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# Characterization of briquettes produced from eucalyptus wood waste generated in agro-industries<sup>1</sup>

Caracterização de briquetes produzidos a partir de resíduos de madeira de eucalipto gerados em agroindústrias

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## HIGHLIGHTS:

*Eucalyptus wood residues were compacted in dense blocks to evaluate the burning properties. Detailed description of the physico-chemical and thermal properties of the wastes were studied. The effect of the compaction pressure of the briquettes showed that the burning of the material occurs more slowly.* 

**ABSTRACT:** Boilers are widely used by industries and thermoelectric plants to generate renewable energy. However, when biomass is consumed, residues are generated, which for the most part cannot be discarded appropriately. This study investigated the reuse of eucalyptus wood waste with the goal of transforming the residue into resistant and durable briquettes for reuse in furnaces and boilers. The physico-chemical properties of the residue were studied in terms of chemical composition by X-ray fluorescence, and its crystalline structure was evaluated by X-ray diffraction. Both thermogravimetric analysis and differential thermogravimetric analysis were used to characterize the thermal decomposition of the wood waste residue. The residue was compacted in a universal press with sufficient humidity to fabricate briquettes under different conditions of mechanical pressure. Apparent density, durability, and burning tests were carried out on the briquettes. The maximum temperature reached during burning is dependent on the fabrication compaction pressure. The briquettes proved suitable for burning in boilers and furnaces, and their durability is advantageous for handling, reuse, and transportation.

Key words: densification of briquettes, thermal analysis, physico-chemical properties, combustion

**RESUMO:** Caldeiras são amplamente utilizadas por indústrias e usinas termelétricas para gerar energia renovável. Porém, quando a biomassa é consumida, resíduos são gerados, que em sua maioria não podem ser descartados de forma adequada. Este estudo investigou o reaproveitamento do resíduo de madeira de eucalipto com o objetivo de transforma-lo em briquetes resistentes e duráveis para reaproveitamento em fornos e caldeiras. As propriedades físicoquímicas do resíduo foram estudadas em termos de composição química por fluorescência de raios-X, e sua estrutura cristalina foi avaliada por difração de raios-X. Ambas análises termogravimétrica e termogravimétrica diferencial foram utilizadas para caracterizar a decomposição térmica do resíduo de madeira. O resíduo foi compactado em prensa universal com umidade suficiente para a fabricação de briquetes sob diferentes condições de pressão mecânica. Os testes de densidade aparente, durabilidade e queima foram realizados nos briquetes. A temperatura máxima atingida durante a queima depende da pressão de compactação de fabricação. Os briquetes se mostraram adequados para queima em caldeiras e fornos, e sua durabilidade é vantajosa para manuseio, reutilização e transporte.

Palavras-chave: densificação dos briquetes, análise térmica, propriedades físico-químicas, combustão

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#### **INTRODUCTION**

In recent decades, population growth has increased the demand for electricity. This has increased the use of fossil fuels, which due to environmental concerns has now led to the search for alternative technologies to meet this energy demand. In Brazil, the 'Operador Nacional do Sistema Elétrico' (ONS) is responsible for coordinating and controlling electricity generation and transmission facilities. According to ONS (2016), between March 2016 and February 2019, thermoelectric plants generated 17.8% of all electricity consumed in Brazil. However, the increased consumption of fossil fuels intensifies environmental pollution and the emission of polluting gases. Thus, to circumvent the effects of this increasing pollution, researchers have become interested in renewable energies, such as the search for alternative fuels, including biofuels, to replace fossil fuels (Cardona et al., 2019; Chen et al., 2015; Davis & Socolow, 2014).

Waste can be compacted into dense blocks, usually called briquettes. Sette Jr et al. (2018), evaluated the use of briquettes from the bark of the eucalyptus, and according to the authors briquettes stand out as an energy source due to their densification during their fabrication process compared to residual shoot. Nunes et al. (2019) studied the production and characteristics of the briquettes composed of *Eucalyptus pellita* wood residues with the addition of *Cocos nucifera*, to study the hypothesis that the addition of wood residues with a high calorific content and low ash concentration increases the energy characteristics. Santos et al. (2020) determined the energetic quality of residues from the jatropha and eucalyptus wood waste composites. According to the authors, the jatropha residue has high ash content and higher concentrations of inorganic materials, which showed greater calorific value.

The aim of this study was to determine the physicochemical and thermal characteristics of briquettes produced from eucalyptus waste to study the effect of compaction pressure on the burning properties.

## MATERIAL AND METHODS

The eucalyptus wood residue was collected from agribusiness in October 2018. The physico-chemical and thermal analyzes were carried out in the following year, with all analyzes completed by August 2019. The location of agribusiness is the region of Rio Verde, Goiás, Brazil (17° 49' 23" S; 50° 57' 24" W, and altitude of 760 m). The waste comes from the burning of eucalyptus chip at 800 °C in a fluidized bed boiler. The samples were dried in a drying oven at 105 °C ( $\pm 2$  °C) to remove any moisture from the residue.

The briquettes were formed under controlled pressure using a universal press. In the structural laboratory of the Instituto Federal Goiano, Rio Verde Campus, they were compressed by up to 37.50 MPa at room temperature for 5 min, and then allowed to cool to room temperature. Figure 1A shows a schematic representation of the sample holder used during pressing, with internal dimensions of H = 0.05 m, W = 0.05 m and L = 0.05 m. It was necessary to adjust the moisture content of the residue to 50%. Figure 1B illustrates a sample of the pressed briquette produced from the eucalyptus wood waste.



**Figure 1.** (A) Schematic representation of the sample holder used to press the residue to obtain the briquette, (B) briquettes of eucalyptus wood waste

As reported in other studies (Sette Jr et al., 2018), the apparent density of the briquettes was calculated according to the following equation:

$$D_{ad} = \frac{M_i}{lwh}$$
(1)

where:

- $D_{ad}$  apparent density (kg m<sup>-3</sup>);
- $M_{i}$  initial mass of the briquettes with 50% humidity (kg);
- l length(m);
- w width (m); and,
- h sample height (m).

In general, the samples produced showed the same measurements as the width and length of the sample holder, with the height of the briquettes varying between 0.05 and 0.005 m while the applied pressure varied from 0 to 37.50 MPa. The volumetric expansion of the briquettes was calculated by means of their geometric measurement with the aid of a digital caliper at two different times: (i) immediately after the briquetting and (ii) 72 hours after the briquetting. This period was chosen in line with literature on the stabilization of the volumetric expansion of briquettes.

The durability of the briquettes was tested using a vibrating sieve (i.e. loss of mass), as adapted from the methods used by Liu et al. (2014), Sette et al. (2018), and Toscano et al. (2013). Each briquette was weighed on a precision scale and then placed on a 6.30-mm size sieve shaker with a base to capture the resulting sediment. Then, with the sieve closed, the agitator was turned on at a frequency of 10 rpm for 2 min. To finish the test, the material retained on the base of the agitator was weighed on the precision scale to assess the loss of mass, thus calculating the percentage mass loss ( $\Delta W$ %). The test was successively repeated for all the briquettes formed at the different pressures.

The chemical composition and thermal behavior of the residue were evaluated after compaction and molding the residue into briquettes. The chemical composition of the eucalyptus wood residue was determined by an X-ray fluorescence (XRF) spectrometer (S8 Tiger WDS; Bruker) equipped with an Rh tube. An aliquot of approximately 6.0 g of the sampled residue was separated by manual quartering, dried at 105 °C for 12 hours, and then used to determine the loss on ignition (LOI). To determine the LOI, the sample was calcined at 1000 °C for 5 hours, with 1.0 g of each sample previously calcined and melted with 9.0 g of lithium tetraborate. In this analysis, five repetitions were performed and the mean value was obtained for the elements present. The crystalline structure of the residue was investigated by powder X-ray diffraction (XRD). Monochromatic radiation from a copper anode tube was used, operating at 40 kV and 40 mA with a Bragg-Brentano configuration over the 2 $\theta$  range from 10° to 80°.

To describe the thermal stability of the eucalyptus wood residue, the thermogravimetry (TG) and differential thermogravimetry (DTG) of the eucalyptus wood residue were investigated by thermal analysis (DTG-60H; Shimadzu). The measurements were carried out in the temperature range of 25-800 °C, with a heating rate of 10 °C min<sup>-1</sup>. The air flow used in the TG experiment was 50 mL min<sup>-1</sup>.

The combustion properties of the briquettes were determined using a conventional cylindrical steel furnace (diameter = 0.57 m, height = 0.85 m) without atmospheric control. The exchange of air between the internal and external parts of the furnace took place through a window built in the base of the oven (0.20 m long, 0.10 m high). A screen was placed 0.25 m above the base to serve as a plate during burning. A thermocouple was installed 0.15 m above the screen. For the first test, 11.33 kg of the 37.5 MPa briquettes was fired. After ignition, the temperature of the sample was monitored for almost 8 hours using thermocouples placed next to the sample. A similar experiment was conducted with samples obtained at 0.38 MPa with the burning of 11.33 kg of briquettes. During the tests, the airflow was not varied. The curves obtained with different compaction pressure were fitted by the non-linear model, using a logistic function.

## **RESULTS AND DISCUSSION**

After pressing, it was possible to verify the apparent density  $(D_{ad})$  of the samples produced on a wet basis and after the drying process. The pressure applied during the pressing and the results obtained for the apparent density are presented in Table 1. In general, the densities obtained on wet basis were higher compared to those obtained after drying. According to the available experimental conditions, the maximum applied pressure (37.50 MPa) resulted in a density of around 2,800 kg m<sup>-3</sup> on a wet basis, which after drying reduced the apparent density by approximately 47%. The values after drying did not statistically differ from those in other studies that have reported densities of 1,320 kg m<sup>-3</sup> for wood briquettes. Araújo et al. (2016) showed that when briquettes have a higher bulk density, they also have a higher energy density. The results from the current study can be compared with briquettes of Wang et al. (2019) biochar, where

 Table 1. Summary of the briquettes applied pressures, apparent densities and mass losses

Applied pressure	D <sub>ad</sub>	D <sub>ad</sub>	ΔW
(MPa)	(kg m⁻³) <sup>i</sup>	(kg m⁻³) <sup>ii</sup>	(%)
0.00	322 (±19)	148 (±9)	-
0.38	-	197 (±12)	-
3.41	1,073 (±64)	530 (±33)	31.9
5.10	1,264 (±71)	636 (±43)	21.9
12.31	1,605 (±97)	801 (±39)	12.4
37.50	2,869 (±180)	1,513 (±91)	7.6

<sup>i</sup>Density of the briquettes with 50% humidity and the standard deviation <sup>ii</sup>Density of the dry briquettes and the standard deviation a binder was used to form the briquettes, which showed results above 1,006 kg m<sup>-3</sup>. Similar values were also found by Teixeira et al. (2010), whose study on sugarcane bagasse briquettes determined a minimum density of 910 kg m<sup>-3</sup> and a maximum density of 1,330 kg m<sup>-3</sup>.

The briquettes produced with higher pressure showed less loss of mass (Table 1). This behavior represents greater durability after the briquetting and drying processes, indicating greater stability and consequently greater durability during handling, transportation, and storage.

Figure 2A presents the percentages of the analyzed chemical elements. The value obtained for the loss on ignition (LOI) was the percentage of organic material present in the sample (74.4%), the most abundant material. The second most abundant (16.5%) material was CaO. The majority of the crystalline phase was calcium carbonate (CaCO<sub>2</sub>), as shown by the diffractogram presented in Figure 2B. The crystallographic planes (h k l) investigated in the  $2\theta$  interval were indexed according to the CaCO<sub>3</sub> phase with rhombohedral structure and space group R-3c. Data of Chessin et al. (1965) were used to index the crystallographic planes. However, no crystalline cellulose was observed, although the presence of crystalline cellulose has been reported for species of eucalyptus wood (Poletto et al., 2012). The diffraction pattern also showed a wide peak in the  $2\theta$  angular range between 15° and 28°, a typical characteristic of an amorphous material. This is indicative of the presence of some material, such as hemicellulose or lignin, remaining in the eucalyptus wood, as has been suggested by other studies (Zheng et al., 2016).

Figure 3 shows the percentage of weight loss by thermogravimetry (TG) and the derivative thermogravimetric



**Figure 2.** (A) Chemical composition by X-ray fluorescence and (B) the X-ray diffraction patterns of the eucalyptus wood waste



**Figure 3.** Weight loss and the derivative of weight loss of eucalyptus wood waste measured during heating in air

curves (DTG), as a function of temperature of eucalyptus wood waste. During the heating of the sample, the TG/DTG curves (Figure 3) showed three significant stages of mass loss. The first, which occurred between 25 and 100 °C, was due to water volatilization (7.7%). This was followed by two-step processes that occurred in the ranges of 100-500 °C, which presented a mass loss of 60.3%, and 500-670 °C, with a mass loss of 9.5%, respectively. The analysis of the profile in Figure 3 between the temperatures of 300 and 500 °C provided evidence of the two processes, with a peak at 410 °C and a shoulder around 450 °C. The decomposition of hemicellulose is reported to occur around 300 °C, although studies on eucalyptus wood submitted to hydrothermal treatment have shown an increase in this temperature (Zheng et al., 2016). The main degradation of cellulose occurs around 350 °C, and a peak appears at the temperature corresponding to the maximum decomposition rate. In other words, due to the presence of calcite, revealed by XRD at room temperature, different thermal processes can occur because they are dependent on a sample's intrinsic (i.e. purity, defect content, surface area, crystallite size, morphology, etc.) and extrinsic factors (i.e. processes conditions, atmospheric conditions, etc.) (Galan et al., 2013). Therefore, regarding the thermal behavior of calcite, it is expected that Ca(OH), decomposes between 350 and 400 °C. Finally, the third process, which occurred above 600 °C, was due to the decomposition of CaCO<sub>3</sub>. Thus, due to the nature of the pyrolysis process, it was evident that the decomposition kinetics was changed. Therefore, the profile between 300 and 500 °C was attributed to the presence of multiple decompositions, with different contributions from the organic and calcite portions coexisting in the residue.

The burning behavior of the briquettes was propagationdependent on the briquette fabrication pressure. As shown in Figure 4A, temperature was measured in the briquettes as a function of time for the different briquette fabrication pressures. The combustion characteristics exhibited two distinctive stages, as indicated by the dashed line separating regions 1 and 2 in Figure 4A, which represent the ignition propagation stage and the char oxidation stage, respectively (Ryu et al., 2007; Kim et al., 2016). For both samples, a very rapid increase was observed during the first 120 min of the ignition propagation stage. However, over time, the temperature measured for the 37.50 MPa briquette reached a maximum of around 1,030 °C. The onset of region 2 was characterized by a decrease in temperature. During stage 2, the volatilization and gasification of the particles at the end of the process gave rise to char. The decrease in temperature was slower for the 37.50 MPa briquette. The 0.38 MPa briquette passed a maximum temperature of 808 °C within 110 min while still in the ignition region. During combustion, some gas exchange processes occur, and the heating caused by burning a fuel is limited by the amount of oxygen available in the furnace. Previous studies on wood waste have indicated the consumption of O<sub>2</sub> with the release of CO and CO<sub>2</sub> during different stages (Ryu et al., 2007). After the completion of stage 1, it was expected that gasification of the activated carbon would occur in this region. As a result, the CO level increased until it reached maximum values, as shown in other studies. A dependence on the maximum temperature with varied airflow during firing has also been reported, but the same dependence related to the amount of briquette compaction has not been reported.

The ignition rate was used to explain the different behaviors discussed above. For this purpose, the fittings were conducted as shown in the upper part of the Figure 4B. Using this fitting, it was possible to extract the ignition rate with respect to time of burning, as shown in the lower part of the Figure 4B. The drop in the ignition rates differed. Comparing the curves obtained for the different fabrication pressures, the rate dropped faster with time for the 0.38 MPa briquette, while the rate for the 37.5 MPa briquette dropped more slowly. In both cases, dependence on the fabrication compaction pressure was evident.

The greater the availability of oxygen, the higher the burning rate expected. Thus, it was relevant to note that the availability of free spaces in the samples could have influenced the burning rate of the briquettes: the more compact the material, the slower the internal burning. In this field of research, other variables also apply, such as the dependence of burning rate on particle size. Further experiments are in progress to clarify the nature of the particle sizes in the briquettes studied in the current study.



**Figure 4.** (A) Variation of temperature of briquettes as a function of time of burning, (B) fitted curves for the ignition propagation stage (upper part) (region 1), and ignition rate curves (lower part) in region 1 for briquettes obtained in two pressure applied

#### **CONCLUSIONS**

1. It is possible to reuse eucalyptus wood residue in the form of briquettes as an alternative energy source.

2. The main element present in the decomposition of the residue is  $CaCO_3$ .

3. Burning occurs for a prolonged period, and the ignition rates depend on the fabrication pressure of the briquette related with the presence of pores.

4. The production of briquettes with 50% humidity under an applied pressure of 37.50 MPa generated briquettes with the greatest density and temperature stability.

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