Research Article

Effect of final temperature on charcoal stiffness and its correlation with wood density and hardness

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Abstract
Mechanical performance is important for charcoal used in blast furnaces as charcoal layers may support the load of iron ore within blast furnaces without breaking. Pyrolysis temperature influences charcoal quality and its chemical composition; however, the effect of temperature on physical-mechanical properties is still unknown. Thus, the aim of this study was to establish the effect of pyrolysis temperature on the density, stiffness and hardness of charcoal and the correlations among them. Four pyrolysis processes were performed at final temperatures of 300, 450, 600 and 750 °C using wood specimens from nine different tree species. Stiffness of wood and charcoal was determined by ultrasound and dynamic hardness by a portable hardness tester. In short, density, dynamic hardness and stiffness of charcoal tend to decrease with increasing temperature. Pyrolysis at 450 °C decreases charcoal stiffness by approximately 30%, while hardness is reduced from 29 MPa in wood specimens to approximately 3 MPa in charcoal pieces produced from the same specimens. Considering the wood, the highest values of stiffness were presented by the same materials which presented highest density, confirming high positive correlation between wood properties. The correlation between density and stiffness in charcoal is higher than in wood. However, correlation between density and dynamic hardness or stiffness and dynamic hardness is higher for wood than for charcoal. Ultrasound was able to determine differences in stiffness between the materials at different pyrolysis temperatures. These findings are useful to identify the best production temperature for industrial charcoal with adequate mechanical properties.

Keywords Carbonization · Rigidity · Specific gravity · Nondestructive testing · Green steel

1 Introduction

Pyrolysis process causes physical, anatomical and mechanical changes within the wood that influence the properties of the resulting charcoal and its final quality. As pyrolysis temperature affects cellular composition and degrades wood components, the carbonized material mainly composed of graphitic carbon [1]. Dufourny et al. [2] have stated that quality of charcoal also depends on the characteristics of the precursory wood. Quality control of charcoal is defined by steel industry, which concern is directly related to its behavior in furnace. Mechanic performance of charcoal is one of the most important properties inside the blast furnace, where charcoal layers should support the load of pig ore within blast furnaces without collapsing.

Among the most important biomass traits, wood density [3–5], wood stiffness as indicated by modulus of elasticity in bending or compression [6, 7] and wood hardness [8] play a key role in charcoal quality. These combined factors may influence critical physical-mechanical properties of charcoal such as density [9], stiffness [10] and hardness

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[11]. The evaluation of mechanical properties of charcoal is useful to classify material raw and use it rationally.

Correlation between hardness and density in wood is well known [11, 12] as well as the high correlation between density and stiffness [13, 14]. Due to variations during the pyrolysis process, the properties of wood and the resulting charcoal can be different [5, 10]. To our knowledge, only two studies have evaluated hardness [15] and stiffness [16] of charcoal specimens.

Brazil is the main producer of charcoal in the world. In 2018, it produced 6.4 tons, accounting for 11% of the world's charcoal production [17, 18]. As one of the few countries in the world to use charcoal on a large scale in the production of "green steel," the country's domestic consumption is highlighted, which one consumed 4.6 million tons of charcoal in the same year [18].

Veiga et al. [10] have evaluated mechanical strength, such as compression tests parallel to fibers and static bending, of charcoal and pointed out the lack of a methodology for charcoal makes it difficult to standardize the tests. Therefore, new solutions are necessary to evaluate the stiffness and hardness of charcoal, and nondestructive equipments can be effective to close this gap.

In the present study, two equipments already successfully tested for wood evaluations were applied to charcoal specimens: (1) an automated portable hardness tester developed to measure dynamic hardness of viscoelastic materials [19], and (2) ultrasound—an effective tool in the inference of physical and mechanical properties of wood that allows indirectly to estimate elastic constants of materials [14]. The combinations of these two approaches can be useful. The ultrasonic method is efficient for fast estimations of mechanical parameters such as modulus of elasticity and rupture [20] while the portable hardness tester has presented promising results for evaluating dynamic hardness of wood [19] and charcoal [15] specimens.

Few studies have dealt with the correlations between mechanical properties of wood and resulting charcoal quality [10]. The relationship among density, stiffness and hardness of charcoal as a function of final pyrolysis temperature remains still unknown. Therefore, the aim of this study was (1) to better understand the effect of pyrolysis temperature on charcoal stiffness and (2) to establish to what extent the relationships between stiffness with density and hardness of charcoal are influenced by pyrolysis temperature.

2 Material and method

2.1 Wood species

Wood specimens from nine different tree species were studied: one *Corymbia citriodora*, six *Eucalyptus* species and two *Eucalyptus* hybrids from commercial plantations. To test the tools as potential quality differentiators, it was sought to vary species, ages and densities of wood, as described in Table 1. These wood species represent a large genetic variation, among progeny test and *Eucalyptus* trees for industrial applications in Brazil. The wood presents potential to be used as charcoal to produce steel (*Eucalyptus grandis* × *Eucalyptus urophylla* hybrids—6 years old) and for pulp and paper production (*Eucalyptus urophylla* × *Eucalyptus grandis* hybrids—6.5 years old). These woods were also investigated in the study by Abreu Neto [15] and Ramalho et al. [21].

Wood specimens with nominal dimensions of 25 × 25 × 40 mm (R × T × L) were produced, 25 pieces of each material were cut (9 × 25), totalling 225 specimens (Fig. 1a). Wood specimens were conditioned (20 °C ± 1 °C and 65% ± 3% RU) until EMC was achieved.

Table 1 Description of the species, ages, origins, wood apparent density and statistical analysis of variance (ANOVA) of the studied trees

Code	Wood species	Age (years)	Source	Apparent density of wood (kg m^{-3})	SD
1	*E. saligna*	37	Pt	988.64^{a}	37.23
2	*C. citriodora*	37	Pt	967.76^{ab}	22.36
3	*E. microcorys*	37	Pt	952.76^{ab}	74.36
4	*E. deglupta*	37	Pt	950.65^{ab}	39.58
5	*E. cloesiana*	37	Pt	893.36^{b}	49.51
6	*E. urophylla* × *E. grandis*	6.5	Cp	635.55^{c}	20.11
7	*E. pilulares*	37	Pt	627.06^{c}	12.74
8	*E. urophylla* × *E. grandis*	6	Cp	595.47^{cd}	45.09
9	*E. dunnii*	37	Pt	512.05^{d}	45.16

Pt progeny test, *Cp* commercial planting, *SD* standard deviation. Means followed by the same lowercase words in columns do not differ by 5% probability by Tukey test

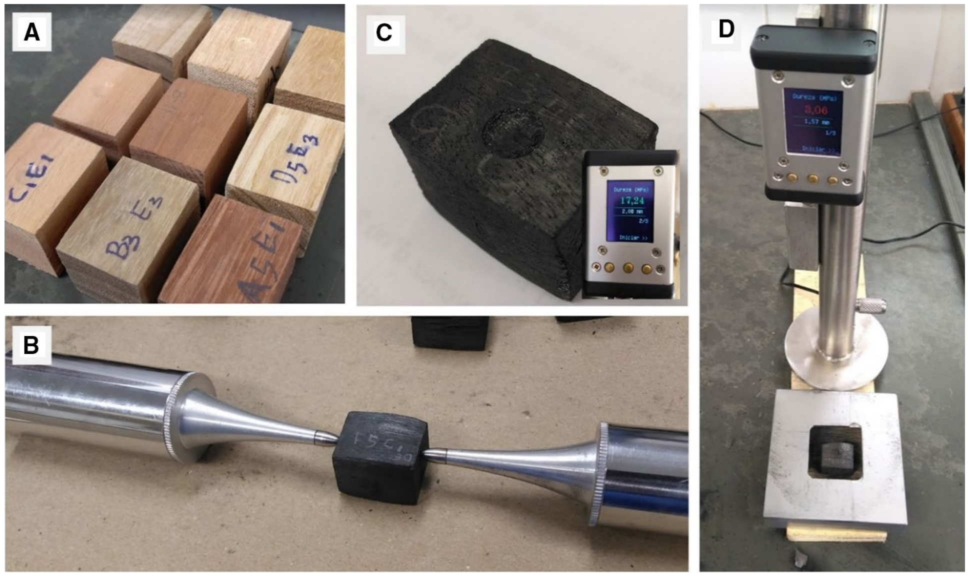

Fig. 1 Wood specimens (**a**), ultrasound testing in charcoal specimens (**b**), charcoal specimen recently tested by durometer (**c**) and dynamic hardness testing prototype (**d**)

2.2 Pyrolysis of the material

To mitigate the propensity to collapse, the wood samples were kiln dried in condition smooth to drastic. Initially, the woods were placed in an oven at 60 degrees until they reached constant mass; after that, they were subjected to treatment at 80 degrees to constant mass and finally, oven set at 100 degrees until reaching constant mass. The samples in dry condition were then subjected to heat treatment at muffle.

Five wood specimens of each species (considered as repetitions) were pyrolyzed inside a carbonization capsule within an electric furnace (muffle) under four thermic treatments, totalling 180 ($5 \times 9 \times 4$) charcoal specimens.

Four pyrolysis processes were performed varying final temperatures: 300 °C, 450 °C, 600 °C and 750 °C. The initial temperature was 100 °C, the heating rate was 1.67 °C min^{-1}, and the residence time at final pyrolysis temperature was 30 min, according to the procedure described in the study of Abreu Neto et al. [15], Neves et al. [22] and Protásio et al. [23].

2.3 Material characterization

2.3.1 Charcoal and wood density

Apparent density of wood was performed by stereometric method, based on Travisan et al. [24] and NBR 14,984 [25]. Specimens were weighed on a 0.01 g analytical balance and measured with digital caliper to obtain mass and volume, respectively.

The apparent relative density of each charcoal specimen was determined according to hydrostatic method, by determining the volume in water and dry weight of the charcoal.

Twenty-seven specimens of wood were performed, with three replicates for each of nine species analyzed. In addition, one hundred and eight samples of charcoal, with three specimens of each of nine species, which were produced under four different temperatures, were measured.

2.3.2 Dynamic hardness

Dynamic hardness (DH) of wood and charcoal was evaluated according to procedure described by Assis et al. [17] and Abreu Neto et al. [15], which used portable hardness tester—DPM3 (Fig. 1d). This device, developed by Brazilian research group, is an electromechanical instrument whose operating principle is similar to the Brinell hardness test [19].

2.3.3 Stiffness by ultrasound

Wood and charcoal stiffness was determined by a *Steinkamp* brand ultrasound device, model BP-7. Device has two equivalent flat piezoelectric transducers—one for emission and another for reception of ultrasound waves, frequency 45 kHz (Fig. 1b). Stiffness (GPa) in longitudinal direction was determined from ultrasound wave propagation speed (dry contact; without gel), as performed by Ballarin et al. [14]. The equipment determines time that the ultrasonic wave takes to cross the material in longitudinal direction. Speed is calculated by the ratio of distance traveled by wave from one end of sample to the other by time (indicated by equipment). Finally, stiffness estimate was obtained using Eq. (1):

$$S = v^2 \times d \quad (1)$$

where: S is stiffness, v is speed of propagation of ultrasound and d is density of wood or charcoal.

A total of 45 measurements were performed on wood specimens, with five replicates for each of nine wood species analyzed. In addition, five specimens of each species, which were produced under four different temperatures, were measured for charcoal, totalling 180 charcoal specimens. Thus, a total of 225 specimens of wood (n = 45) and charcoal (n = 180) were analyzed.

Collapses and cracks easily and frequently arise during the wood-to-charcoal conversion process. After pyrolysis process, these specimens were visually verified and apparently were free from collapse, but this was not actually measured. The charcoal specimens showed only small cracks that we consider negligible for the purpose of this study.

2.3.4 Statistical analysis of the data

Analysis of variance (ANOVA), Tukey's test and regressions were performed to analyze differences between species and temperatures in the variables stiffness, hardness and density. The level of significance was 5%.

3 Results and discussion

3.1 Wood stiffness by ultrasound

Figure 2 shows the variation of wood stiffness and density according to the wood. The mean wood stiffness was 3.920 GPa, and mean wood density was 0.791 g cm^{-3}. The highest values of wood stiffness were presented by *C. citriodora* (Code 2), *E. deglupta* (Code 4) and *E. microcorys* (Code 3). The same materials also presented highest densities, suggesting high positive correlation between these wood properties, as expected. *E. saligna* (Code 1) presented the largest specific modulus (ratio between stiffness and density), indicating the most light and resistant material.

Many studies have shown that ultrasound technique is able to estimate wood stiffness [13, 14, 20]. However, the application of this equipment in brittle materials such as charcoal is scarce. If wood-to-charcoal correlations exist, estimating wood stiffness can be useful for identification of potential materials for use as charcoal.

Almeida et al. [13] studied *Dinizia excelsa* Ducke and reported wood stiffness of approximately 23,919 MPa. The authors found a low correlation with modulus of elasticity and static bending and attributed this result to differences in moisture conditions. Stangerlin et al. [20] used an ultrasound device to evaluate *Peltophorum dubium* (Spreng.) Taub. wood and found elastic constants close to 7800 MPa. The authors mention morphological and anatomical properties may have influenced results. The authors observed dynamic constant is 1.3 times greater than modulus of elasticity due to the viscoelastic behavior of wood. Ballarin et al. [14] performed ultrasonic stiffness measurements to evaluate juvenile wood and adult wood of *Pinus taeda* L. and found values of 11,816 and 17,914 MPa, respectively. Wood stiffness values founded in the literature are greater than observed in this study. Specimens size used in this study

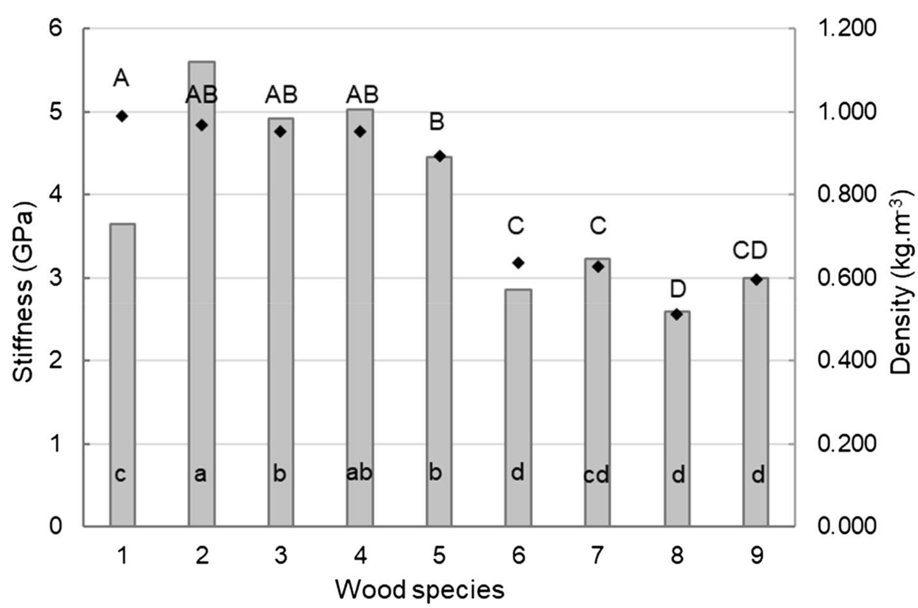

Fig. 2 Wood stiffness (GPa) by wood species in longitudinal direction (bar) and apparent density (kg m^{-3}) (line). Means followed by the same lowercase letters in a column and capital letters on the lines did not differ significantly by Tukey test at 5% significance

may have influenced this result, since ultrasonic stiffness is measured as a function of distance travelled by wave.

3.2 Stiffness of wood species as a function of final pyrolysis temperature

Figure 3 shows charcoal stiffness and density results of different species at temperatures of 300, 450, 600 and 750 °C. In general, the *E. saligna* (Code 1), *C. citriodora* (Code 2) and *E. microcorys* (Code 3) had the highest stiffness values; concomitantly, these materials presented highest values of density, regardless of thermal treatment used (Fig. 3). Interestingly, wood and charcoal of materials 2 and 3 presented highest stiffness and density values while wood and charcoal of material 9 presented lowest stiffness and density values. It is important to note that wood and charcoal were considered as an anisotropic material.

Ultrasound was able to estimate stiffness of materials even after thermal treatment. The equipment was also able to determine differences in stiffness between studied materials at different pyrolysis temperatures. These findings are useful to identify the best production temperature of charcoal based on mechanical properties.

Veiga et al. [10] have observed that *C. citriodora* presented higher stiffness, 1.585 MPa, compared to *E. urophylla*, around 900 MPa. *Citriodora* specimens are also more resistant (5 MPa) than *Eucalyptus urophylla* (3 MPa). Poncsák et al. [26] have investigated birch wood (*Betula papyrifera*) with MOE around 1.500 MPa. According to them, after thermal treatment around 120 °C the value decreases to nearly 1.200 MPa. The authors have reported a maximum peak in the MOE value at 160 °C, and MOE decreases with increasing temperature. It was also mentioned that exothermic chemical reactions start between 150 and 160 °C, probably due to the decomposition of hemicelluloses and cellulose polymers. This behaviour appears to be almost constant under experimental conditions below 250 °C.

The findings reported in this study are close to those observed in the literature [10, 26]. Here, the ultrasound technique allows to quickly and nondestructively observe the tendency to decrease stiffness.

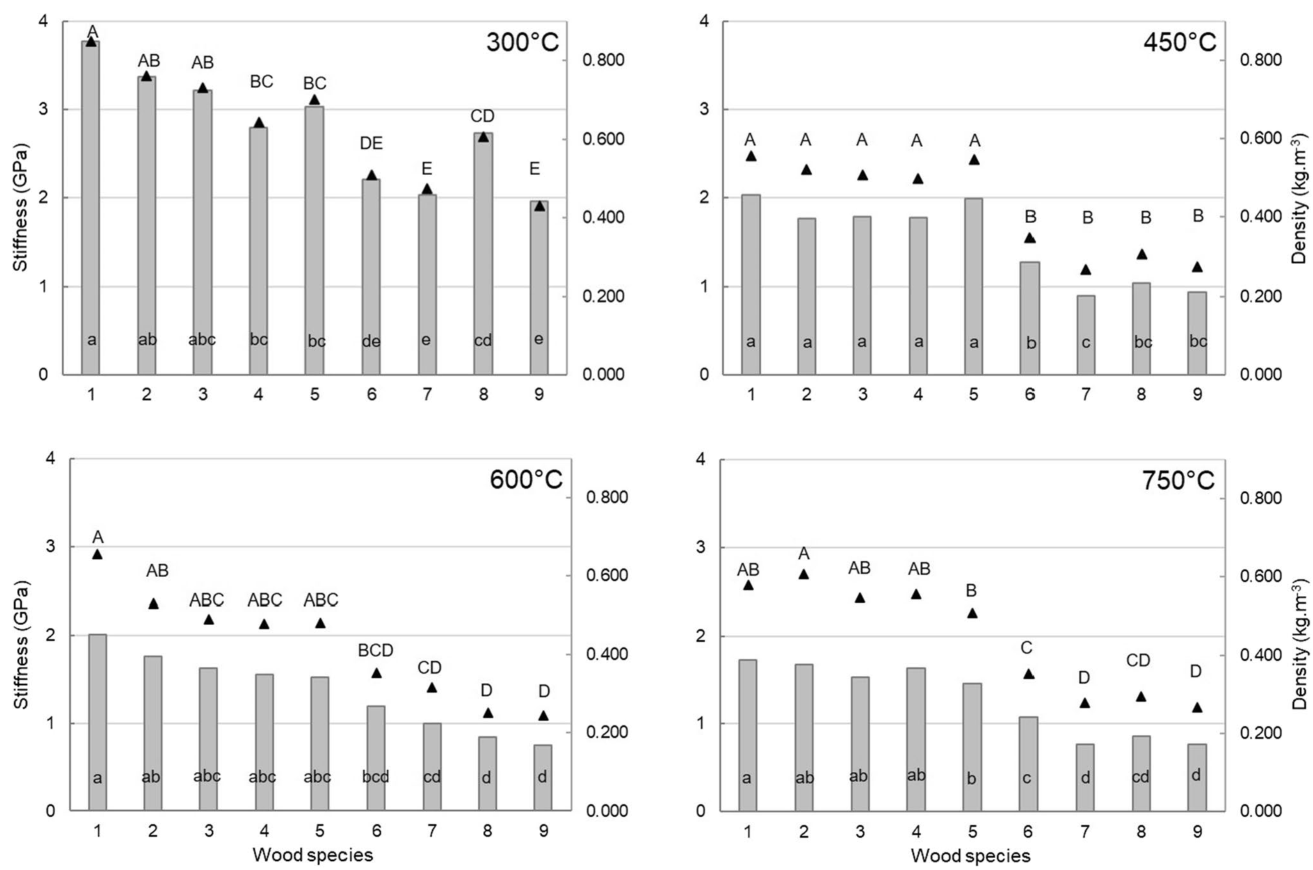

Fig. 3 Charcoal stiffness and density of materials produced at final temperatures of 300, 450, 600 and 750 °C. Means followed by the same lowercase letters in a column and capital letters on the lines did not differ significantly by Tukey test at 5% significance

Table 2 Apparent density, stiffness in longitudinal direction and dynamic hardness of wood and charcoal of nine species at different final pyrolysis temperatures

Final temperature (°C)	Density (kg m^{-3})	Dynamic hardness (MPa)	Stiffness (GPa)
Control	791.48^{a}	29.82^{a}	3920^{a}
300	634.88^{b}	10.72^{b}	2794^{b}
450	424.86^{c}	3.25^{c}	1496^{c}
600	422.85^{c}	3.59^{c}	1362^{c}
750	444.15^{c}	4.63^{c}	1278^{c}

Averages followed by the same lowercase letter in column are not considered statistically different using the Scott Knott test, at significance of 5%

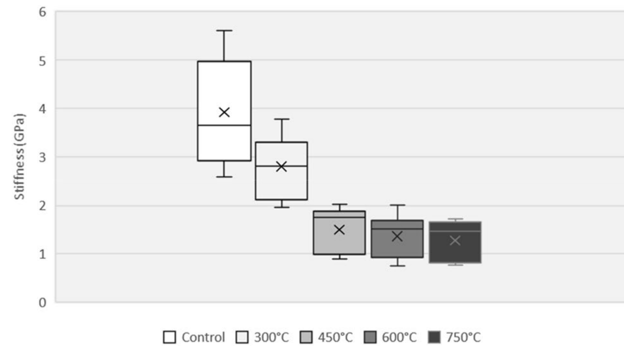

Fig. 4 Variation of charcoal stiffness as a function of final pyrolysis temperature

3.3 Physical-mechanical properties of charcoal as a function of final pyrolysis temperature

Physical and mechanical properties of materials decreased with temperature (Fig. 3). Compared to wood, density of material converted to charcoal produced at 450, 600 and 750 °C presented lower and statistically equal density values (Table 2). According to Veiga et al. [10], charcoal quality and charcoal stiffness are controlled by the final temperature of carbonization. Here, the results showed that pyrolysis temperature also decreases material hardness.

Wood dynamic hardness presented higher value, approximately 29 MPa, compared to charcoal (Table 2). The analysis of variance indicates a significant effect of temperature and material on hardness, with interaction between factors. Dynamic hardness tends to decrease with increasing temperature. For instance, the temperature of 300 °C decreases dynamic hardness to approximately 10 MPa, 1/3 of wood hardness. Above 450 °C, the hardness is statistically equivalent, with values close to 3.5 and 4.6 MPa for 600 and 750 °C, respectively. Stiffness of material produced at 300 °C is 2794 MPa, 28% lower compared to wood (3920 MPa) as shown in Table 2. Charcoal presented lower stiffness with increasing production temperature. Charcoal specimens produced at 450, 600 and 750 °C showed lower values of stiffness, and statistically equal to each other.

In addition to decreasing mechanical properties, temperature also decreases material heterogeneity (Fig. 4).

The heat treatment decreases the stiffness variation of material; control treatment presents amplitude of variation around 3013 MPa. The final pyrolysis temperature of 300 °C produced material with a range of variation of approximately 1334 MPa. At temperature of 750 °C, variation is 963 MPa. Compared with wood variation, pyrolysis temperature of 750 °C produces charcoal with variation approximately 3 times lower.

Veiga et al. [10] have studied *C. citriodora* and *E. urophylla* also observed higher stiffness of wood, an average of 7552 MPa, compared to charcoal produced at 450 °C, average of 1115 MPa. The authors performed mechanical tests in a universal test machine, in accordance with BS-373 [27] for wood, and adapted the methodology for analysis of charcoal stiffness.

Coutinho et al. [28] have reported a reduction in density, from 310 to 180 kg.m^{-3}, with an increasing temperature from 400 to 1000 °C. Trugilho et al. [9] observed decrease in apparent relative density with an increase in pyrolysis temperature (300 to 700 °C). The authors concluded there is a minimum density point in carbonized materials at 660 °C; above this temperature, for instance 900 °C, density increases.

Poncsák et al. [26] have investigated mechanical properties of birch and observed that there is a maximum hardness at about 160 °C, values close to 22 and 28 MPa for samples analyzed radially and transversely, respectively. The authors observed birch hardness reduction with an increase in holding time at the maximum temperature 220 °C, due to a greater structural degradation. Values found are very close to those found in this study. According to Veiga et al. [29], the reduction in mechanical traits observed can be explained by the formation of cracks during carbonization process.

In short, the ultrasound technique was effective for identifying stiffness of charcoal. The results showed that the ultrasound equipment was sensitive to variations in mechanical properties of different wood species at all pyrolysis temperatures analyzed. Abreu Neto et al. [15] presented results on charcoal hardness suggesting that this hardness tester equipment can be used as a reference for mechanical classification of charcoal in steel industries.

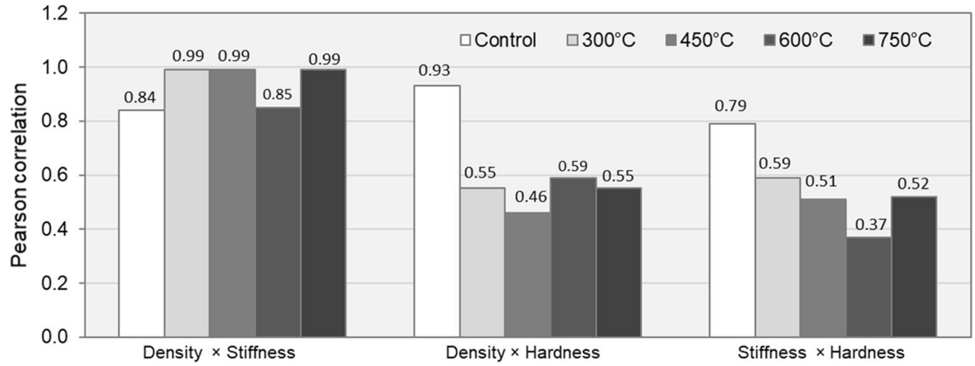

Fig. 5 Correlations among physical-mechanical properties of wood (control) and charcoal produced at different final temperatures

3.4 Correlations among physical-mechanical properties of wood and charcoal

Correlation among physical-mechanical properties of charcoal is higher than found in wood, regardless of final pyrolysis temperature. There was high correlation between wood density and stiffness, *r* = 0.84 (Fig. 5); this correlation increases to *r* = 0.95 with application of heat treatment (Fig. 6a). These results were consistent with those found in the literature [30, 31].

It is possible to observe correlation of *r* = 0.93 between density and dynamic hardness (DH) of wood (Fig. 5). Final pyrolysis temperature negatively affects the correlation between these properties; correlation decreases to *r* = 0.53 (Fig. 6b). Correlation between stiffness and dynamic hardness of wood was *r* = 0.79 (Fig. 5). Correlation of thermally treated material is lower compared to Control material, around *r* = 0.50 (Fig. 6c).

It is known mechanical properties of wood correlate well with density [30]. For charcoal, Couto et al. [32] observed a higher relative apparent density and greater stiffness. The authors observed rearrangement and greater organization of carbon chains under higher final carbonization temperature, producing charcoal with higher stiffness. Veiga et al. [10] found a high correlation between basic wood density and stiffness (*r* = 0.732). In addition to these results, same authors observed a high correlation between wood strength and wood stiffness (*r* = 0.898) but found a lower correlation for strength and stiffness of charcoal (*r* = 0.607).

A high correlation between hardness and density is in agreement with findings of Antal et al. [31] and Chrzazvez et al. [33], who evaluated relationship between density and mechanical strength. According to data found in the literature, there are positive correlation between basic wood density, modulus of elasticity, gravimetric yield and density of charcoal [34, 35].

4 Concluding remarks

Heat treatment decreases the stiffness of wood. Pyrolysis at 450 °C reduced charcoal stiffness by approximately 30% compared to wood. Considering the wood species, the highest values of stiffness were presented by *C. citriodora* and *E. deglupta*, same materials presented highest density, confirming high positive correlation between wood properties.

Correlation between density and stiffness is higher for charcoal (*r* = 0.95, in average) than for wood (*r* = 0.84). However, correlation between density and dynamic hardness or stiffness and dynamic hardness is higher for wood than for charcoal. Correlation between density and dynamic hardness was 0.93 for wood specimens and 0.53, in average, for charcoal specimens. Correlation between stiffness and dynamic hardness was 0.79 for wood and 0.50, in average, when the materials are converted to charcoal.

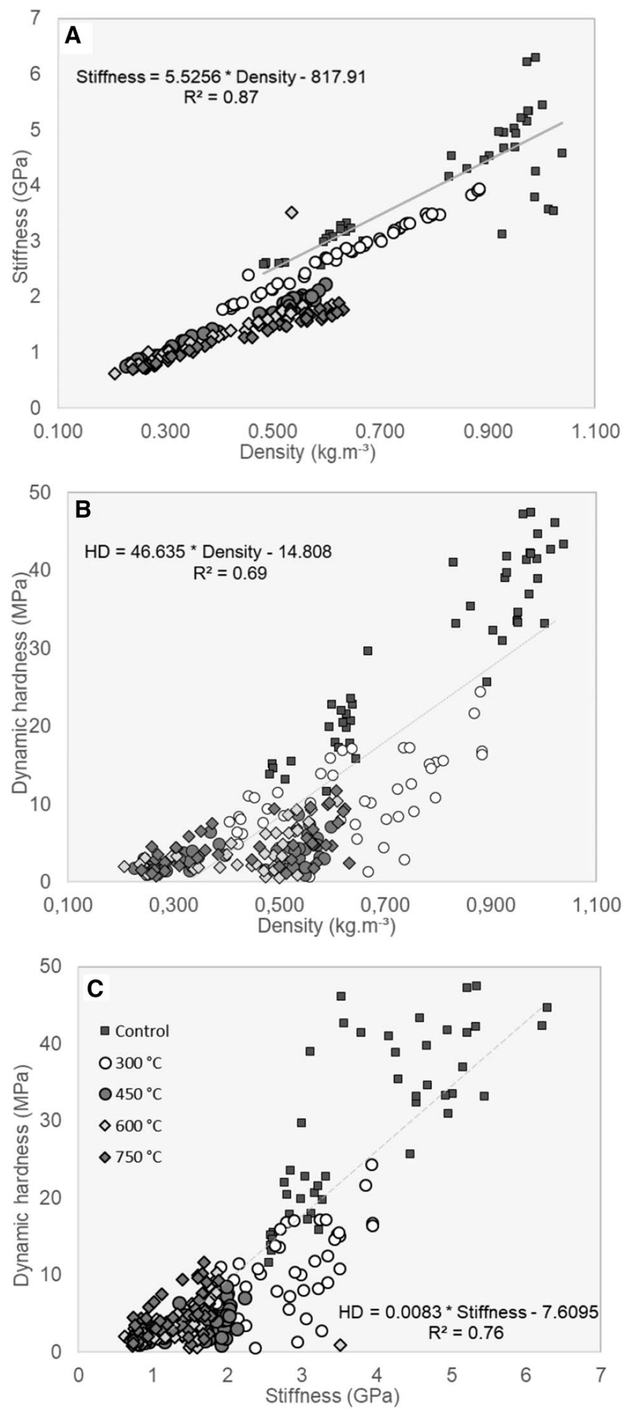

Fig. 6 Correlations between apparent relative **a** density × stiffness, **b** density × HD (dynamic hardness) and **c** stiffness × dynamic hardness for all final pyrolysis temperatures

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Availability of data and material The material and database are available.

Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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