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Environmental Technology

Investigating the use of hybrid clones of Corymbia torelliana and Corymbia citriodora for the production of renewable bioreducers

Investigando o uso de clones híbridos de *Corymbia torelliana* e *Corymbia citriodora* para a produção de biorredutores renováveis

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ABSTRACT

Charcoal is of paramount importance in the steel industry, where it is used as a reductant and heat source in the combustion of iron ore and the production of pig iron. Research has been conducted to identify wood species that produce charcoal with superior physical, mechanical, and chemical characteristics compared to those currently used in the industry. This study aimed to characterize hybrid clones of Corymbia torelliana, Corymbia citriodora, and a clone of Eucalyptus urophylla at two final pyrolysis temperatures, 350°C and 550°C, to determine which is most suitable for use in the steel industry. Two hybrid clones of Corymbia torelliana and Corymbia citriodora and one clone of Eucalyptus urophylla were analyzed, with the latter serving as a comparison standard due to its prevalent use in charcoal production. The clones, seven years old and planted in Bom Despacho, Minas Gerais, Brazil, were subjected to laboratory pyrolysis at final temperatures of 350°C and 550°C, with a heating rate of 5°C.min⁻¹. The lignin and extractive contents of the species were measured, along with the gravimetric yields of the charcoal produced. The clones were characterized for apparent density, chemical analysis, higher, lower, and useful calorific values, energy density, and scanning electron microscopy (SEM) images. The Corymbia citriodora clone, at a pyrolysis temperature of 350°C, produced the best charcoal for steelmaking, evidenced by its high fixed carbon content (65.24%), energy density (1.26 Gcal.m⁻³), and a visually observed reduction in cracks and fissures. The hybrid clone of Corymbia citriodora presents a promising alternative, offering rapid growth and qualities that are comparable or superior to species currently used in the industry.

Keywords: Steel bioreducer; Renewable fuels; Sustainable steel industry; Charcoal quality



RESUMO

O carvão vegetal é de suma importância na indústria siderúrgica, onde é utilizado como redutor e fonte de calor na combustão do minério de ferro e na produção de ferro-gusa. Pesquisas têm sido realizadas para identificar espécies de madeira que produzam carvão vegetal com características físicas, mecânicas e químicas superiores às atualmente utilizadas na indústria. Este estudo teve como objetivo caracterizar clones híbridos de Corymbia torelliana, Corymbia citriodora e um clone de Eucalyptus urophylla em duas temperaturas finais de pirólise, 350°C e 550°C, para determinar qual é o mais adequado para uso na indústria siderúrgica. Dois clones híbridos de Corymbia torelliana e Corymbia citriodora e um clone de Eucalyptus urophylla foram analisados, sendo este último utilizado como padrão de comparação devido ao seu uso predominante na produção de carvão vegetal. Os clones, com sete anos de idade e plantados em Bom Despacho, Minas Gerais, Brasil, foram submetidos à pirólise em laboratório em temperaturas finais de 350 °C e 550 °C, com uma taxa de aquecimento de 5°C.min⁻¹. Foram medidos os teores de lignina e extrativos das espécies, juntamente com os rendimentos gravimétricos do carvão produzido. Os clones foram caracterizados quanto à densidade aparente, análise química, valores caloríficos superior, inferior e útil, densidade energética e imagens de microscopia eletrônica de varredura (MEV). O clone de Corymbia citriodora, a uma temperatura de pirólise de 350 °C, produziu o melhor carvão vegetal para a siderurgia, evidenciado por seu alto teor de carbono fixo (65,24%), densidade energética (1,26 Gcal.m⁻³) e uma redução visualmente observada nas rachaduras e fissuras. O clone híbrido de Corymbia citriodora apresenta uma alternativa promissora, oferecendo crescimento rápido e qualidades comparáveis ou superiores às espécies atualmente utilizadas na indústria.

Palavras-chave: Bioredutor siderúrgico; Combustíveis renováveis; Indústria siderúrgica sustentável; Qualidade do carvão vegetal

1 INTRODUCTION

In 2022, Brazil solidified its position as the world's largest producer of charcoal, reaching a production of 7.0 million tons, predominantly destined for the domestic market (IBÁ, 2023). Of this total, 6.9 million tons were derived from wood sourced from planted forests, which reflects a 15% growth over the past five years. According to data from the Iron Industry Union of the State of Minas Gerais (SINDIFER), in 2021, the steel and iron sector used charcoal to produce 7.8 million tons of pig iron, accounting for 24.1% of the total national pig iron production, which amounted to 32.4 million tons in 2022 (SINDIFER, 2022). Approximately 10% of Brazilian steel is produced using charcoal. This method offers environmental and competitive benefits. It results in high-quality pig iron and contributes to a lower intensity of CO₂ emissions in the Brazilian steel industry, since forests absorb CO₂ during photosynthesis, offsetting greenhouse gas emissions in the industrial process (IAB, 2024).

Even though Brazil is the largest producer of charcoal in the world, there are barriers that must be overcome in the charcoal production process, either through the homogenization of wood or control of oven temperatures (Costa et al., 2024; Pereira et al., 2023). As a result, insufficient knowledge limits producers. Due to the expressiveness in the production and consumption of charcoal in our country, there are needs for new technological demands, which are concentrated in the context of the origin and quality of the raw material used and in the productive control (Costa et al., 2024). Therefore, it is necessary to characterize charcoal, with the aim of improving the production process. Hybrid clones of Corymbia have been presented as a viable alternative for the production of steel charcoal. However, despite the information acquired so far, there is a need for more in-depth studies on the characteristics and quality of charcoal from these clones. Obtaining operational gains, both in energy and economic efficiency, requires knowing how to correctly use these clones in the production chain, as well as understanding all their characteristics. After all, with different characteristics, which are the most crucial for the steel sector? The quality of charcoal is one of the main parameters for acceptance in the market. Several factors influence the quality of charcoal. For example, apparent density, energy density, reactivity, friability, content of volatile materials, ashes, fixed carbon, and microstructure from scanning electron microscopy analysis are important parameters to be analyzed (Abhi et al., 2023; De Meira et al., 2021; Silveira et al., 2020).

Species of the genus *Corymbia* exhibit high growth rates and wood density, comparable to that of *Eucalyptus spp.* (Loureiro *et al.*, 2021). Massuque *et al.* (2023) emphasize that the use of hybrids is an efficient alternative, as they are tolerant to most pests and diseases and have a lower cost in silvicultural treatments, showing satisfactory characteristics for the production of charcoal. Studies have highlighted the satisfactory yield and high density of charcoal when produced with *Corymbia* wood (Loureiro *et al.*, 2021). The high density of its wood, which ranges between 0.550 to 0.650 g.cm⁻³, provides high density and mechanical strength to the charcoal (Loureiro *et al.*, 2021; Massuque *et al.*, 2023). Considering this approach, knowing and quantifying

the gains in charcoal quality from new species, such as *Corymbia* hybrids, with characteristics and wood quality different from *Eucalyptus* species, is a gap that must be investigated for several charcoal applications, supporting greater efficiency in the pig iron production chain and, consequently, in steel. Therefore, the objective of this research is to determine the quality of charcoal produced from *Corymbia* and *Eucalyptus* clones at two final pyrolysis temperatures, aiming for high gravimetric yield and high-quality charcoal suitable for steelmaking.

2 MATERIALS AND METHODS

2.1 Study area, sampling and wood characterization

In this study, the wood and charcoal quality of three clones were analyzed, two hybrid clones of *Corymbia torelliana* and *Corymbia citriodora* (C1 and C2, respectively) and one clone of *Eucalyptus urophylla* (E3) at seven years of age, from from an experimental plantation, in the municipality of Bom Despacho, eastern region of Minas Gerais, Brazil. The region had an average temperature of 24.6 °C and precipitation of 835 mm.year⁻¹ between the years 2012 and 2019. The planting area was 2.5 ha, with 0.5 ha for each clone, with spacing of 4x1.75 meters, with the same silvicultural management used for each clone. For sampling, five trees of each clone were used.

After being collected, the samples were sent to the Department of Forest and Wood Sciences at the Federal University of Espírito Santo (DCFM) in the municipality of Jerônimo Monteiro, where the characterization of the material was carried out. To conduct the experiment, specimens measuring 2 x 3 x 5 cm (radial x tangential x longitudinal) were made. For the characterization, wood samples were ground from the six positions along the bole, and composite sawdust was obtained in granulometry between 40 (0.40 mm) and 60 mesh (0.25 mm) and, according to the TAPPI standard T 257 cm-85, for carrying out chemical analyses. The structural chemical analysis for determination of extractives and lignin contents was carried out using the

methodologies proposed by the TAPPI T 264 cm-97, TAPPI T 280 pm-99 norms and Klason lignin content by the procedures determined by Gomide; Demuner (1986).

2.2 Thermal process and characteristics of charcoal

The wood samples, previously dried in an oven at 103 ± 2 °C, were subjected to pyrolysis in a metallic cylinder reactor inside a muffle furnace at the Biomass Energy Laboratory (LEB/UFES). Subsequently, pyrolysis was carried out at final temperatures of 350 and 550 °C without nitrogen flow. The amount of wood used in each repetition was approximately 260 ± 10g, pyrolyzed at heating rates of 5 °C min⁻¹, remaining ninety minutes after reaching the stipulated final temperature (fixed level). The gravimetric yields were quantified by the solid product (charcoal), liquid product (pyroligneous liquid) and gaseous (non-condensable gases (Equation 1, 2 and 3).

$$CY = \left(\frac{Cm}{Dm}\right) \times 100 \tag{1}$$

$$PLY = \left(\frac{PIM}{Dm}\right) \times 100$$
 (2)

$$PLY = \left(\frac{PIM}{Dm}\right) \times 100$$
(3)

Where: CY = charcoal yield (%); Cm = charcoal mass (g); Dm = dry wood mass (g); PLY = pyroligneous liquid yield (%); PLM = pyroligneous liquid mass (g); NCGY = noncondensable gas yield (%)

To determine the apparent density, the methodology was carried out by dividing the mass of the charcoal sample by its volume, according to Equation 4:

$$AD = \frac{m}{v} \tag{4}$$

Where: AD = apparent density (g.cm⁻³), m = mass (g), v = volume (cm³)

The apparent density density was performed according to the methodology proposed by the D5057-17 standard (ASTM, 2017). Humidity was determined using the procedures described in the D1762-84 standard (ASTM, 2021). The immediate chemical

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analysis of the charcoal (moisture, volatile materials, ash and fixed carbon) was carried out in accordance with the D1762-84 (2021) standard. The fraction called volatile materials is emitted when charcoal is heated to a temperature of 950 °C, ash is the residue made up of mineral oxides obtained after complete combustion of charcoal, in order to have this complete charcoal remains in the muffle for a period of 6 hours at a temperature of 750 °C. Subsequently, by difference calculations, the fixed carbon content is determined.

To determine the higher calorific value of the biomass, 6% of hydrogen was considered, while the useful calorific value was considered the moisture content of the material. To determine the lower calorific value (LHV) Equation 5 was used and Equation 6 is useful.

$$LHV = HHV - \left(600\frac{9H}{100}\right)$$
(5)
$$UCV = LHV x(1 - U) - 600U$$
(6)

Where: LHV = lower calorific value (kcal.kg⁻²); HHV = superior calorific value (kcal.kg⁻²), UCV = useful calorific value (kcal.kg⁻²) and U = wet base humidity (%)

The energy density was determined by calculating the useful calorific value (PCU) and apparent density, following the European Standard EN 14918 (DIN, 2010). To evaluate the charcoal microstructure, the samples were previously dried and fixed on a metal support with carbon tape. Subsequently coated with gold for microscopic analysis in the Balzers Union SCD 030 system, with a scanning electron microscope JSM-IT200 (Tokyo, Japan), operating at 30kV in the SEI mode (secondary electrons).

2.3 Data analysis

The data obtained were subjected to normality (Shapiro-Wilk) and homoscedasticity (Bartlett) tests. Analysis of variance (ANOVA) was performed following a completely randomized design (DIC). For the statistical analysis of the data, a double

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factorial was performed, the first being temperature (350 and 550 °C) and the second factor the clones (*Corymbia* and *Eucalyptus*). It evaluated the factors, as well as the interaction between them. The ANOVA F test was used for the first factor and the Tukey test for the second factor. For all tests, a 5% significance level was used. R core Team software was used for all statistical analyses. Subsequently, the species were grouped using Principal Component Analysis (ACP). The scores resulting from the ACP allow for the evaluation of the similarity between species and the identification of the most influential variables in the formation of the groups. The variables with the greatest impact on the principal components (CP) were determined based on the eigenvectors.

3 RESULTS AND DISCUSSION

Figure 1 shows the results of the evaluation of extractives and lignin contents of hybrid clones of *C. torelliana*, *C. citriodora* and clone of *E. urophylla*.

Figure 1 – Determination of the extractives and lignin content of hybrid clones of *C. torelliana*, *C. citriodora* and clone of *E. urophylla*



Means followed by the same letters within each temperature showed no significant difference by Tukey test ($p \ge 0.05$). Source: Authors (2024)

The clones exhibiting the highest levels of extractives were C1 (9.96%), C2 (5.54%), and E3 (5.12%). In terms of lignin content, clone E3 displayed the highest percentage (29.12%), followed by C2 (24.91%) and C1 (24.27%). These values are close to those reported by Oliveira *et al.* (2023), who found lignin contents of 27.5% in *Eucalyptus urophylla* and 27.9% in hybrids of *Corymbia torelliana* × *Corymbia citriodora*. For the production of charcoal, mainly in the steel industry, preference is given to wood with a higher lignin content, as the energy potential is directly related to lignin, the greater this property, the slower the fuel burns (Massuque *et al.*, 2023; Rocha, 2022; Silveira *et al.*, 2020). In Figure 2, the yields of the products and by-products of the pyrolysis of the clones under study are displayed. The gravimetric yield in charcoal, pyroligneous liquid and non-condensable gases, obtained after the pyrolysis process, showed no significant difference between the studied species and at different pyrolysis temperatures.

Figure 2 – Charcoal yields of hybrid clones of *C. torelliana, C. citriodora* and clone of *E. urophylla* at the two final temperatures of pyrolysis. Where: CY = charcoal yield (%); PY = yield of pyroligneous liquid (LP) and NCY = yield of non-condensable gases (%)



Means followed by the same letters within each temperature showed no significant difference by Tukey test ($p \ge 0.05$). Source: Authors (2024)

Note that clone C1 obtained the highest charcoal yield at the two temperatures under study, followed by C2 at a temperature of 350 °C and clones E3 and C2 at a temperature of 550 °C. Lignin directly contributes to the yield of charcoal (Dias Junior et al., 2020), however, such behavior did not occur in the present research, since the species with the highest lignin content was clone E3, which presented the lowest charcoal yield. This can be justified due to the reduction in the extractive content, and the reduction of extractives decreases the yield of charcoal (Massuque et al., 2023; Oliveira et al., 2023). According to Ramos et al. (2019), from a production industrial perspective, achieving higher charcoal yield is desirable. As the gravimetric yield of charcoal increases, the efficiency of wood utilization in carbonization ovens improves, leading to a greater overall production of the bio-reducer. However, clone C1 obtained the highest values of non-condensable gases, this is a disadvantage, as it increases the emission of gases into the atmosphere and contributes to air pollution and global warming (Protásio et al., 2015). The yields obtained for these clones were higher than those found by Loureiro et al. (2021), which ranged from 32.52% to 34.11% in charcoal for the clones of Corymbia citriodora x Corymbia torelliana and Corymbia torelliana x Corymbia citriodora.

Figure 3 addresses the apparent density of the clones under study at the two final temperatures.

Clones C1, C2 and E3 showed the highest densities, respectively. It is observed that the apparent density decreased with increasing temperature, the same occurred with Dias Junior *et al.* (2020) for the species of *Eucalyptus saligna*. When the density of wood and charcoal increases, greater mechanical instability of charcoal occurs. Some research has noted that with the increase in apparent density, the consumption of charcoal for the production of pig iron decreases, ensuring a reduction in iron ore and an advantageous economic relationship (Couto *et al.*, 2023; Da Silva *et al.*, 2024). Having a charcoal with high mechanical stability is desirable in the steel blast furnace, preventing the passage of the air current by compacting the charcoal (Couto *et al.*, 2023; Figueiredo *et al.*, 2018). Low density is not desirable, as it can reduce the amount of

fixed carbon and increases the cost of transport and storage, as it takes up more space. Figure 4 shows the levels of volatile materials, ash and fixed carbon of the clones.

Figure 3 – Charcoal densities of hybrid clones of *C. torelliana, C. citriodora* and clone of *E. urophylla* at the two final temperatures of pyrolysis



Means followed by the same letters within each temperature showed no significant difference by Tukey test ($p \ge 0.05$). Source: Authors (2024)

The moisture of the clones were 12.69, 12.83 and 13.01% for clones C1, C2 and 03, respectively. Humidity directly influences charcoal yield parameters, ignition time and heating rate. Less humid woods are preferable to obtain higher gravimetric yields (Protásio *et al.*, 2015). Clone C2 provided charcoal with a high content of fixed carbon and lower content of volatile materials, however, clone E3 had the lowest ash content at both study temperatures. Charcoal with a high fixed carbon content is advisable for the steel industry, due to its energetic power and thermal stability. The volatiles promote an increase in the permeability of the charge in the blast furnace, consequently reducing the reactivity of the charcoal. These findings suggest that, in order to produce charcoal suitable for steelmaking, it is preferable to increase the final carbonization temperature, as this will enable a biofuel with a higher calorific value. Thus, to ensure combustion in blast furnaces, it is essential to prioritize those with

higher fixed carbon contents and lower volatile material contents. With regard to the ash toer, it is desirable that the charcoal present reduced amounts, as the minerals present do not go through the combustion process, consequently reducing the calorific value of the fuel (Lima *et al.*, 2023; Massuque *et al.*, 2023). In addition, high concentrations of ash increase the corrosion of equipment used in energy conversion, especially in thermochemical systems (Massuque *et al.*, 2023; Moutinho *et al.*, 2017). The presence of high levels of ash in charcoal can result in the accumulation of impurities in the center of solidified metal pieces, causing variations in the properties of pig iron (Massuque *et al.*, 2023). Figure 5 presents the values of higher, lower, useful calorific value (Figure 5A) and energy density (Figure 5B) of the clones at different pyrolysis temperatures.

Figure 4 – Immediate chemical analysis of hybrid clones of *C. torelliana*, *C. citriodora* and clone of *E. urophylla* at the two final temperatures of pyrolysis. Where: VMC = content of volatile materials (%); AC = ash content (%) and FCC = fixed carbon content (%)



Means followed by the same letters within each temperature showed no significant difference by Tukey test ($p \ge 0.05$). Source: Authors (2024). Source: Authors (2024)

Figure 5 – Heat and energy of hybrid clones of *C. torelliana*, *C. citriodora* and clone of *E. urophylla* at the two final temperatures of pyrolysis. Where: HHV = higher calorific value (Kcal.m⁻³); LHV = lower calorific value (Kcal.m⁻³); UCV = useful calorific value (Kcal.m⁻³); ED = energy density (Gcal.m⁻³)



Means followed by the same letters within each temperature showed no significant difference by Tukey test ($p \ge 0.05$). Source: Authors (2024)

It is observed that the calorific value (higher, lower and useful) in comparison parameters between clones, the temperature of 550 °C was statistically equal between these variables. The clones that obtained the highest calorific value E3 (350 °C) and C2

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(550 °C). Charcoal with higher calorific value provides, especially for steelmaking, lower consumption of reducing input, considering the same productivity (Lima *et al.*, 2023; Massuque *et al.*, 2023; Sen; Roy, 2022). The apparent density and higher calorific value influence the energy density, since the energy density showed the same behavior as the apparent density. The clones that presented the highest energy density values were the C2 (350 °C) e C1 (550 °C), respectively. It is preferable to use charcoal with higher energy density, as it will increase the amount of energy per unit volume of fuel and increase the mechanical resistance of charcoal in blast furnaces in the steel industry (Couto *et al.*, 2023; Massuque *et al.*, 2023). In Figure 6 it can be observed in the SEM images that the charcoal of C1 clones at 350 °C (6A), 550 °C (6B), C2 clone at 350 °C (6C), 550 °C (6D) and clone E3 at 350 °C (6E) and 550 °C (6F) pyrolysis temperatures.

It is notorious that with the increase in the pyrolysis temperature between the species under study, the amount of cracks and internal cracks increased, consequently decreasing the mechanical resistance. The species that was visually more propitious was the C2 clone (Figure 6C). Note that even raising the pyrolysis temperature, it obtained interesting characteristics, with few cracks and cracks. Unlike clone E3 (Figure 6E and 6F), which of all species had the highest amounts of cracks and cracks, and when the pyrolysis temperature was raised, it worsened. This behavior is related to the apparent density of the material, carbon with low density will have less mechanical resistance and limits fixed carbon for thermal reactions, thus increasing energy consumption and decreasing productivity. These areas with cracks and internal cracks negatively compromise the mechanical strength of the charcoal and, consequently, increase the generation of fines during transport and handling (Dias Junior *et al.*, 2020). For steelmaking purposes, it is crucial that charcoal has high strength. Therefore, it is extremely important to characterize and know other species.

Figure 6 – Scanning electron microscopy, highlighting the cracks and internal cracks of hybrid clones of *C. torelliana*, *C. citriodora* and clone of *E. urophylla* at the two final temperatures of the study



Source: Authors (2024)

Figure 7 – Distribution of the treatments of the studied species subjected to different pyrolysis temperatures, with the ordination diagram of the eigenvectors corresponding to the first two principal components



Figure 7 presents diagrams that arrange the variables according to the first two principal components of the analysis. Of the six identified components, the two that accounted for the highest percentage of the total variation, representing 81% of the observed variability, were selected. Principal Component Analysis (ACP) allowed the identification of three distinct sample groups. Source: Authors (2024)

The first group, composed of samples of *Corymbia torelliana* and *Corymbia citriodora* pyrolyzed at 350 °C, showed a positive correlation with charcoal yield and volatile matter, and a negative correlation with fixed carbon content, higher heating value, and useful heating value. The second group consisted of samples of *Corymbia torelliana* and *Corymbia citriodora* pyrolyzed at 550 °C, which demonstrated a strong correlation with variables related to higher heating value, lower heating value, useful heating value, and fixed carbon content. These findings are consistent with the data presented in Figures 4 and 5, which indicate that higher temperatures during pyrolysis result in charcoal with higher fixed carbon content and greater heating value. These findings confirm previously observed trends: charcoal with a high fixed carbon content

generally exhibits lower volatile matter, higher heating value, and consequently, greater carbon storage capacity. On the other hand, increasing the final pyrolysis temperature tends to decrease the charcoal yield, highlighting a necessary balance between the quantity of charcoal produced and the energy quality of the final product. The third group, composed of Eucalyptus samples pyrolyzed at 350 °C and 550 °C, stands out for its high lignin content, which may influence the production of non-condensable gases, differentiating this species from the others. The data indicate that pyrolysis temperature is a determining factor in the final characteristics of charcoal, particularly with regard to fixed carbon content and heating value. The species Corymbia torelliana and Corymbia citriodora exhibit similar behaviors at elevated temperatures, favoring the production of charcoal with high energy density. In contrast, Eucalyptus urophylla shows greater variation in its properties, particularly in lignin content, which has a direct influence on the production of pyrolytic gases and liquids. Thus, our results may serve as a basis for future research aimed at understanding the properties of charcoal produced from different species and subjected to varying temperatures, with a focus on applications in the steel industry.

4 CONCLUSION

This study underscores the significant impact of pyrolysis temperature and species selection on the properties of charcoal relevant to industrial applications, particularly in the steel industry. The findings reveal that while *Corymbia torelliana* and *Corymbia citriodora* clones produced charcoal with favorable fixed carbon content and calorific value, the highest energy density was not achieved at the pyrolysis temperature of 550 °C. Instead, optimal energy density was observed at 350 °C, suggesting that 550 °C may not be ideal for maximizing the energy density of charcoal, despite its advantages for other properties.

In contrast, *Eucalyptus urophylla* exhibited greater variability in its properties, particularly in lignin content, which influenced the production of pyrolytic gases and

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liquids. Despite the remarkable quality characteristics of *Corymbia citriodora* charcoal, making it highly suitable for use in the steel industry, it is evident that its full quality potential has not been reached. This is largely because the current management and production methods were developed for *Eucalyptus* species. Therefore, specific research is necessary to develop methodologies tailored to *Corymbia* hybrids, enabling them to achieve their maximum quality potential. Future research should explore varying final pyrolysis temperatures, heating rates, and operational methods to optimize charcoal production processes and fully harness the quality potential of these clones.

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