

Communication

Influence of Pyrolysis Temperature on Critical Variables Related to Charcoal Spontaneous Combustion

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Abstract

Spontaneous combustion of charcoal is still not fully understood, generating uncertainties among producers, regulatory agencies, and the scientific community. This study evaluated the influence of final pyrolysis temperature (350, 450, 550, and 650 °C) on the properties of *Eucalyptus* spp. charcoal and its relation to ignition behavior. Gravimetric yield, proximate composition, calorific value, and ignition temperature were determined. Charcoal yield decreased by 31% between 350 °C and 650 °C. Fixed carbon content increased from ~65% to ~93%, accompanied by a reduction in volatile matter (~35% to ~6%) and a corresponding rise in calorific value. Step-heating experiments, conducted in a furnace with infrared camera monitoring, showed that ignition temperature increased from ~273 °C in charcoal produced at 350 °C to ~424 °C in charcoal produced at 650 °C. Strong correlations indicated that higher fixed carbon and lower volatile matter contents are directly associated with higher ignition temperatures. These results demonstrate that increasing the final pyrolysis temperature improves both the thermal stability and the energy quality of charcoal, although at the expense of gravimetric yield. Since the methodology was based on forced heating rather than spontaneous combustion under near-ambient conditions, complementary tests are required to evaluate spontaneous combustion propensity. Overall, the findings provide practical insights to balance yield, quality, and safety while reinforcing the importance of standardized assessment protocols to ensure safer storage and transport of charcoal.

Keywords: ignition temperature; reactivity; thermal stability; charcoal



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1. Introduction

Charcoal is a solid biofuel widely used in industrial, agricultural, and domestic sectors, playing a strategic role in the metallurgical and energy chains, particularly in Brazil. Its classification as a material subject to spontaneous combustion, under UN number 1361 of Risk Class 4.2, imposes significant logistical and commercial restrictions [1]. In Brazil, Resolution No. 5998/2022 of the National Land Transport Agency (ANTT) establishes strict requirements for road transportation of charcoal, including specific signage, mandatory

safety equipment, and labeling [1]. These measures increase operational costs, hinder interstate circulation, and limit access to international markets.

This classification is based on the assumption that charcoal presents a significant risk of spontaneous combustion, especially during transport and storage [2,3]. Spontaneous combustion is a self-accelerating physicochemical process in which the interaction between charcoal and oxygen promotes an exothermic reaction through physical adsorption, raising the surface temperature of the material [4,5]. Oxidizable functional groups release heat, leading to thermal energy accumulation [6,7]. When heat dissipation, either by ventilation or other mechanisms, is insufficient, the internal temperature may reach the ignition point, resulting in spontaneous combustion with potential for fires, explosions, and toxic gas emissions [5,8]. However, under specific production and handling conditions, charcoal may not exhibit such behavior. The absence of standardized technical criteria and consistent experimental evidence prevents the revision of this classification at the national level, keeping charcoal categorized as hazardous cargo.

Although most of the literature on spontaneous combustion focuses on mineral coal, differences in composition and reactivity may render direct extrapolation to charcoal/biochar inadequate. Mineral coal often presents significant levels of organic and inorganic sulfur, as well as inert minerals, which favor early oxidation reactions, influence activation energy, and contribute to heat and gas generation at low temperatures, thereby increasing its oxidative reactivity [9–11]. In contrast, charcoal (or biochar) exhibits behavior conditioned by the feedstock and pyrolysis conditions, particularly with regard to pore structure, surface functional groups, and residual volatile matter [12,13]. The spontaneous combustion of biochar is strongly related to the presence of surface oxygenated groups and oxygen chemisorption, processes that, combined with the structural modifications induced by pyrolysis temperature, determine its reactivity and thermal stability [12]. Thus, understanding the specific mechanisms of charcoal is essential to avoid generalizations derived from mineral coal.

Pyrolysis, a thermochemical process of biomass decomposition in the absence of oxygen, is the predominant method for producing charcoal [14]. The operational conditions of this process, particularly the final temperature, decisively influence the physicochemical properties of the material, including density, volatile matter, ash content, fixed carbon, thermal stability, and reactivity [5,15,16]. These characteristics may affect the potential for spontaneous combustion, although this relationship still lacks systematic scientific validation. The technical gap identified in this field lies in the lack of data relating the final pyrolysis temperature to the spontaneous combustion variables of charcoal. The main methodological innovation consists of the use of an infrared thermographic camera (Testo 868), which enables non-invasive and continuous monitoring of the thermal evolution of the samples, overcoming the limitations of conventional techniques based on thermocouples, which may interfere with sample oxidation. Furthermore, this study establishes a direct link between processing conditions (pyrolysis) and regulatory safety considerations, providing evidence that may support the revision of the current classification of charcoal as a hazardous cargo (Risk Class 4.2).

This study presents initial experimental data aimed at clarifying the influence of final pyrolysis temperature on charcoal susceptibility to spontaneous combustion. The findings are expected to support a critical assessment of current legislation, contributing to the revision of regulations that, in the absence of consolidated scientific evidence, may impose disproportionate restrictions on the production sector.

2. Materials and Methods

2.1. Production and Characterization of Charcoal

To investigate the influence of the final pyrolysis temperature on charcoal susceptibility to spontaneous combustion, samples were produced from seven-year-old *Eucalyptus* spp. wood. The logs were sectioned into wedges, weighed on a precision balance, and subjected to pyrolysis in a muffle-type furnace under controlled conditions. The process was carried out in a low-oxygen atmosphere, without gas entry, at four distinct final temperatures: 350 °C, 450 °C, 550 °C, and 650 °C (covering the temperature ranges commonly used in Brazilian charcoal industries), with a heating rate of 6 °C·min^{−1} and a residence time of 120 min. During pyrolysis, the non-condensable gases were directed to a condenser for collection of the pyroligneous liquid. At the end of the process, with the reactor still sealed, the charcoal remained inside the reactor until naturally cooled to ambient temperature (approximately 30 °C). The gravimetric yields of solid, liquid, and gaseous products were quantified (Equations (1) and (2)). The non-condensable gases were estimated by subtracting the liquid and solid yields from the total yield.

$$CY = \left(\frac{Mc}{Mw} \right) \times 100 \quad (1)$$

$$PY = \left(\frac{Mpl}{Mw} \right) \times 100 \quad (2)$$

where: Y = charcoal yield (%); Mc = mass of charcoal (g); Mw = mass of dried wood (g); PY = pyroligneous liquid yield (%); and Mpl = mass of pyroligneous liquid (g).

For subsequent analyses, the charcoal samples were previously dried, ground, and sieved to obtain particles with a granulometry between 250 µm and 420 µm (corresponding to 60 and 40 mesh, respectively). To characterize the charcoal produced at different temperatures, proximate analyses were performed (moisture, volatile matter, ash, and fixed carbon) according to the standard method [17]. The higher heating value (HHV) was determined using an IKA C200 bomb calorimeter, following the applicable European standard [18]. The lower heating value (LHV) was estimated based on the theoretical hydrogen content of the material, assumed to be 6% [19]. The lower and useful heating values were determined using Equation (3).

$$NHV = \left(LHV \times \left(\frac{100 - U}{100} \right) - 6 \times U \right) \times 4.1868 \times 10^{-3} \quad (3)$$

where: NHV = Net heating value (MJ.kg^{−1}); LHV = Lower heating value (kcal.kg^{−1}), obtained by subtracting $HHV - 304$ (formation energy of water vapor, considering a 6% hydro-gen content in the biomass); and U = moisture (%); 4.1868×10^{-3} = conversion factor from kcal.kg^{−1} to MJ.kg^{−1} [20].

2.2. Spontaneous Combustion Analysis of Charcoal

For the evaluation of spontaneous combustion, five grams of each sample was carefully weighed and placed in a porcelain crucible, which was inserted into a muffle-type furnace with air inlet. The material was initially heated to 100 °C and maintained at this temperature for three minutes, a procedure adopted to ensure thermal stabilization of the sample and to eliminate possible initial thermal variations that could interfere with the test results.

The ignition test consisted of exposing the samples to heating cycles of progressively increasing durations, while the thermal behavior was continuously monitored using a thermal imaging camera (Testo 868). This combination of equipment allowed precise monitoring of the surface and internal temperatures of the samples, which was essential

for identifying exothermic reactions and potential signs of spontaneous combustion. Each test cycle comprised a heating period followed by a five-minute holding time, considered sufficient to detect possible self-sustaining thermal reactions.

The initial exposure time to heat was set at 20 s and progressively increased after every three repetitions, depending on the thermal resistance observed in the samples, allowing a detailed assessment of the material's susceptibility to ignition. Temperatures were recorded after thermal stabilization of each cycle and at the end of the holding period, enabling the analysis of thermal evolution and the determination of ignition temperature. The ignition temperature was defined as the point at which an abrupt increase greater than 10% relative to the stabilized temperature was observed, characterizing the onset of spontaneous combustion (Figure 1).

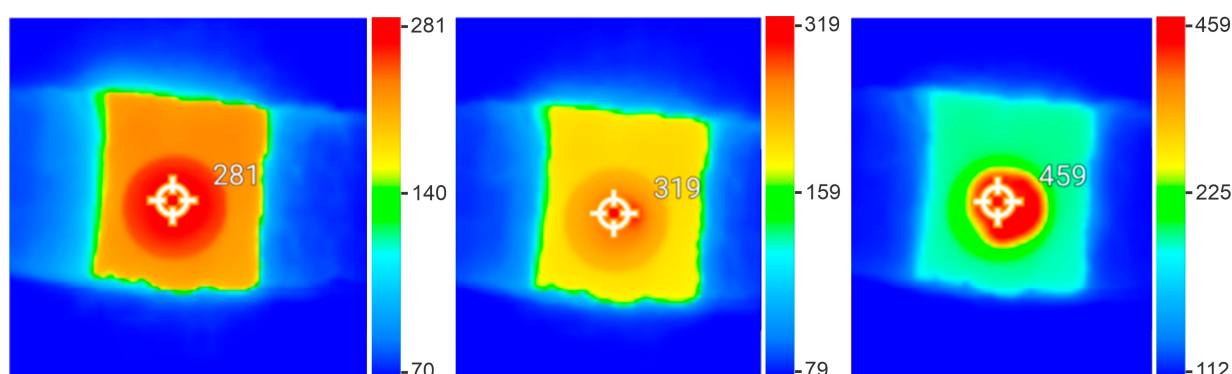


Figure 1. Infrared images indicating the ignition temperature (°C) of charcoal obtained by the thermal imaging camera.

2.3. Data Analysis

The data were subjected to normality testing (Shapiro–Wilk) and homoscedasticity testing (Bartlett). Subsequently, Pearson's correlation analysis was performed. Analysis of variance (ANOVA) was carried out following a completely randomized design, with four response variables related to pyrolysis temperature (350, 450, 550, and 650 °C), each with three replications, totaling 12 samples. When significant differences were detected, regression models were fitted to best predict the behavior of ignition temperature. All analyses were conducted with a 95% significance level. Statistical analyses were performed using R (R Core Team) software, version 4.5.1, released in 2025. All analyses were conducted at a 95% probability level.

3. Results and Discussion

3.1. Pyrolysis Yield and Properties of Charcoal

Table A1 presents the averages of the studied variables, including the pyrolysis yields, which varied significantly depending on the final carbonization temperature, as illustrated in Figure 2. The average yields of char, pyroligneous liquid and non-condensable gases were, respectively, $36.26 \pm 0.15\%$, $44.24 \pm 1.16\%$ and $19.50 \pm 1.30\%$ at 350 °C; $30.2 \pm 0.90\%$, $45.86 \pm 0.78\%$ and $24.12 \pm 1.67\%$ at 450 °C; $27.20 \pm 0.19\%$, $46.79 \pm 0.82\%$ and $26.01 \pm 0.69\%$ at 550 °C; and $24.85 \pm 0.13\%$, $45.52 \pm 1.27\%$, and $29.63 \pm 1.15\%$ at 650 °C. A reduction of approximately 31% in charcoal yield was observed between 350 °C and 650 °C, attributed to the intensified thermal degradation of wood constituents. In contrast, there was an increase in non-condensable gases, which rose from 19.50% to 29.63% over the same interval. The yield of pyroligneous liquid showed no significant variations ($F = 1.05$; $p = 0.42$), remaining stable under the evaluated conditions.

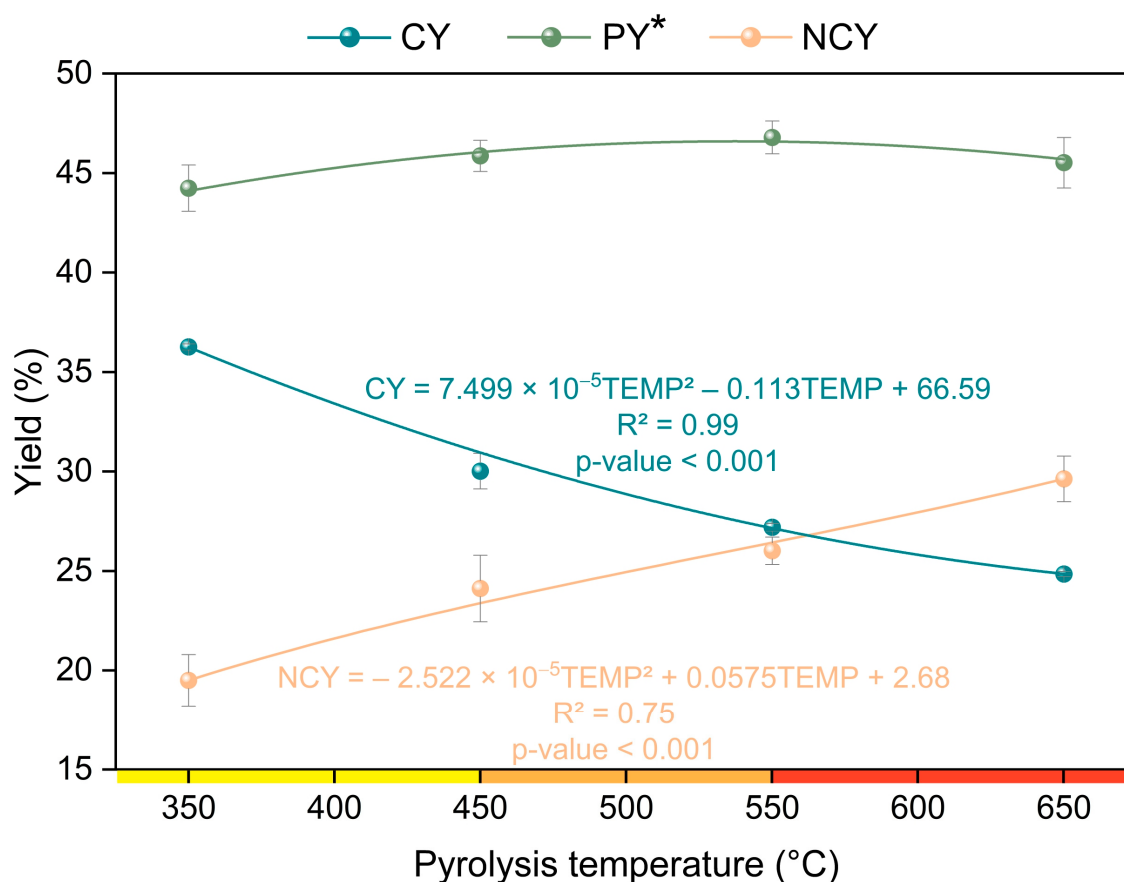


Figure 2. Yield of products and by-products from the pyrolysis of Eucalyptus wood. CY = charcoal yield; PY = pyroligneous liquid yield; NCY = non-condensable yield; * = not significantly different.

The pyrolysis temperature directly influences charcoal yield [21], which explains the reduction observed with increasing final temperature. Similar results were reported in [22] in studies with *Eucalyptus* spp. at five years of age, using final pyrolysis temperatures of 300 °C, 450 °C, and 600 °C, where a decrease in charcoal yield was observed, ranging from 34.75% to 28.03%. This behavior was also described in [23], which investigated the species *Dipteryx panamensis*, *Gmelina arborea*, *Hieronyma alchorhoides*, and *Tectona grandis*. Likewise, Ref. [24] studied rice straw, wheat straw, barley straw, and maize straw, and reported higher charcoal yields at lower temperatures.

As the temperature rises, progressive biomass decomposition occurs, with greater release of volatile compounds. Cracking reactions convert part of the condensable fraction into gases, resulting in an increase in the non-condensable fraction and a consequent decrease in charcoal yield [25]. In contrast, the yield of pyroligneous liquid remains relatively constant, since its formation occurs predominantly at intermediate temperature ranges and is less affected by variations between 350 °C and 650 °C [26]. It is important to emphasize that higher charcoal yields do not necessarily correspond to better quality, as the chemical and physical properties of the material are decisive for its different applications. Although higher yields are economically desirable, they are associated with lower temperatures [27], at which the charcoal exhibits a higher volatile matter content [28,29] and, therefore, a greater risk of spontaneous combustion. In this context, the balance between productivity and safety must be considered in industrial charcoal production.

Figure 3 shows the influence of pyrolysis temperature on the proximate and energetic properties of charcoal. Significant variations were observed in fixed carbon content, volatile matter, ash, and calorific value. In general, the average moisture content of charcoal

remained around 3%, which facilitates the evaluation of aspects directly related to the final pyrolysis temperature.

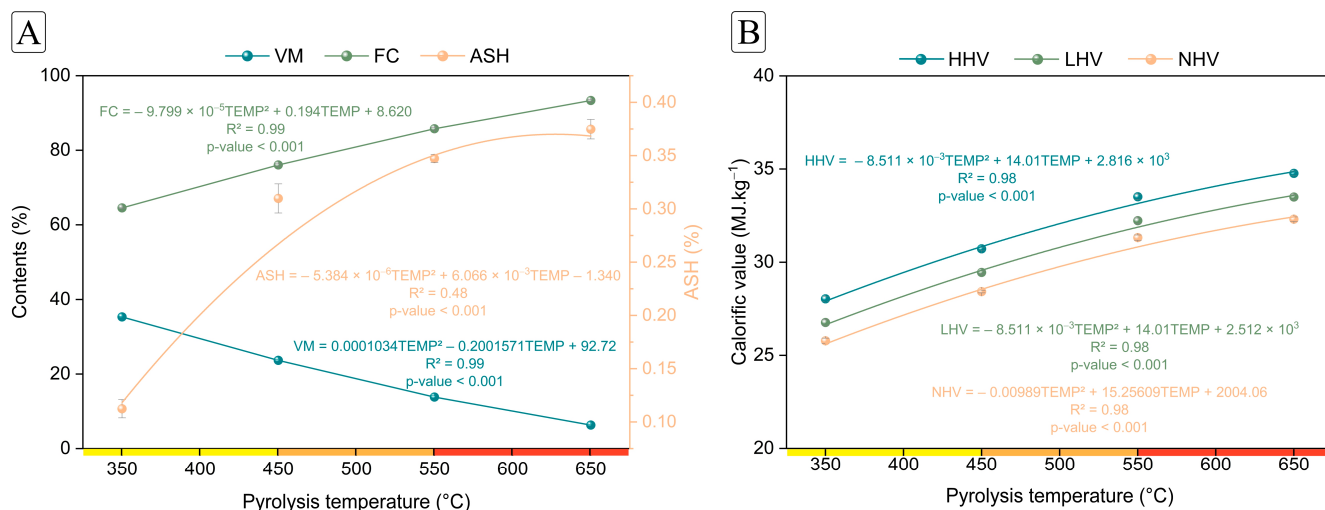


Figure 3. Proximate composition (A) and calorific values (B) of charcoal produced at different pyrolysis temperatures. VM = volatile matter; FC = fixed carbon; Ash = ash; HHV = higher heating value; LHV = lower heating value; NHV = net heating value.

The results of the proximate analysis demonstrate that pyrolysis temperature has a strong effect on the chemical composition and energy performance of charcoal. A progressive increase in fixed carbon content was observed, from 64.57% at 350 °C to 93.36% at 650 °C (Figure 3A). Conversely, volatile matter showed a marked decrease, from 35.3% to 6.32% over the same temperature range. The increase in the final pyrolysis temperature enhances the ignition characteristics of charcoal, as well as the overall combustion indices [30]. High volatile matter contents tend to facilitate the ignition of the combustion process, regardless of the type of biomass [30,31]. In contrast, the increase in fixed carbon content raises the ignition temperature of charcoal and influences its thermal stability [32]. Studies conducted by Barros et al. [3], when analyzing wood from *Dinizia excelsa* Ducke, *Parinari rodolphii* Huber, *Licania canescens* Benoist, *Couratari oblongifolia* Ducke & Kunth, among other 19 timber species, found fixed carbon values ranging from 69.61% to 73.92% and volatile material contents ranging from 21.87% to 27.58%. In addition, charcoals produced at 650 °C presented higher ash content compared with those obtained at lower temperatures. Although ash does not contribute to calorific value, it plays an important role in ignition assessment, since high values reduce energy density and interfere with combustion behavior [33].

In parallel, the higher heating value of charcoals produced at higher temperatures increased (Figure 3B). There was an approximate rise of 25% between 350 °C and 650 °C and 4% between 550 °C and 650 °C. This result is directly associated with the higher fixed carbon content, since carbon-rich charcoals exhibit greater energy density and, therefore, higher combustibility [34].

Such modifications in proximate composition and calorific value have direct implications for energy performance and, more importantly, for the risk of spontaneous combustion. Charcoals with higher volatile matter content present greater reactivity, lower thermal stability, and an enhanced tendency toward self-heating, increasing the likelihood of spontaneous combustion under inadequate storage and transportation conditions [35]. In contrast, the increase in fixed carbon and calorific value at higher temperatures provides greater resistance to self-heating. However, once combustion is initiated, it becomes more intense and prolonged, making its control more difficult in cases of accidental ignition [36].

3.2. Ignition Temperature and Its Relationship with Charcoal Properties

Figure 4 illustrates the influence of final pyrolysis temperature on the ignition temperature of charcoal. A clear and consistent trend of progressively increasing ignition temperature is observed with rising carbonization temperature. This evidences the greater thermal stability of charcoals produced at higher final pyrolysis temperatures.

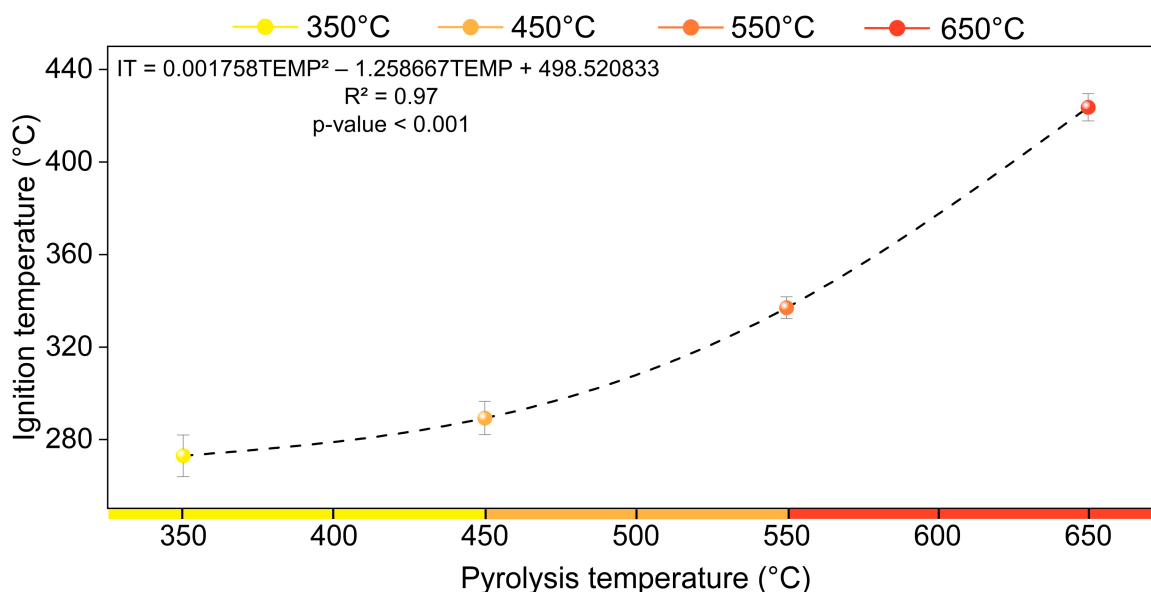


Figure 4. Ignition temperature of charcoal at different final pyrolysis temperatures.

Charcoals obtained at 350 °C showed an average ignition temperature of 273 °C. Increasing the pyrolysis temperature to 450 °C raised the ignition temperature to 289 °C, corresponding to an increment of 16 °C (Figure 4). This increase was even more pronounced at higher temperatures, with charcoal produced at 550 °C exhibiting an average ignition temperature of 337 °C and that obtained at 650 °C reaching 424 °C, representing a significant rise of 151 °C compared to the initial condition (Figure 4). Studies conducted by [37] with woody biomasses subjected to the pyrolysis process reported an ignition temperature of 371 °C for charcoal produced at 600 °C. The author also observed that the higher the volatile matter content, the lower the ignition temperature, which corroborates the findings obtained in the present study. This behavior is associated with the fact that high levels of volatiles can promote faster combustion and hinder the increase in temperature [38]. Moreover, this indicates greater thermal stability conferred by the increase in pyrolysis temperature. Such stability reflects profound structural and chemical modifications in the carbonized material, including the reduction of volatile compounds, increased aromaticity of the carbon matrix, and reduces the presence of oxygenated groups [31,39,40]. As a result, a more stable and less reactive material is obtained, with greater resistance to initial oxidation, thus justifying the need for higher temperatures to initiate ignition.

To elucidate the interrelationships between ignition temperature and other physico-chemical properties of charcoal, Figure 5 presents the statistical correlation matrix among the analyzed parameters.

The correlation matrix presented in Figure 5 highlights statistically significant relationships among the physicochemical and energetic variables of charcoal. A strong negative correlation between final pyrolysis temperature and volatile matter content ($r = -0.99$) stands out, indicating a reduction in volatile compounds as pyrolysis temperature increases (Figure 5). Conversely, fixed carbon content and higher heating value show strong positive

correlations with temperature ($r = 1.0$). This reflects the improvement in charcoal’s energy quality with rising pyrolysis temperature (Figure 5).

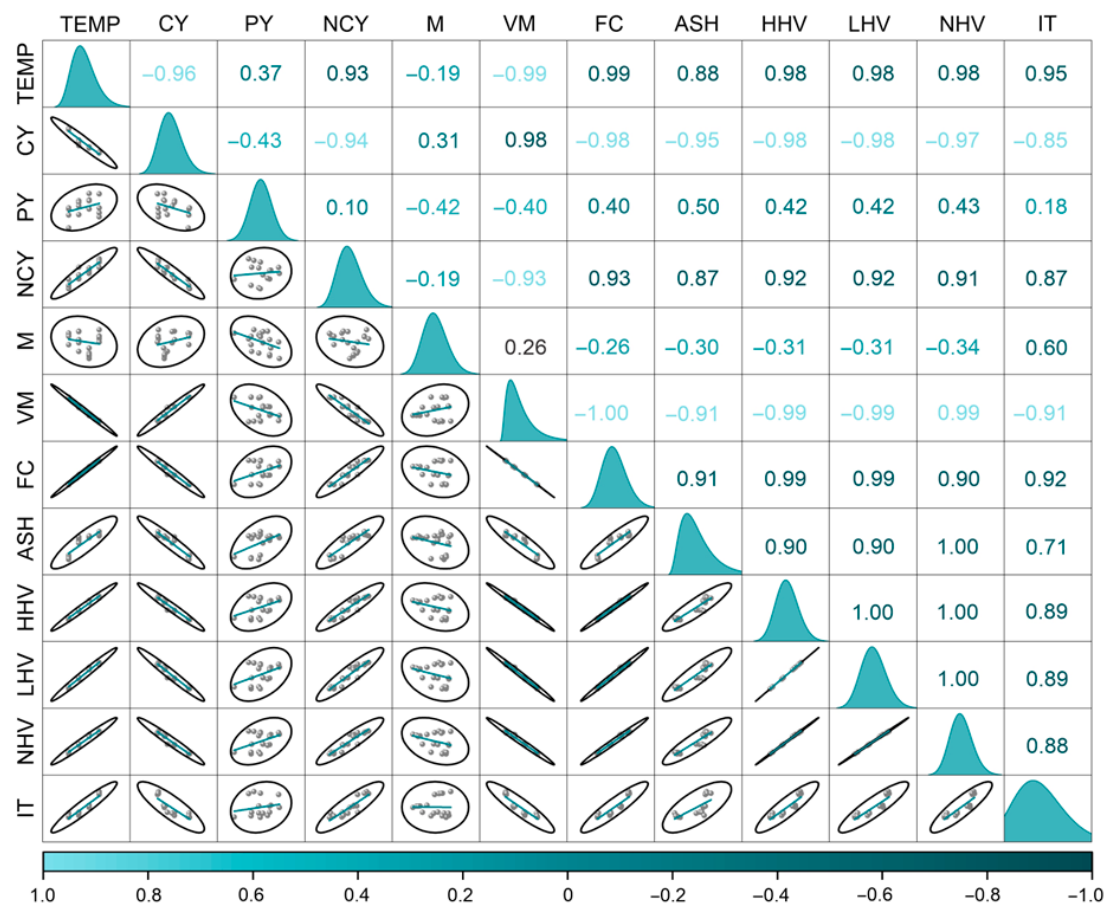


Figure 5. Ignition temperature and related parameters of charcoal produced at different pyrolysis temperatures. TEMP = pyrolysis temperature; CY = charcoal yield; PY = pyroligneous liquid yield; NCY = non-condensable gas yield; M = moisture; VM = volatile matter; FC = fixed carbon; ASH = ash; HHV = higher heating value; LHV = lower heating value; NHV = net heating value, IT = Ignition temperature.

Additionally, energetic properties exhibit high intercorrelation and strong association with fixed carbon content, emphasizing the influence of chemical composition on the material’s energy performance. Ignition temperature also correlates positively and significantly with both pyrolysis temperature and fixed carbon content, consistent with the greater thermal stability observed in charcoals produced at elevated temperatures (Figure 5).

These results indicate that charcoals produced at higher pyrolysis temperatures tend to have lower volatile matter content and greater thermal stability [4], which may contribute to reducing the risk of spontaneous combustion during storage and transport. This characteristic is particularly relevant for the operational safety and logistics of the charcoal production chain.

From a practical perspective, optimized control of pyrolysis temperature is essential not only to maximize charcoal’s energy efficiency but also to minimize risks associated with its flammability. Future research could explore the relationship between pyrolysis parameters and thermal properties under real storage conditions. This would support the develop of protocols that ensure greater safety and performance of charcoal for industrial applications. Correlations may reflect collinearity among the variables, as pyrolysis temperature directly influences both fixed carbon and volatile matter.

3.3. Relevance of the Data and Future Studies

In Brazil, most charcoal is destined for the steel industry, as a substitute for mineral coal in pig iron reduction [41]. However, the high demand often compromises the production process, mainly due to inadequate cooling of the material, which, when exposed to oxygen, may reignite and cause significant losses. According to the ANTT Resolution, charcoal is classified as a material subject to spontaneous combustion (Class 4.2), requiring specific labeling during transportation. Nevertheless, the legislation provides exemption upon presentation of a technical report, conducted according to the United Nations, protocol (2009) [42], which subjects samples to forced heating at 140 °C for 24 h to assess their thermal stability. Despite this regulation, studies have questioned the actual susceptibility of charcoal to spontaneous combustion, indicating that the main determining factor is the ignition temperature [43].

In this context, the results obtained in this study aim to provide information on the ignition temperature of charcoals produced within the temperature ranges commonly employed in the Brazilian industrial process. These data may contribute to a better understanding of ignition intervals in forced heating tests and, consequently, to the exemption of transportation of charcoals produced under such conditions. Although there is an official testing manual for issuing exemption reports, the use of alternative protocols, such as infrared chamber ignition, may be considered.

Nevertheless, further experiments focused on assessing the ignition temperature and spontaneous combustion of charcoal are recommended. Such studies should address different temperature ranges, as well as complementary analyses. These may include isothermal self-heating, adiabatic calorimetry, crossing-point test, thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), small bed, and Raman or Fourier-transform infrared spectroscopy (FTIR), which can provide more consistent information on the influence of charcoal structure on its ignition temperature. Furthermore, it is essential to consider environmental factors that may modify these conditions, such as moisture, air mass, biomass type, and density. Finally, future research should include experiments under realistic conditions, such as warehouse holds and truck bodies, accounting for oxygen gradients and long storage periods, in order to reproduce the practical conditions of charcoal storage and transportation.

4. Conclusions

The ignition of charcoal is directly related to the pyrolysis temperature employed during its production. The properties acquired throughout the process, such as the increase in fixed carbon content and the reduction in volatile matter fraction, significantly influence the thermal behavior and ignition response of the material. The results obtained from forced heating tests show that charcoals produced at higher pyrolysis temperatures exhibit greater apparent thermal stability and require relatively high temperatures to ignite. These findings reinforce the role of pyrolysis temperature as a determining factor in the characteristics of charcoal and indicate that, under controlled conditions, the material is less susceptible to spontaneous combustion. Nevertheless, it is important to emphasize that the applied tests do not fully reproduce large-scale storage conditions. In real scenarios, factors such as thermal insulation, oxygen gradients, and long storage periods may act differently. In this regard, complementary studies, such as isothermal self-heating, bulk heat accumulation, crossing-point tests, and TGA/DSC, are recommended to broaden the understanding of charcoal behavior under real scenarios of spontaneous combustion and to support possible revisions of its risk classification.

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Data Availability Statement: Data is available upon request.

Conflicts of Interest: The authors declare no conflicts of interest.

Appendix A

Table A1. Average physical, chemical and energetic properties of charcoal produced at different pyrolysis temperatures.

Properties	Temperature (°C)			
	350	450	550	650
Charcoal yield (%)	36.26 (±0.15)	30.02 (±0.90)	27.19 (±0.19)	24.85 (±0.13)
PLY (%)	44.24 (±1.16)	45.86 (±0.78)	46.79 (±0.82)	45.52 (±1.27)
NCY (%)	19.50 (±1.30)	24.12 (±1.67)	26.01 (±0.69)	29.63 (±1.15)
Volatile matter (%)	35.30 (±0.16)	23.66 (±0.30)	13.83 (±0.37)	6.32 (±0.22)
Fixed carbon (%)	64.57 (±0.17)	76.07 (±0.30)	85.78 (±0.35)	93.36 (±0.25)
Ash (%)	0.11 (±0.1)	0.31 (±0.1)	0.35 (±0.0)	0.37 (±0.0)
HHV (MJ.kg ^{−1})	28.03 (±0.14)	30.72 (±0.08)	33.51 (±0.14)	34.77 (±0.12)
LHV (MJ.kg ^{−1})	26.76 (±0.14)	29.45 (±0.08)	32.23 (±0.14)	33.50 (±0.12)
NHV (MJ.kg ^{−1})	25.78 (±0.14)	28.42 (±0.07)	31.32 (±0.14)	32.31 (±0.12)
IT (°C)	273.00 (±9.02)	289.33 (±7.17)	337 (±4.73)	423.67 (±5.93)

PLY = Pyrolygneous liquid yield; NCY = Non-condensable yield, HHV = Higher heating value, LHV = Lower heating value, NHV = Net heating value, IT = Ignition temperature, () = standard error.

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