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Quality and physicochemical characteristics of *Eucalyptus* charcoal produced at different ages and carbonization temperatures

Qualidade e características físico-químicas do carvão de eucalipto em diferentes idades e temperaturas de carbonização

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ABSTRACT: Brazil is a leader in global charcoal production. The *Eucalyptus* genus has good growth rates and wood properties, allowing the production of quality charcoal. However, characteristics such as age and carbonization temperature influence product quality. Thus, the aim of this study was to evaluate the influence of the wood age of a hybrid GG100 (*Eucalyptus urophylla* × *Eucalyptus grandis*) and carbonization temperature on charcoal quality. Trees were sampled at 12, 24 and 42 months of age. Carbonization was performed in an electric oven (furnace) at three carbonization temperatures to determine the influence of wood parameters (age, specific gravity, insoluble lignin, and extractive contents) and carbonization temperature on charcoal quality (gravimetric yield, immediate chemical composition, and apparent specific gravity). Overall, we observed differences in the yields of charcoal and pyroligneous liquid and in charcoal properties across ages and carbonization temperatures, as well as gas yield across the ages. In particular, wood age and insoluble lignin content were positively associated with fixed carbon content and negatively associated with pyroligneous liquid content. Additionally, the carbonization temperature was negatively associated with volatile matter content. According to standards, the charcoal produced from trees aged 24 and 42 months and at temperatures of 500 ºC and 550 °C, meets the requirements of the steel industry.

RESUMO: *O Brasil é um dos líderes mundiais na produção de carvão vegetal. O gênero Eucalyptus se destaca pelo seu incremento e propriedades da madeira, permitindo a produção de um carvão de qualidade. Características como idade e temperatura de carbonização influenciam na qualidade do produto. Assim, objetivou-se avaliar a influência da idade da madeira do híbrido GG100 (Eucalyptus urograndis) e da temperatura de carbonização na qualidade do carvão vegetal produzido. Amostraram-se árvores com 12, 24 e 42 meses de idade. As carbonizações foram realizadas em forno elétrico tipo mufla, em três marchas de carbonização. Avaliou-se, portanto, a influência dos parâmetros da madeira (idade, massa específica e teores de lignina insolúvel e extrativos) e temperaturas de carbonização na qualidade do carvão produzido (rendimento gravimétrico, composição química imediata e massa específica aparente). Observou-se diferenças nos rendimentos em carvão e líquido pirolenhoso e nas propriedades do carvão entre as idades e marchas de carbonização, bem como no rendimento em gás entre as idades. A idade e o teor de lignina insolúvel se associaram positivamente com o teor de carbono fixo e negativamente com o rendimento em líquido pirolenhoso. Além disso, a marcha de carbonização se associou negativamente com a quantidade de materiais voláteis. De acordo com a norma, o carvão produzido nas idades de 24 e 42 meses e nas temperaturas 500 e 550 °C atende às exigências do setor siderúrgico.*

KEYWORDS
Carbonization
Carbonization temperature
Gravimetric yield
Eucalyptus urograndis
Juvenile wood

PALAVRAS-CHAVE
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1 Introduction

In 2018, the consumption of charcoal in Brazil was 4.57 million tons, 2.5% higher than that in 2017, with 91% of the charcoal being produced from planted forests (IBÁ, 2019). The usage of wood from planted forests has increased in recent years, conforming to the trend that every year the consumption of native wood decreases because of the greater control exercised by inspection agencies and increasing social pressures to preserve natural resources.

The area occupied by planted forests in Brazil was 7.83 million ha in 2018, with Eucalyptus plantations occupying 5.67 million ha (IBÁ, 2019). The steel industry utilized 12% of the total planted area in 2018, cementing Brazil's leadership in charcoal production, contributing 19% of all charcoal produced worldwide (IBÁ, 2019).

The genus *Eucalyptus* is used in the steel industry because of its fast-growing characteristics, and its technological characteristics, which favor it as an input for thermal conversion and quality charcoal generation (Silva & Ataíde, 2019; Brito *et al.*, 2020). Further, the soil and climate characteristics of Brazil favor the development of *Eucalyptus*, ensuring its dominance in charcoal production.

Considering the demand for *Eucalyptus* charcoal, several studies have been conducted to optimize the charcoal production process. Some of the proposed improvements concern to genetic (Protásio *et al.*, 2019; Protásio *et al.*, 2021), silvicultural (Rocha *et al.*, 2017), wood technological (Protásio *et al.*, 2019; Protásio *et al.*, 2020) and industrial processes (Rodrigues & Junior, 2019). Improvements have been sought in increment, specific gravity, lignin content, and reduction of extractives and ash contents in wood, to ensure the production of quality charcoal. However, little is known about the quality of the wood obtained from *Eucalyptus* species established in the state of Mato Grosso, which focuses on charcoal production.

Knowledge regarding the effect of age on wood properties allows industries to determine the ideal harvest age based on wood characteristics and quality for the eventual use of the harvested wood (Brito *et al.*, 2020). Several physical, chemical, and anatomical modifications occur in the wood during tree development, especially during the transition years between juvenile and mature wood (Castro *et al.*, 2016). Several studies on different ages of wood in energy production (Soares *et al.*, 2015; Castro *et al.*, 2016) have demonstrated the potential in using younger materials (Ferreira *et al.*, 2017).

Wood carbonization must also be considered in the charcoal production process. Some properties of charcoal are related to the temperature used during carbonization. For example, higher carbonization temperatures reduce charcoal yield and increase the fixed carbon content (Dufourny *et al.*, 2019). Thus, the carbonization process must be considered based on the raw material. Therefore, the objective of this study was to evaluate the effect of the wood age of hybrid GG100 (*Eucalyptus urophylla* × *Eucalyptus grandis*) and the carbonization temperature on its charcoal quality.

2 Materials and Methods

We collected 12, 24, and 42 months old *Eucalyptus* hybrid GG100 (*Eucalyptus urophylla* × *Eucalyptus grandis*) trees, from a commercial plantation with a spacing of 3.6 m × 2.0 m, located in the northern region of the state of Mato Grosso. Five trees, located in the center of the planting area, were sampled for each age, totaling 15 trees.

The trees were sectioned into logs with lengths of 0, 25, 50, 75, and 100% that of commercial height. Discs (2.5 cm thick) were removed from the center of the logs and subdivided into four parts, passing through the pith. We randomly selected two opposite parts of each disc to determine the basic specific mass.

The remainder of the logs were sectioned into discs (2.5 cm thick) and subdivided into four parts passing through the pith. Two opposite parts of each disc were destined for charcoal production and the other two parts were transformed into chips for chemical analysis.

To determine the wood specific gravity, we used the immersion in water method. The green volume was determined after saturating the wood in water, and the dry weight was determined after drying the wood in an oven (105 ± 2 °C). To analyze the chemical composition of the wood, the samples (chips) were transformed into sawdust in a Wiley mill. Subsequently, sieves with meshes between 40 and 60 were used to analyze the material retained between the sieves.

To determine the extractive content, extraction was conducted using cyclohexane, ethyl acetate, and methanol solvents. The lignin content was determined using the "Klason" (insoluble) method, with 0.3 g extractive free material in 72% sulfuric acid and distilled water.

Before carbonization, the wood was dried in an oven at 65 ± 3 °C until the local equilibrium moisture content (15 ± 3%) was reached.

For carbonization, a cylindrical metallic container made of cast iron was used and heated in an electric oven (furnace) under temperature control. We considered three different treatments (carbonization temperatures) based on the heating control, as shown in table 1. Final temperatures of 450, 500, and 550 ºC with durations of 5 h 30 min, 7 h, and 8h, respectively, were used. The start time was considered to be the moment the furnace reached the first temperature (150 ºC). We performed six repetitions at each carbonization temperature.

After carbonization, the yields of the solids (charcoal), liquids (pyroligneous), and gases (non-condensable) were determined based on the dry mass of the wood.

To determine the apparent specific gravity of the charcoal, the immersion in mercury method was used in accordance with ABNT NBR 9165 (ABNT, 1985).

For the immediate chemical composition analysis of the charcoal, the samples were transformed into sawdust in a Wiley mill to determine the ash, volatile materials, and fixed carbon contents, in accordance with ABNT NBR 8112 (ABNT, 1986).

The ash content was determined by burning the charcoal samples in a melting crucible without lid for 6 h at 700 ºC. The volatile material content was determined by placing the charcoal in a melting crucible with a lid and heating it at 900 ºC using furnace. The samples were placed at the furnace door for 3 min and then placed inside for 7 min with the door kept closed. To determine the fixed carbon content, the contents of the ash and volatile materials were added and then subtracted from 100.

The experiment was conducted in a completely randomized design. The physicochemical properties of the wood were assessed using analysis of variance and Tukey's post hoc test (α: 0,05). To analyze the charcoal yields and properties, a 3 × 3 factorial scheme (three ages and three carbonization temperatures) was used, which was then tested using factorial analysis of variance to assess the effect of each factor and interaction, and Tukey's post hoc test (α: 0,05).

Additionally, we used the redundancy analysis (RDA) method to verify the associations observed between wood properties, carbonization temperature, and charcoal properties. The test was conducted using the vegan package in R software (Oksanen et al. 2019) with 999 permutations under a full permutation model.

3 Results and Discussion

The specific gravity, total extractives, and insoluble lignin content of the wood did not reveal significant differences between ages (Table 2).

Table 1. Time and carbonization temperature.

Tabela 1. Tempo e temperatura de carbonização.

Treatment	Temperature (°C)							Time
	150	200	250	350	450	500	550	
T1	1 h	1 h	1 h 30 min	1 h 30 min	30 min			5 h 30 min
T2	1 h	1 h	1 h 30 min	1 h 30 min	1 h	1 h		7 h
T3	1 h	1 h	1 h 30 min	1 h 30 min	1 h	1 h	1 h	8 h

Conversely, the charcoal (CY), pyroligneous liquid (LY) and gas yields (GY) showed significant differences between ages (Table 3). Specifically, the CY was the highest at 24 and 42 months, LY was the highest at 12 months, and GY was the highest at 42 months. There was a significant difference in the CY across the carbonization temperatures, showing greater efficiencies in the T1 and T2 treatments than in T3 treatment. However, LY was higher in theT3 treatment than in the T1 and T2 treatments (Table 3).

Properties of charcoal such as volatile materials (VM), ash (AC), and fixed carbon (FC) contents and apparent specific gravity of charcoal (SGc) (Table 3) were significantly different across the ages of the wood. In particular, the VM content was the highest at 12 and 24 months, AC content was the highest at 12 months, FC content was the highest at 42 months and, SGc content was the highest at 12 months (Table 3). Regarding carbonization temperature, the VM content was the highest at T1, and the AC and FC contents were the highest at T3 (Table 3).

Table 2. Wood properties of GG100 hybrid (*Eucalyptus urophylla* × *Eucalyptus grandis*) with 12, 24 and 42 months.

Tabela 2. Propriedades na madeira do híbrido GG100 (*Eucalyptus urophylla* × *Eucalyptus grandis*), com idade de 12, 24 e 42 meses.

Age (months)	Specific gravity (g cm^{-3})	Total extractives (%)	Lignin (%)
12	0,414 a	1,56 a	14,36 a
24	0,414 a	2,61 a	20,58 a
42	0,407 a	2,31 a	17,73 a

Values followed by the same letter (column) do not differ statistically at the 5% significance level by Tukey's post hoc test.

Valores seguidos pela mesma letra (coluna), não diferem estatisticamente ao nível de significância de 5% pelo teste de tukey.

The analysis of the interaction between age and carbonization temperature showed significant differences in the properties of charcoal (VM, AC, FC, and SGc) (Table 4). The VM content was the highest when carbonization temperature T1 was used for wood at 12 months, T2 was used for wood at 12 and 24 months, and T3 was used for wood at 24 months. Regarding VMs in each age group, T1 had the highest content for all three ages.

Table 3. Charcoal yield and properties of GG100 hybrid (*Eucalyptus urophylla* × *Eucalyptus grandis*) in different ages and carbonization temperature.

Tabela 3. Rendimentos e propriedades do carvão do híbrido GG100 (*Eucalyptus urophylla* × *Eucalyptus grandis*), em diferentes idades e marchas de carbonização.

	Age						
	CY (%)	LY (%)	GY (%)	VM (%)	AC (%)	FC (%)	SGc (g cm^{-3})
12	28,66 b	47,19 a	24,52 c	22,63 a	2,51 a	69,12 c	0,314 a
24	30,24 a	43,92 b	25,83 b	22,02 a	0,97 b	77,01 b	0,291 b
42	30,76 a	40,83 c	28,41 a	19,91 b	1,06 b	78,95 a	0,280 b
	Carbonization temperature						
T1	31,06 a	42,36 b	26,95 a	26,92 a	1,16 b	71,84 c	0,291 a
T2	30,03 a	44,22 ab	25,76 a	20,01 b	1,50 b	75,90 b	0,299 a
T3	28,58 b	45,37 a	26,05 a	17,62 c	1,88 a	77,34 a	0,295 a

Values followed by the same letter (column) do not differ statistically at the 5% significance level by Tukey's post hoc test.

Valores seguidos pela mesma letra (coluna), não diferem estatisticamente ao nível de significância de 5% pelo teste de tukey.

The AC content at T2 and T3 were higher at 12 months, whereas at T1 there was no significant difference between the ages. AC content was the highest at T3 only for the wood aged 12 months (Table 4). The highest FC content was observed for the wood aged 24 and 42 months in the T1 treatment and 42 months in the T2 and T3 treatments. Further, it was observed that at 24 months, T3 had the highest FC content, while at 42 months, T2 and T3 had the highest FC content.

The SGc values at T1 and T3 were the highest at 12 months, whereas at T2, there was no significant difference observed among the ages. Regarding the SGc values of each age group, there was a significant difference between the treatments aged 24 months, in which T2 attained the highest value (Table 4).

Table 4. Charcoal properties of GG100 hybrid (*Eucalyptus urophylla* × *Eucalyptus grandis*) in different interactions between ages and carbonization temperature.

Tabela 4. Propriedades do carvão do híbrido GG100 (*Eucalyptus urophylla* × *Eucalyptus grandis*), em diferentes interações entre idades e marchas de carbonização.

Volatile materials (VM - %)			
	12 months	**24 months**	**42 months**
T1	29,00 aA	25,18 bA	26,60 bA
T2	21,24 aB	21,43 aB	17,36 bB
T3	17,66 abC	19,46 aC	15,76 bB
Ash (AC - %)			
	12 months	**24 months**	**42 months**
T1	1,54 aC	0,95 aA	0,98 aA
T2	2,58 aB	0,98 bA	0,94 bA
T3	3,42 aA	0,98 bA	1,24 bA
Fixed carbon (FC - %)			
	12 months	**24 months**	**42 months**
T1	69,46 bA	73,87 aC	72,17 aB
T2	68,42 cA	77,59 bB	81,69 aA
T3	69,46 cA	79,57 bA	83,00 aA
Apparent specific gravity of charcoal (SGc – g cm^{-3})			
	12 meses	**24 meses**	**42 meses**
T1	0,318 aA	0,279 bB	0,277 bA
T2	0,298 aA	0,312 aA	0,287 aA
T3	0,328 aA	0,281 bB	0,277 bA

Values followed by the same lowercase letter in the line (compare ages within each treatment) and uppercase letter in the column (compare treatments within each age) do not differ statistically at the 5% significance level by Tukey's post hoc test.

Valores seguidos da mesma letra minúscula na linha (comparam idades dentro de cada tratamento) e maiúscula na coluna (comparam tratamentos dentro de cada idade) não diferem estatisticamente ao nível de significância de 5% pelo teste de tukey.

The multivariate RDA revealed that wood properties and carbonization temperatures were significantly associated with charcoal properties (p = 0.001). Specifically, RDA 1 and 2 accounted for 65.86% of the data variability (Figure 1). The diagram suggests that the FC content was positively associated with age and insoluble lignin content and negatively associated with the specific gravity of the wood, which corroborates the results of the other analysis. This association occurred because the 12-month-old wood had the highest specific gravity, although not significantly (Table 2).

Nevertheless, the yield of pyroligneous liquid was negatively associated with age and insoluble lignin content and positively associated with the specific gravity of the wood, consistent with the other results (Table 3). Moreover, the carbonization temperature was negatively associated with the volatile material content, which also validates the data previously presented (Table 4).

In this study, the effect of age on wood from the same clone was analyzed. Previously, Castro *et al.* (2016) found no significant difference in specific gravity between the ages of 3 and 5 years, corroborating the results of this study. In particular, they observed values that were higher (0.452 - 0.461 g cm-3) than those found in this study (0.407 - 0.414 g cm-3).

The same authors observed an increase in the total extractive content with increasing wood age. Although in our study there was no significant difference in the total extractive contents between ages, an increase was observed between the ages of 12 and 24 months (1.56% and 2.61%, respectively). This result is related to the heartwood formation process that occurs in the wood (deposition of extractives), which causes extractive content to be more pronounced at younger ages (Brito *et al.*, 2020).

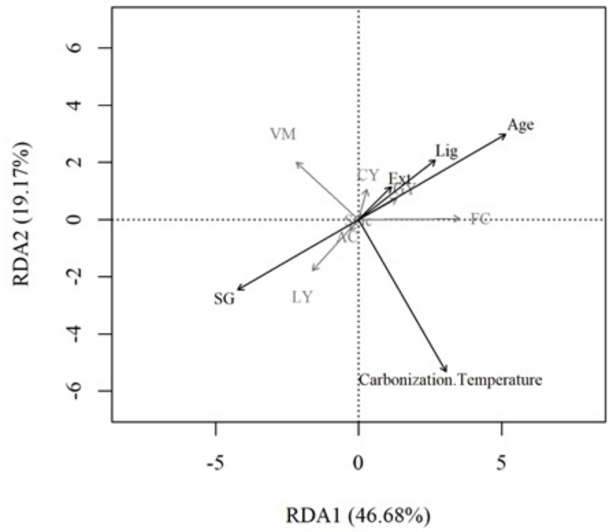

Figure 1. Ordination diagram of the redundancy analysis between wood properties carbonization temperature and charcoal properties of eucalyptus wood.

Figura 1. Diagrama de ordenação da análise de redundância entre as propriedades da madeira e marcha de carbonização com as propriedades do carvão da madeira de eucalipto.

The insoluble lignin content observed in this study was lower than that observed in other studies. Ferreira *et al.* (2017) obtained a lignin content of 27.24% for wood of the same clone used in this study at 1 year. This may be related to environmental and silvicultural factors, that affect the formation of the juvenile wood of these trees (Moore & Cown, 2017).

The high CYs observed in the older materials (24 and 42 months) could be related to the chemical and physical properties of the wood. Although we did not observe significant differences in their insoluble lignin contents and specific gravities, their higher CY may have been influenced by the type of lignin present and the C/H ratio (Soares *et al.*, 2015). They observed an increase in GY and a decrease in LY with increasing material age, which is consistent with the results of this work. However, they stated that such variations cannot be explained exclusively by material age.

Regarding carbonization temperature, the higher the final carbonization temperature, the lower the CY. As the carbonization temperature increase, the degradation of the wood's chemical constituents occurred, which decreased the CY (Rodrigues & Junior, 2019). The GY was not influenced by an increase in the final carbonization temperature but was influenced by the age of the wood. The generation of gases during charcoal production can cause environmental pollution by contributing to atmospheric emission. However, these gases can be used in a kiln-furnace system to obtain energy for the pyrolysis process (Rodrigues & Junior, 2019), reducing environmental pollution and increasing energy efficiency.

The CYs found in this study are consistent with those of previous studies. Specifically, Dufourny *et al.* (2019), evaluated final carbonization temperatures ranging from 500 ºC to 800 °C and obtained CYs between 33.8% and 28.1% for *Eucalyptus globulus* wood. In addition, for a 3-year-old hybrid wood of *Eucalyptus urophylla* and *Eucalyptus grandis*, Soares *et al.* (2015) found a CY of 31.61%. Further, for the same clone at 3 and 4 years, Castro *et al.* (2016) obtained yields of 32.98% and 33.10%, respectively.

The VM content observed in this study, decreased with the increasing material age. Castro *et al.* (2016) found VM contents ranging between 21.34% and 23.77% for the same clone, with ages ranging between 3 and 7 years.

We observed a reduction in VM content with an increase in the final carbonization temperature, which is consistent with previous research (Silva *et al.*, 2018; Rodrigues & Junior, 2019). According to Rodrigues & Junior (2019), a reduction in VM content improves charcoal quality. For industry requirements, the VM content of the charcoal must be below 23.5% (Souza *et al.*, 2016). Thus, in this study, only T1 across all ages does not meet the required standards.

The reduction in AC content with age observed in this study is consistent with previous studies (Soares *et al.*, 2015; Castro *et al.*, 2016). According to Brito *et al.* (2020), such behavior is because of the increased demand for minerals by younger trees, which occurs because of accelerated metabolism in this phase. With respect to the carbonization temperature, an increase in the amount of AC with increasing final carbonization temperature can be explained by the increased concentration of minerals caused by mass losses (Silva *et al.*, 2018).

For use in the steel industry, the AC content should, in general, be less than 1.5%, according to the standard PMQ 3-03 (São Paulo, 2003). Thus, only the 24 and 42 month old woods meet the parameters required by the industry. A high AC content is undesirable because it reduces the calorific value of the fuel and can cause equipment corrosion and the accumulation of impurities in the process, reducing product quality (Souza *et al.*, 2016; Harun *et al.*, 2019; Yao *et al.*, 2020). This is because ash is made up of a fraction of inorganics that are not degraded during the combustion process and do not contribute to energy generation (Carneiro *et al.*, 2016).

In this study, the highest FC content was observed in the oldest material (42 months). Kumar *et al.* (2011) did not obtain an increasing trend in FC content with age in the wood of *Eucalyptus* hybrids aged 2-6 years. However, they found that in the adult woods of the species, the FC concentrations were higher. Nevertheless, when evaluating the same hybrid species considered in this study, Soares *et al.* (2015) did not observe an increasing trend of FC content with age. Thus, they stated that variation in FC content cannot be explained by the age of the wood.

Higher FC contents at higher final carbonization temperatures (T3) were also observed by Marchesan *et al.* (2019), who evaluated the same 6-year-old *Eucalyptus* hybrid at two carbonization temperatures with final temperatures of 450 ºC and 500 °C. They found that the FC content is inversely proportional to the VM content, which is consistent with our results.

According to the PMQ 3-03 standard (São Paulo, 2003), the steel industry should use charcoal that has a FC content above 75%. In this study, the charcoal produced at 24 and 42 months and in treatments T2 and T3 (Table 4) presented levels above those required by the standard.

The apparent SGc values observed in this study were consistent with those of previous research. For 3-7-year-old trees of the same clones, Castro *et al.* (2016) observed SGc values between 0.262 g cm^{-3} and 0.330 g cm^{-3}, but it was not correlated with the material age. Moreover, when evaluating the charcoal produced from a 7-year-old wood of *Eucalyptus grandis* × *Eucalyptus camaldulensis* at different planting spacings, Rocha *et al.* (2017) observed SGc values between 0.35 g cm^{-3} and 0.39 g cm^{-3}.

Wood properties are related to charcoal properties. For example, it has been reported that woods with higher specific gravity (SG) values produce charcoal with higher specific gravity (SGc) (Carneiro *et al.*, 2016; Castro *et al.*, 2016). Similarly, woods with higher specific gravity values produce charcoal with higher FC contents (Marchesan *et al.*, 2019). Among the physical properties of wood, SG is considered to be a useful index for evaluating wood quality, as it is related to the properties of charcoal in terms of yield, density, and quality.

However, this tendency was not observed in the study. This can be explained by the fact that we had used juvenile wood in our treatments, which has a more variable nature than that of adult wood (Moore & Cown, 2017). Thus, anatomical, and chemical properties, which were not considered in this study, may influence the results obtained.

Another aspect to be considered is ideal harvest age. Forest-based industries have stipulated harvest age based on the volumetric productivity of the forest. However, wood properties must be considered to determine the ideal harvest age (Brito *et al.*, 2020). In this sense, the use of Monte Carlo simulations can be employed as an alternative for future studies. This methodology has been used for forest economic analyses (Gonçalves *et al.*, 2017); therefore, it can be used to determine the economic feasibility of using 24-month-old wood.

4 Conclusion

Age and carbonization temperature also influenced the charcoal produced from *Eucalyptus urophylla* × *Eucalyptus grandis* wood. Overall, the charcoals produced using wood aged 24 and 42 months, at carbonization temperatures of 500 ºC and 550 ºC and for durations of 7h and 8h, respectively, met the quality standards required by the steel industry.

This research demonstrates that the young wood of clone GG100 has the potential to produce quality charcoal. Nevertheless, further research on the economic feasibility of using this low-age wood should be conducted.

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