Results of the ammability test, proximate analysis, and higher heating value for the species studied.

| Biomass       | TTI (s) | FD (s) | VM (%) | FC (%) | A (%) | HHV (J/g) |
|---------------|---------|--------|--------|--------|-------|-----------|
| *P. elliottii*| 34.5±2.7| 128.5±0.8| 87.44±0.22| 12.32 | 0.24±0.03| 20381± |
| T. grandis    | 39.0±0.6 | 88.8±0.7 | 81.41±0.09 | 14.05 | 4.54±0.11| 19163± |
| *E. urograndis*| 37.8±5.6| 85.5±0.9 | 81.52±0.19 | 15.60 | 2.88±0.12| 18798± |
| *S. officinarum*| 38.0±7.3| 101.8±14 | 83.85±0.29 | 13.71 | 2.44±0.03| 17988± |
| B. vulgaris   | 51.0±6.8 | 34.4±0.5 | 75.95±0.30 | 19.01 | 5.04±0.17| 18602± |
| A. speciosa   | 43.3±4.6 | 74.3±0.5 | 76.93±0.42 | 17.41 | 5.66±0.18| 17531± |
| F. elástica   | 39.8±5.0 | 78.0±0.7 | 81.11±0.85 | 17.10 | 1.79±0.15| 19123± |
| C. nucifera   | 56.3±7.7 | 40.7±0.8 | 71.10±0.20 | 23.78 | 5.12±0.07| 18139± |
| A. leiocarpa  | 37.9±3.7 | 92.6±0.7 | 80.61±0.33 | 16.97 | 2.42±0.12| 19461± |
| D. excelsa    | 41.3±6.2 | 101.6±0.8 | 81.30±0.27 | 18.05 | 0.65±0.01| 19494± |

*Time-to-ignition (TTI); flaming duration (FD); volatile matter (VM); fixed carbon content (FC); ash content (A); higher heating value (HHV)

*C. nucifera* presented the most optimal results for combustion, with a high TTI (56 s) and short flaming duration FD (41 s). According to Kovalsyki et al. [12], the higher the TTI and shorter the FD, the better is the fuel.

*P. elliottii* presented burns with high intensities, with a short TTI (34.5 s), long FD (128.5 s), greater amount of fuel burned (20381 J/g), and lower ash content (0.24).

Ash values above 1% indicate the presence of impurities. Most of the material for the samples was collected from sawdust on a floor, where impurities may have been present in silica.

According to Rybak et al. [21], the TTI measures the interval from the moment the sample is introduced into a hot atmosphere until the appearance of a flame around the sample. This relationship was observed in the muffle furnace. Figure 3 shows the relationship between the content of volatile materials, TTI, and FD.

Figure 3 (a) shows that the higher the volatile content, the smaller the TTI of the material, presenting a strong correlation between the variables ($\rho = -0.93$). This is consistent with the literature [7–9]. In addition,
the higher volatile matter in biomass results in a higher reactivity and a lower ignition temperature for biomass compared to coal [22]. Rybak et al. [21] stated that the TTI varies according to the volatile matter.

Figure 3 (b) shows that the lower the volatile content, the shorter the FD of the tested biomasses, showing a strong correlation (\(\rho = +0.92\)). In the tested samples, homogeneous ignition was observed; that is, when ignition occurs first in the gas phase, the flame envelope around the sample prevents oxygen from reaching the sample surface [21, 22].

**Conclusion**

Muffle furnace methodology is effective, relatively simple, inexpensive, and fast, allowing for the determination of the TTI and FD of different samples of biomass. The volatile material content of the samples determines the TTI and FD of the biomass.

**Declarations**

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**Availability of data and material (data transparency):** xlsx file attached.

**Code availability:** Not applicable.

**Authors' contributions** Diego Aleixo da Silva: Conceptualization, Methodology, Validation, Formal analysis, Investigation, Writing - Original Draft, Writing - Review & Editing, Visualization. Natália Rodrigues de Carvalho: Methodology, Investigation, Writing - Review & Editing. Ariane Aparecida Felix Pires: Collection and treatments of materials and higher heating value analysis. Elias Ricardo Durango: proximate analysis. Ana Larissa Santiago Hansted: Investigation, Writing - Review & Editing. Fábio Minoru Yamaji: Conceptualization, Methodology, Supervision, Writing - Review & Editing.

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Figures

![Figure 1](http://aff.org.au/wp-content/uploads/Gill_ignitibility_final.pdf)

**Figure 1**

- Collection of the 10 different biomasses
- Standard sample (*T. grandis*)
- Temperature calibration
- Trials with 10 biomasses
- Correlation of VM and TTI; VM and FD. Methodology confirmation.
- Proximate analysis (VM, FC, and A)
- TTI and FD
Flowchart of the process to test the muffle furnace as a new methodology. Time-to-ignition (TTI); flaming duration (FD); volatile matter (VM); fixed carbon content (FC); ash content (A)

**Figure 2**

Temperature versus time curves adjusted for different muffle furnace temperatures.

**Figure 3**

Volatile matter as a function of ignition time (a) and flame duration (b)