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Sampling sufficiency for mechanical properties of wood

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ABSTRACT

Based on most recently published studies, there is a large variability in both the mechanical properties of wood and sample sizes selected to evaluate them. This study aims to define sampling sufficiency for some mechanical properties of wood, which were bending strength, bending modulus, compressive strength, compressive modulus, hardness, and shear strength. The mechanical tests were carried out according to the ASTM D143 on wood samples cut from clonal *Eucalyptus* planted in southern Brazil. Sampling sufficiency was determined by an intensive computational method based on resampling of original data using Monte Carlo simulations. The experimental tests data conformed to the normal distribution and most of the obtained sufficient sample sizes determined by Monte Carlo simulation were above those sample sizes used in most already published studies. Furthermore, properties related to wood stiffness presented smaller variabilities than their respective properties associated with wood strength, leading to smaller sample sizes for the former cases.

Keywords: Monte Carlo simulation, resampling, sampling sufficiency, wood stiffness, wood strength.

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INTRODUCTION

Efficient use of any material depends on its physical-mechanical properties, including mechanical features related to strength and stiffness. However, a reliable determination of mechanical properties usually implies the destruction of the samples, which may also represent expensive costs, as well as time and effort spent (Bros and Cowell 1987). Therefore, the minimum number of necessary samples to be tested must be determined to ensure a proper characterization of the targeted group of samples, avoiding unnecessary losses, which is called sampling sufficiency (Bros and Cowell 1987). However, a suitable sample size may not be simply determined, although this information is not clearly defined by most standards (DePatta Pillar 1998).

The distribution of the sample mean (also called "expected value" of M) is a widely used method to determine sample sizes used in experimental studies and it is a valid solution for some properties of many materials (Adcock 1997). However, mechanical properties vary in different ranges depending on the targeted material, this uncertainty affects the number of samples that must be evaluated to ensure reliable results (Bros and Cowell 1987, DePatta Pillar 1998).

The multiple variables that influence the mechanical properties in woods generate a complex system of correlations, making the determination of sample sizes extremely difficult, uncertain, and sometimes, even unreliable. Wood is a natural, heterogeneous, and anisotropic material that may display a wide range of mechanical, chemical and hygroscopic properties (Amer *et al.* 2019), which also may depend on wood specie, tree maturity, and density (Amer *et al.* 2021). Moreover, wood characteristics also depend on forest factors (cultivation, location, local climate, etc.) (Hein *et al.* 2016), harvesting factors (overthrow process, different radial position, different axial position, etc.) (Hein *et al.* 2016, Moraisa and Pereira 2007), cutting processes (Svensson and Toratti 2002,

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Rapp *et al.* 2007) and supply factors (transportation condition, storage condition and wood age) (Bao *et al*. 2001, Dünisch *et al*. 2010).

ASTM D143 (ASTM 2021) is a globally known standard procedure devoted to establishing good practices for determining physical and mechanical properties of small and clean wood samples. However, this standard does not specify how many samples should be evaluated for the correct determination of each mechanical property. This uncertainty has been leading to a wide variety of adopted sample sizes in recent studies on physical-mechanical properties of wood. For that, Bao *et al.* (2001) evaluated strength and stiffness of juvenile and mature woods from ten species and 30 samples were selected for each test. Ghorbani-Kookandeh *et al*. (2014) investigated the beech wood impregnated and heat-treated. They carried out static bending, compression strength parallel to grain, and hardness tests by using five samples for each test. Furthermore, Taghiyari (2011) used 60 samples to determine modulus of rupture (MOR) and modulus of elasticity (MOE) in compression parallel to grain tests for the heat-treated *Populus nigra* wood. In addition, some studies even did not report the number of samples used for each mechanical test (Santos 2000, Wessels *et al*. 2016).

Resampling methods could be an alternative to determine sampling sufficiency and avoid unnecessary large samplings. It estimates the accuracy of means, medians, variances, and percentiles of sampling by using subsets of the available data or randomly generating new samplings by resampling the data set (Edwards *et al*. 2011). The resampling methods such as Bootstrapping (Edwards *et al.* 2011), Jackknifing (Wang and Yu 2020), Permutation (Fieberg *et al.* 2020) and Cross-Validation (Shimodaira 2016) have been used for estimating the sample sizes in many areas, such as ecology (DePatta Pillar 1998), biomedicine (Dwivedi *et al*. 2017), zoology (Dimauro *et al*. 2009), and genetics (Kess and El-Kassaby 2014).

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The resampling methods based on Monte Carlo simulations have many advantages since this type of method relies upon the use of randomness to solve problems from a deterministic system, encompassing a large number of possible outcomes from the targeted sample (Papadopoulos and Yeung 2001). Taking these facts into account, this study aimed to use Monte Carlo simulations to determine sampling sufficiency for mechanical properties of *Eucalyptus* wood, which were determined according to ASTM D143 (ASTM 2021).

MATERIALS AND METHODS

Material selection and samples preparation

The studied wood samples were obtained from a clonal test of interspecific hybrids of *Eucalyptus* clones. 29 *Eucalyptus* clones were randomly selected in a planted forest located in southern Brazil following the ASTM D5536 (ASTM 2010). 8 seven-year-old trees by clone were felled, totaling 232 trees. The selected trees had straight trunks, an absence of bifurcation, good phytosanitary conditions and a low presence of defects. Boards were cut from the 1,2 m long baseline logs, which presented a diameter at breast height of around 21 cm.

The boards were air dried and their thicknesses were reduced from 8 cm to 6 cm using a thinner plane. For each test, 54 samples were cut according to the requirements of ASTM D143 (ASTM 2021), as shown in Figure 1, avoiding the presence of growth defects, such as knots, splits, etc. Before the mechanical tests, the samples were placed into a climatic chamber (20 ºC and 65 % relative humidity) until reaching equilibrium humidity of approximately 12 %. Weight and dimensions (measured in the sample centre) were determined using an analytical scale and a digital calliper, respectively.

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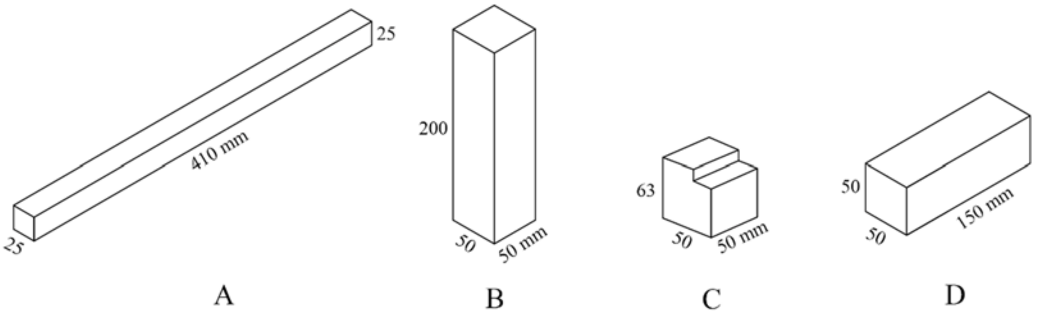

Figure 1: Three-point static bending (a), Compression parallel to grain (b), shear parallel to grain (c) and Janka hardness (d) samples geometry for the mechanical tests.

Mechanical tests

Three points static bending

The three points static bending tests were performed according to ASTM D143 (ASTM 2021). The wood samples were cut with dimensions of 25 × 25 × 410 mm³ (radial × tangential × longitudinal) and a span length of 360 mm was adjusted, keeping a minimum span-to-depth ratio of 14. An Emic DL 30000 universal testing machine was used for applying the load continuously in the centre of the beam throughout the test at a rate of motion of 1,3 mm/min. The load was applied at the tangential plane of the samples and the mid-span deflection was determined by the crosshead position.

Flexural strength (σ_f) was calculated using the relationship between the applied load and the measurements of mid-span deflection of the test sample in accordance with Eq. 1. Modulus of elasticity (E_f) was calculated by Eq. 2 using the gradient (∇m) obtained from the ratio of the load vs. deflection curve in the elastic region. These samples presented an equilibrium moisture content mean of 14,05 % with a standard deviation of 0,63 %.

$$\sigma_f = \frac{3 \cdot \mathrm{Fmax}\ (\mathrm{N}) \cdot \mathrm{L}\ (\mathrm{mm})}{2 \cdot \mathrm{b}\ (\mathrm{mm}) \cdot \mathrm{h}^2\ (\mathrm{mm}^2)} \tag{1}$$

$$E_f = \frac{\mathrm{L}^3\ (\mathrm{mm}^3) \cdot \nabla\mathrm{m}\ (\mathrm{N} \cdot \mathrm{mm}^{-1})}{4 \cdot \mathrm{b}\ (\mathrm{mm}) \cdot \mathrm{h}^3 (\mathrm{mm}^3)} \tag{2}$$

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Where: b, h, and L are the width and height of the specimen, and support span length, respectively.

Compression parallel to grain

Compressive modulus and compressive strength were determined using compression parallel to grain tests in samples with dimensions of 50 x 50 x 200 mm³ (radial × tangential × longitudinal directions). These samples were cut assuring that the end grain surfaces were parallel to each other and at right angles to the longitudinal axis. A crosshead speed of 0,6 mm/min was applied using an Emic DL 30000 universal testing machine, following ASTM D143 (ASTM 2021).

The compressive strength parallel to grain (σ_c) was obtained by the relation of the maximum achieved load (F_{max}) and the cross-sectional dimension at the middle of the specimen (A) according to Eq. 3. Compressive modulus (E_c) was the ratio obtained from the stress (σ_c) vs. deformation (ε_c) curve in the elastic region (Eq. 4). These samples presented an equilibrium moisture content mean of 12,82 % with a standard deviation of 0,75 %.

$$\sigma_c = \frac{F_{max}\ (N)}{A\ (mm^2)} \tag{3}$$

$$E_c = \frac{\sigma_c\ (MPa)}{\varepsilon_c\ (\%)} \tag{4}$$

Shear Parallel to Grain

The shear parallel to grain tests were performed to determine shear strength. Specimens with the dimensions of 50 x 50 x 63 mm³ (radial × tangential × longitudinal directions) were cut and tested according to ASTM D143 (ASTM 2021). A shear tool was used to provide a 3 mm offset between the inner edge of the supporting surface and the plane of its adjacent edge. The load was continuously applied at a rate of motion of 0,6 mm/min in an Emic DL 30000 universal testing machine. The ultimate shear stress

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was calculated according to Eq. 5. Using the maximum load (F_{max}) and the shear cross-section (A). These samples presented an equilibrium moisture content mean of 13,04 % with a standard deviation of 1,22 %.

$$\sigma_s=\frac{F_{max}\ (N)}{A\ (mm^2)} \tag{5}$$

Janka hardness

Janka hardness was determined on the tangential plane of 50 x 50 x 150 mm³ (radial × tangential × longitudinal directions) specimens. A hemisphere with 11,3 mm in diameter penetrated 5,65 mm on the tangential plane of samples. The hardness is considered as the ratio between the maximum load and the indentation area, as shown in Eq. 6. These tests were carried out at a rate of motion of the movable crosshead of 6 mm/min in an Emic DL 30000 universal testing machine. Two penetrations were performed for each sample, which were far enough from the sample edges to prevent splitting or chipping, according to ASTM D143 (ASTM 2021). These samples presented an equilibrium moisture content mean of 13,85 % with a standard deviation of 1,33 %.

$$\text{Hardness} = \frac{F_{max}\ (N)}{A\ (mm^2)} \tag{6}$$

Monte Carlo simulation

Data normality was verified using Shapiro-Wilk tests and sampling sufficiency to infer mechanical properties was performed through confidence intervals. This method was similar to some previous studies on anatomy, ecology, limnology, and phytotechny , Storck *et al*. 2012). The sample sizes (n= 54) and confidence levels ($1 - \alpha = 0,95$) were fixed in all mechanical tests considered. In the simulation procedure, 30 sample sizes (from 2 to 30) were designed for each property. After that, 2000 resampling with replacements from the pseudo-population of the original 54 data were done for each sample size. Minimum, 2,5th percentile, mean, 97,5th percentile, and maximum were

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determined and the confidence level of each sample size was equal to the difference between the 97,5th percentile and 2,5th percentile. Sampling sufficiency was considered as the lowest sample size in which the amplitude of the confidence interval was lower than 20 % of its respective mean since this is an adequate variation range based on similar studies for the determination of mechanical properties of wood. These statistical analyses were performed using three software packages: Statgraphics 19® (for the descriptive statistics, R Development Core Team (for the Monte Carlo simulation), and Microsoft Excel (for graphics). The simplified flowchart of the used Monte Carlo simulation algorithm is presented in Figure 2.

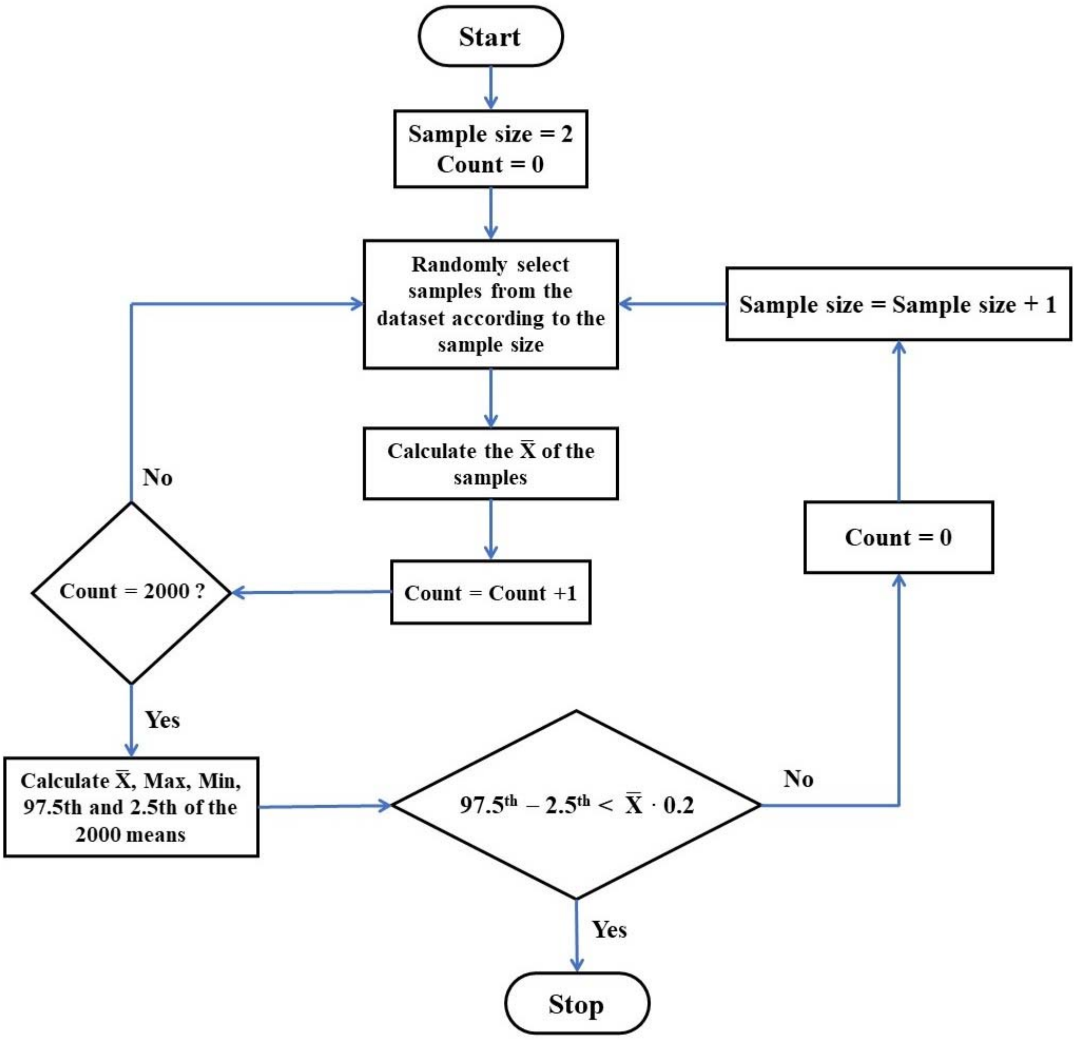

Figure 2: Simplified flowchart of the used Monte Carlo simulation algorithm.

RESULTS AND DISCUSSION

Mechanical test results

Table 1 shows descriptive results of the collected data from mechanical tests. All the mechanical properties presented small values of standard deviations than those presented in the literature, which is probably due to the large sample sizes (n= 54) selected in the present study.

Table 1: Descriptive statistics of obtained results for the original sample size (n=54).

	Bending modulus	**Bending strength**	**Compressive modulus**	**Compressive strength**	**Shear strength**	**Hardness**
Maximum (MPa)	12699,381	90,271	17413,400	52,907	10,560	48,980
97,5th (MPa)	11803,210	88,840	16786,984	51,396	10,363	47,271
X̅ (MPa)	9468,585	72,462	11634,509	41,623	7,508	37,613
sd (MPa)	1197,680	9,560	2509,308	4,695	1,420	4,999
CoV (%)	12,649	13,193	21,568	11,280	18,918	13,292
2,5th (MPa)	7525,300	56,209	7449,340	33,263	5,043	28,444
Minimum (MPa)	7443,974	53,974	7378,900	32,790	4,350	27,600
Where: X̅= mean; sd= standard deviation; CoV= coefficient of variation.						

The CoV values are 12,54 % and 13,05 % for bending modulus and bending strength, respectively. These CoV values are, in general, lower than those obtained for each of the 14 species studied by Carrillo *et al.* (2011), who used 30 samples for each species. The variations in the bending test may be attributed to the typical behaviour of small samples in a bending test, which is marked by an initial yielding on the compression side, accompanied by the enlargement of the compression zone, and then the neutral surface shifts toward the tensile side of the sample. As the tensile stress continues to increase, the ultimate stress is reached when a brittle failure at the tensile side occurs (Green 2001). However, according to Crespo *et al.* (2020), some specimens submitted to

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bending tests may present a brittle failure due to small grain deviations without yielding at the compression side.

The highest and the lowest CoV values were obtained for the compression parallel to grain results. Compressive modulus and compressive strength reached CoV values of 17,91 % and 12,15 %, respectively. This corroborates Crespo *et al.* (2020), who studied an *Eucalyptus globulus* Labill wood by using a 20 samples and reported a higher CoV value for compressive modulus (29 %) compared to that associated with the compressive strength (18 %). According to Mohebby *et al*. (2014), the anisotropy of the wood ultrastructure is one of the main factors that influences compressive strength (parallel to grain). The crushing is the most common failure in compression tests and is usually characterized by folding of the cellulose microfibrils that may begin in low stress levels (Crespo *et al.* 2020). As the stress levels increase, the folding takes place at the cell wall level and leads to the failure of the specimen (Green 2001).

Shear strength presented a variability similar to the other strength properties (bending and compression), with a CoV of 12,97 %. This value is lower than those obtained by authors that used smaller sample sizes than those adopted in this research. Crespo *et. al* (2020) used a sample size of 44 for *Eucalyptus globulus* wood and obtained a CoV of 19 %. Also, Trockenbrodt *et al*. (1999) achieved a 17 % CoV for 9 samples of *Azadirachta excelsa* wood. Moreover, attempts to obtain shear failure perpendicular to the grain according to certain standards, may result in other failure modes, such as compression perpendicular to the grain. According to Kretschmann (1991), that may occur because of the notch used to prevent the rotation of the rectangular prismatic specimens proposed in the ASTM D143 (ASTM 2021), which causes an asymmetry in the application of the force that promotes shear, inducing the appearance of normal tensile stresses and normal compression in wood fibres. Finally, hardness data had a CoV value

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of 12,67 %, which is smaller than those achieved by Salca and Hiziroglu (2014) for the 4 species studied, who used a 5 sample size for each species.

Determination of sample sizes

The results in this section present the sample size for each mechanical test, which were obtained by the Monte Carlo methodology previously described. During the determination of sample sufficiency, increases in small sample sizes cause a large decrease in the standard error, while the same increase in large sample sizes may not greatly affect the standard error (DePatta-Pillar 1998). Based on that, the determination of sample size is crucial to achieve reliable results. Furthermore, this knowledge is extremely necessary since obtaining experimental data is sometimes constrained by the availability of raw materials and time.

Figure 1 displays histograms, in which the number of classes (k) was determined according to the empirical rule ($k = \sqrt{n}$), where n is the sample size. Except for the shear strength data, the p-values were above the significance level (0,05), which indicates that all the obtained data conformed to the normal distribution according to the performed Shapiro-Wilk tests. This is a valuable feature since most Monte Carlo simulations may yield inaccurate results for non-parametric data, which also depends on the resampling method.

As shown in Figure 3, the data of elasticity modulus in bending and axial compression follow the normal distribution, resulting in small sample sizes for sufficiency (9 and 12 samples, respectively). Despite this, many researchers adopted lower sample sizes than those achieved for sampling sufficiency in this research. For instance, Zhang *et al*. (2015) used 8 specimens of *Eucalyptus regnans* wood, while Kothiyal (2014) used 4 to 6 samples to study intra clonal variations of *Eucalyptus tereticornis*. However, it is also possible to find researches with much larger samples sizes

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than those indicated by Monte Carlo in this study as sample sufficiency. In this sense, Taghiyari (2011) used a 60 sample size of *Populus nigra* wood and Ferreira *et al*. (2019) used 30 specimens of *Eucalyptus* clones.

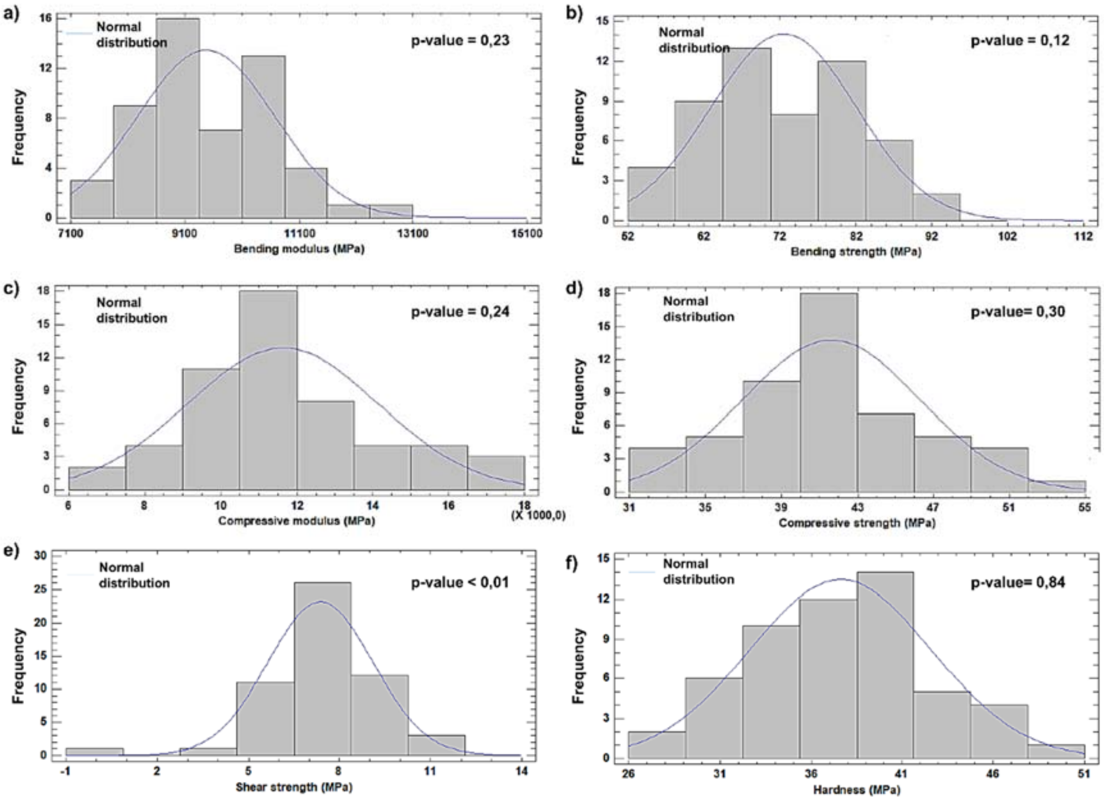

Figure 3: Histograms and bell curves for the obtained data.

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The sampling sufficiency results for the mechanical characteristics of the *Eucalyptus* woods are shown in Figure 4. In general, the sample sufficiency for the test was given in samples size wherein a low convergence rate was still visualized. This can be related to the confidence level set as stop criteria (20 %), in which a lower confidence level, the sample sufficiency would be given at a much higher sample size, where the curves would decrease monotonically. We added this statement in the main text. Regarding the wood stiffness, bending modulus reached sample sufficiency in 9 sample size (Fig. 4a), while compressive modulus obtained sample sufficiency in 12 sample size (Fig. 4c). The larger sampling sufficiency presented by the compressive modulus is attributed to its higher CoV value when compared to the bending modulus.

Regarding wood strength, in both bending and compressive strength, the sample sufficiency reached 7 samples (Figs. 4b, 4d). This sample size is larger than the sizes adopted in some recent searches, such as Ghorbani Kookandeh *et al*. (2014) (n = 5) and Mohebby *et al*. (2014) (n = 6) and lower than those adopted by Crespo *et al*. (2020) (n = 22).

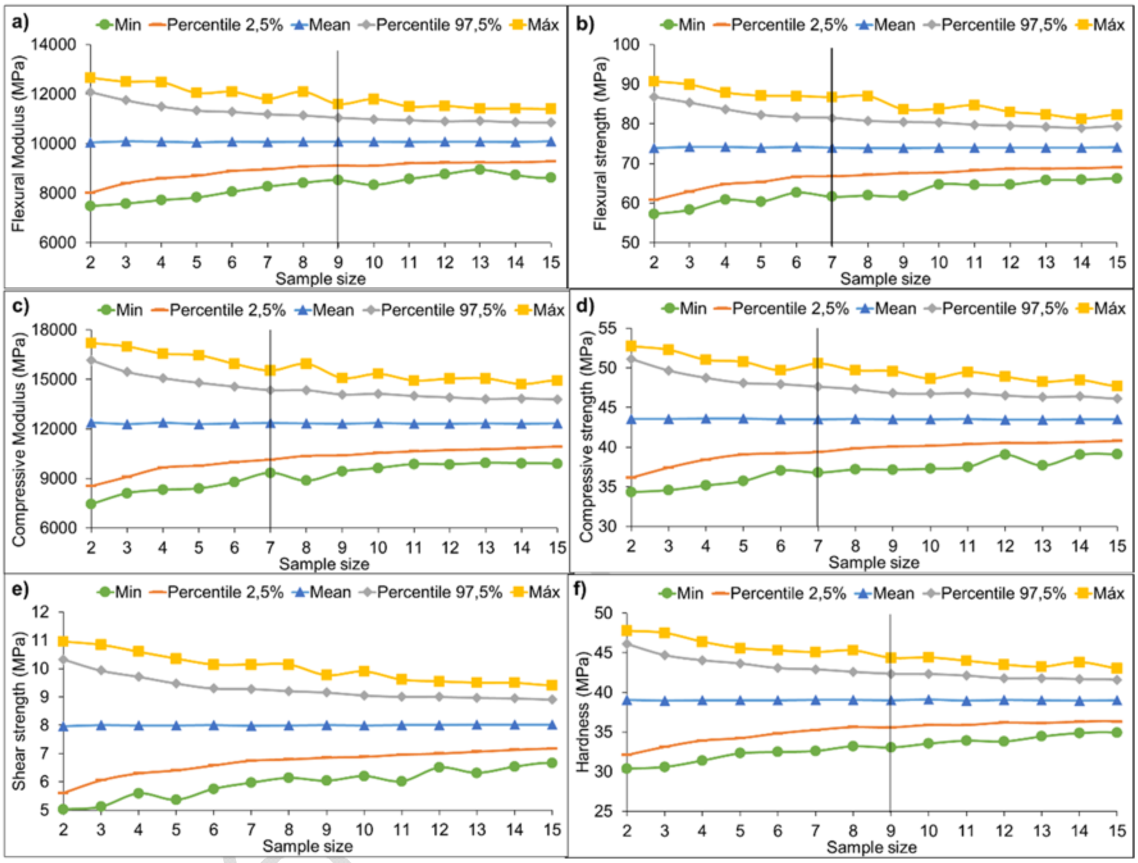

Figure 4: Monte Carlo confidence intervals for means for bending modulus (a), bending strength (b), compressive modulus (c), compressive strength (d), shear modulus (e), and hardness (f).

A sample sufficiency of 25 was determined for shear strength (Fig. 4e), which can be attributed to the low normality of the data obtained for this property (Fig. 3). This probably increased the sample size to this number of specimens, which is unusual based on recent researches. Although the CoV associated with the shear strength test did not differ from the other tests (12,97 %), it yielded a much larger sample size than the other properties, this indicates that the determination of the sampling sufficiency by the applied resampling method is more related to data normality than that of CoV values. Finally, hardness achieved sampling sufficiency in a 9 sample size, a higher size than those adopted by Salca and Hiziroglu (2014), who used 5 specimens per group in their study.

Small samples are usually related to methodologies involving the utilization of wood associated with expensive chemicals or laborious treatments, thus is understandable that the utilization of larger samples sizes is sometimes unfeasible (Ghorbani-Kookandeh *et al*. 2014, Mohebby *et al*. 2014). However, the variation in the results could be related to the inherent variation of the wood instead of the studied treatment is extremely important.

CONCLUSIONS

The scope of the present study provides for a preliminary elucidation on the sampling sufficiency for some mechanical tests according to the ASTM D143, three points static bending, compression parallel to the fibres, hardness, and shear. Thus, *Eucalyptus* clones were studied regarding those properties and all obtained data conformed to the normal distribution, which enabled the implementation of Monte Carlo simulations. The analysis of the mechanical properties data showed that the wood stiffness presents a smaller variability than its strength, which indicates that the elastic region of the load *vs.* deformation curve can be studied with fewer samples than the modulus of rupture. Finally, the sampling sufficiency achieved in this research is above those sample sizes used in many recent studies, which indicates that a greater scientific effort should be focused on further conclusive studies for other properties of interest, which are also foreseen in ASTM D143, such as tensile perpendicular and parallel to the fibres and other properties. Finally, the sample sufficiency should also be determined for different species of wood, as the statistical variability strongly depends on this factor.

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