

TWO METHODS OF DRYING EUCALYPTUS WOOD AFTER IMPREGNATION WITH SILVER NANOPARTICLES

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ABSTRACT - In view of its importance in the forestry industry and the commercialization of wood, there is a clear need to implement technologies that provide better quality wood and improved drying processes. Therefore, this study aimed to evaluate two methods of drying *Eucalyptus grandis* W. Hill wood after impregnation with silver nanoparticles. For this purpose, logs of this species were used, which, after making the specimens and impregnating silver nanoparticles at concentrations of 40 and 100 mg L⁻¹, were subjected to drying in an air-conditioned chamber with an average temperature of 20°C and relative humidity of 65% and a laboratory oven with forced circulation at a constant temperature of 60°C, for subsequent analysis of moisture content, drying rate, and defects. The results showed a greater loss of moisture in the pieces impregnated with 40 mg L⁻¹, dried in an oven and air-conditioned chamber, and a higher drying rate in the oven. In addition, there was a higher incidence of defects in wood impregnated with 100 mg L⁻¹ and dried in an oven. The specific mass obtained average values similar to those referenced in the literature for this species. Based on the results of this study, it was possible to conclude that the impregnation of Ag NP's brings benefits in terms of the drying rate, allowing for a greater loss of moisture from the wood. In addition, the treatment reduces warping defects. These effects are found mainly in wood impregnated with 100 mg L⁻¹.

Keywords: *Eucalyptus grandis* W. Hill, colloidal silver, wood resistance.

DOIS MÉTODOS DE SECAGEM DE MADEIRA DE EUCALIPTO APÓS IMPREGNAÇÃO COM NANOPARTÍCULAS DE PRATA

RESUMO - Tendo em vista sua importância na indústria florestal e de comercialização da madeira, é notória a necessidade de implantação de tecnologias que proporcionem uma madeira de melhor qualidade e processos de secagem aprimorados. Por conta disso, o objetivo deste estudo é avaliar dois métodos de secagem da madeira de *Eucalyptus grandis* W. Hill após impregnação de nanopartículas de prata. Para tanto, foram utilizadas toras dessa espécie, o qual após confecção dos corpos de prova e a impregnação nanopartículas de prata, nas concentrações de 40 e 100 mg L⁻¹, as mesmas foram submetidas à secagem em câmara climatizada, com temperatura média de 20°C e umidade relativa 65% e estufa laboratorial com circulação forçada com temperatura constante de 60°C, para posterior análises de teor de umidade, taxa de secagem e defeitos. Os resultados mostraram que ocorreu maior perda de umidade naquelas peças com impregnação de 40 mg L⁻¹, com secagem em estufa e câmara climatizada, assim como, uma maior taxa de secagem em estufa. Além disso, ocorreu maior incidência de defeitos naquelas madeiras impregnadas com 100 mg L⁻¹ e secas em estufa. A massa específica obteve valores médios semelhantes aos referenciados na literatura para a espécie trabalhada. A partir dos resultados da presente pesquisa, foi possível concluir que a impregnação de NP's Ag traz benefícios quanto à taxa de secagem, possibilitando uma maior perda de umidade da madeira. Além disso, o tratamento proporciona uma diminuição dos defeitos de empenamentos. Tais efeitos são encontrados principalmente naquelas madeiras com impregnação de 100 mg L⁻¹.

Palavras-chave: *Eucalyptus grandis* W. Hill, prata coloidal, resistência da madeira.

INTRODUCTION

Today's timber market seeks to optimize quality and yield, boosting profits and reducing costs. In view of this, forestry techniques such as drying are necessary to add value to wood. Specifically, drying wood offers the possibility of reaching an equilibrium moisture content, so that the variation in moisture input and output is minimal, thus achieving the quality of the final product. Improving wood quality and optimizing industrial splitting and drying

processes are fundamental for forest-based industries, as they can provide knowledge of new splitting and drying techniques, especially when it comes to drying refractory wood such as *Eucalyptus*.

Conceptually, *Eucalyptus* wood is difficult to dry. The behavior of these woods during water removal is also unique, and different from *Pinus* or the native species traditionally used. Because of this, many industries face problems with the quality of the dried pieces or prefer to use

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other species as their main raw material (ZEN et al., 2019). Therefore, for the species of this genus to be put to better use, an appropriate drying method is required depending on their purpose. In this sense, conventional drying stands out because it is faster, with control of the drying rate and is also able to accelerate the loss of moisture from the wood with a lower incidence of possible defects, generating a higher quality product (REZENDE et al., 2015) and, consequently, lower costs.

Open air drying can be considered more efficient for reducing wood moisture, especially in the initial phase (ZANUNCIO et al., 2014), where water loss occurs at an accelerated rate. However, the method has the disadvantage of requiring more time than the others, until the moisture content of the wood passes the fiber saturation point (FSP), reaching the desired moisture content (TALGATTI et al., 2018). According to the same authors, outdoor installation is simple and cost-effective, but some precautions must be taken, such as: keeping the piles in a dry and ventilated place, preferably in sunny locations and, when possible, covering the piles to prevent sunlight and rain from falling on them.

In order to obtain better results, there are many technologies that can be used to improve the use of wood, however, the technology of impregnating wood with silver nanoparticles prior to the drying process can give the material relevant characteristics, such as serving as a catalyst in the drying of wood (DASHTI et al., 2012). Evaluating the drying process is essential in order to provide subsidies to minimize defects, reduce drying time and improve wood quality. The aim of this study was to evaluate two methods of drying eucalyptus wood after impregnation with silver nanoparticles.

MATERIAL AND METHODS

The eucalyptus trees (*Eucalyptus grandis* W. Hill) used in the study came from experimental plantations located in the city of Santa Maria (RS). For this study, three trees were felled from a homogeneous plantation with a spacing of 3 x 2 m. Only the first 1.50 m log was used, after 0.10 m above ground level. Subsequently, the primary splitting of this material was carried out, which generated a central plank 0.12 m thick, from which the central part of the pith was discarded, so that only the sapwood was used. The test specimens were then made, measuring 0.025 x 0.10 x 0.40 m in thickness, width and length respectively.

The silver nanoparticles were obtained commercially and selected in two concentrations, 40 and 100 mg L⁻¹. This solution contains colloidal silver with nanometric sizes and is therefore considered to be a colloid with exceptional stability and purity. After the specimens had been prepared, the silver nanoparticles were impregnated into the wood using a laboratory autoclave with a capacity of approximately 6 liters, using the full cell method (i.e. vacuum combined with pressure). Initially, a counterpressure was applied for 15 min. and then the liquid containing the silver nanoparticles (Ag NPs) was transferred to the inside of the autoclave and a pressure of 8 bar was applied for 1 h.

The experiment was divided into two methods (air-conditioned chamber and laboratory oven) and three treatments (control sample, 40 and 100 mg L⁻¹). Each treatment was repeated 6 times, totaling 36 specimens. For the first method, after impregnating the Ag NP's, the specimens were dried in the climate chamber of the Forest Products Laboratory (LPF) under conditions of 20°C ± 2 temperature and 65 ± 2% relative humidity, simulating drying in the open air of the Santa Maria (RS) region, for 962 h. The material was distributed on metal shelves until it reached hygroscopic equilibrium, at a moisture content of approximately 20%.

Controlled drying was carried out in a Marconi model MA 035 laboratory oven with air circulation and renewal, using a constant temperature of 60 ± 2°C. During the drying process, every 2 h, the weight of all the samples and their respective dimensions (thickness, width and length) were taken. As soon as the boards reached a constant moisture content of approximately 20%, the measurement was stopped and the drying time was 109 h.

The method used to determine the moisture content of the wood was gravimetric. To do this, all the 0.40 m boards were selected and the first 0.025 m were removed from each end, in order to minimize the influence of the tops drying out. Subsequently, two more 0.025 m specimens were removed as a control sample. This left 0.30 m specimens as the final material for drying analysis and mechanical tests.

During wood drying, the initial moisture content of each control sample was calculated using the arithmetic mean of the two sections (A and B). The moisture content of each section was determined by Equation 1, using a natural convection oven with a constant temperature of 103 ± 2°C.

$$Tui = \frac{(Pu - Ps)}{Ps} \times 100 \quad (\text{Equation 1})$$

Where:

Tui = Initial moisture content (%),

Pu = Wet sample weight (g) e

Ps = Dry weight of the sample (g).

Two control samples were placed in each wood pile in different locations that were easy to remove and replace, so as to allow a real and representative measurement of their moisture content. The moisture content of each pile was calculated using Equation 2:

$$Tua = \frac{(Pa(TUi + 100))}{Pi - 100} \quad (\text{Equation 2})$$

Where:

Tua = Current moisture content (%),

Pa = Current sample weight (g)

Pi = Wet weight of sample (g) e

TUi = Initial moisture content (%).

The pieces remained in the piles until they reached an average moisture content of approximately 17.8%, which

is considered to be the equilibrium moisture content for wood in the central region of Rio Grande do Sul at this time of year, from June to August. In order to determine the drying rate, it was necessary to use Equation 3, described by Santini (1980).

$$TS = \frac{Q_{ae}}{Ae \times T} \quad (\text{Equation 3})$$

Where:

Ts = Drying rate (g cm⁻² h),

Qae = Amount of water evaporated (g),

Ae = Surface area of the part (cm²) e

T = Elapsed drying time (h).

With the help of a circular saw, de-bushing machine, trimming machine and degrossing machine, straight and flat specimens were made, i.e. without defects, so that defects could be observed after the kiln and air-conditioned chamber drying processes. When the boards were dried, it was possible to observe the appearance of curling. To analyze this defect, we used the warping classes of NBR 14.806 for eucalyptus sawn timber (ABNT, 2002). The defect found in this study was measured using a digital caliper with a precision of 0.01 mm.

A completely randomized design was used, with a 2x3 factorial, presenting the experiment with 2 factors (air-

conditioned chamber and greenhouse), each with 3 levels (control, 40 and 100 mg L⁻¹). In addition, Bartlett and Shapiro-Wilk checked the homoscedasticity of the variance ($p > 0.05$) and the normality of the distribution of the experimental errors ($p > 0.05$), respectively. In cases where there was no significant difference, the data had to be transformed using the square root.

For data with significant values, it was necessary to perform the Tukey test, with a 5% probability of error, in order to identify these differences. Normality and homoscedasticity were checked using Past statistical software and the test of means with Rstudio Team software (2020). The procedures recommended by standard NBR 14.806 (ABNT, 2002) were used to assess post-drying defects.

RESULTS AND DISCUSSION

Figure 1 shows the drying curves of the kiln-dried wood. Relating the moisture content to the drying time, it can be seen that the curve shows the normal characteristics of drying curves, with the control showing less loss of moisture content from 28 h onwards compared to the wood impregnated with Ag NPs. This is because the nano-metals are spread throughout the cell walls, vessels and punctuations, facilitating better heat transfer to the interior of the wood. Thus, the TUF of the control was higher than that of the wood impregnated with silver nanoparticles.

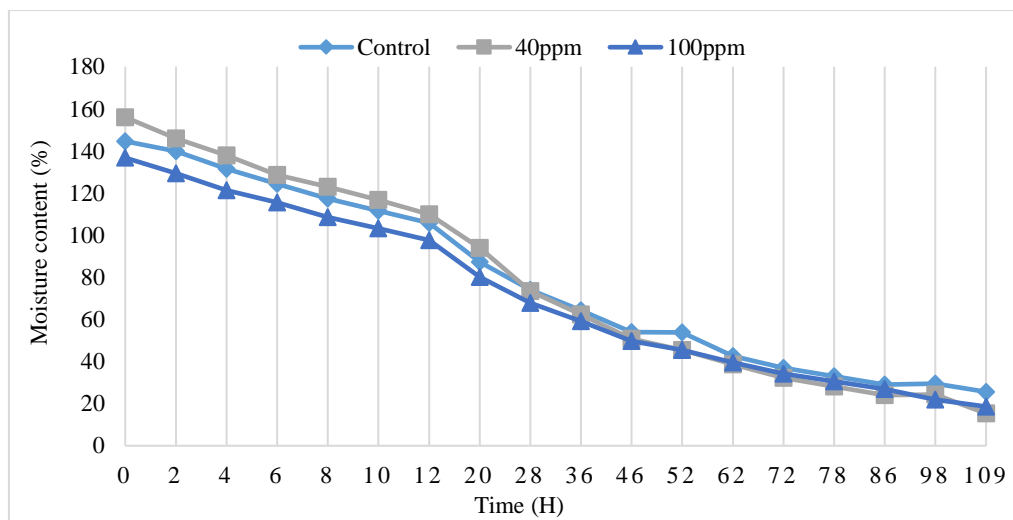


FIGURA 1 - Oven-drying curve of the samples as a function of moisture content (%) and elapsed time (h).

Analyzing Figure 1, we can see from the moisture curve for removing water from the wood that it took approximately 78 h for the FSP to be reached. A further 31 h were needed to remove the impregnation water, totaling 109 h of drying, reaching approximately 19% moisture content.

Water loss from wood occurs differently depending on the moisture content of the piece, or whether it is above or below the fiber saturation point (FSP). Therefore, in order to remove free water from the wood by capillarity, some care must be taken at the start of the process for species with low permeability, such as the

Eucalyptus genus, so as to minimize the occurrence of defects (GALVÃO; JANKOWSKY, 1985).

The average initial moisture content, h of drying and final moisture content of the laboratory oven drying are shown in Table 1. Analysis of the average moisture content of the treatments shows that there was a 125.94% reduction in moisture during the 109 h of drying. It can also be seen that the highest initial average (155.98%) and the lowest average at the end (15.32%) were obtained when the 40 mg L⁻¹ concentration was used, which showed the greatest loss of moisture during the 109 h of drying.

The results obtained for the initial moisture content are similar to those observed in other studies for the *Eucalyptus grandis* W. Hill species, using vaporization (REZENDE et al., 2015). Moisture losses in relation to the drying time of the wood are higher than those found by authors such as Eleotério et al. (2015) and Duarte et al. (2015), who dried the same species in a conventional oven, in a similar oven.

Figure 2 illustrates the drying curve with the same treatments mentioned above, but for drying in an air-

conditioned chamber. This method is similar to open-air drying, as it uses an air-conditioned room with the average temperature and relative humidity of the city in question. Together with the temperature and relative humidity curve of the air-conditioned chamber, it can be seen that up to 98 h, i.e. the 5th day of drying, when the average moisture content was 112% TU, there was a greater distance between the measurements. As a result, there was a greater gap in the drying curve.

TABLE 1 - Initial moisture content (TU_i), final moisture content (TU_f) and oven drying time.

Concentrations of NP's Ag (mg L ⁻¹)	Initial moisture content (%)	Elapsed time of drying (h)	Final moisture content (%)
Control	144.58	109	25.60
40	155.98	109	15.32
100	136.74	109	18.58
Averages	145.77	-	19.83

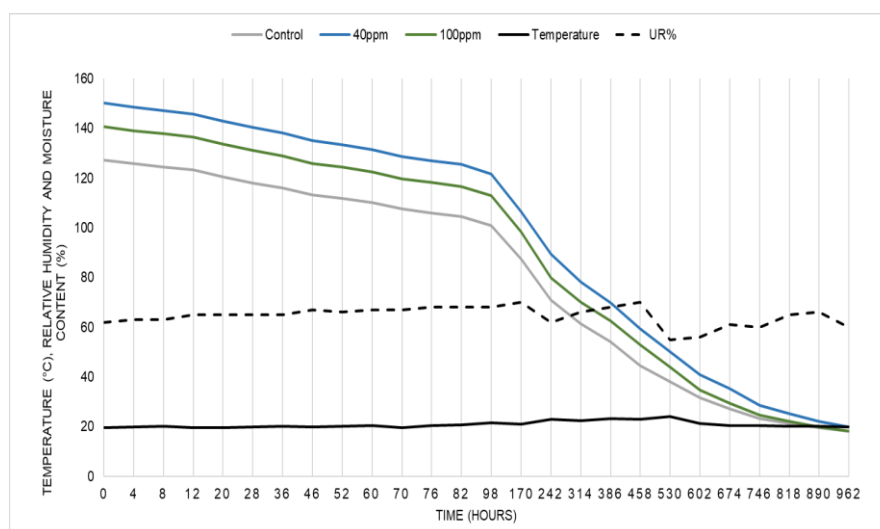


FIGURA 2 - Drying curve of the samples in an climatized chamber, as a function of initial moisture content (TU_i) and elapsed time (h).

We can also see that, before FSP, the curve showed a linear characteristic with capillary water, with the drying rate being almost constant. Near 30%, in the transition to hygroscopic water, the curve decreased. After reaching the fiber saturation point (FSP), hygroscopic drying predominated and the curve had a lower slope, represented by the drying rate. In this drying condition, the control had a lower TU_f and greater total moisture loss than the pieces impregnated with silver nanoparticles.

França et al. (2019), in a study using *Eucalyptus grandis* W. Hill x *E. urophylla* S.T. Blake boards, presented a drying curve similar to the one seen in this work, in which the average FSP of the experiment was reached after 446 h of drying. In another study, Mangini et al. (2023), using *Eucalyptus grandis* W. Hill x *E. urophylla* S.T. Blake, observed that a transition range in drying rate was reached

between the species studied, at 312 h. In addition, Zen et al. (2019), studying *Eucalyptus* spp., found that 936 h were required to reach the FSP. In the present study, FSP was reached in 674 h, which is slightly longer than the study by França et al. (2019) during the winter season. This difference in time can be explained by the fact that the pieces were dried in an air-conditioned chamber, with an average temperature of 20°C and relative humidity of 65%, rather than in the open air, as in these studies.

The average initial and final moisture content, together with the drying times, are shown in Table 2. When comparing the total moisture loss of the wood (TU_i - TU_f), the values obtained by the air-conditioned chamber (120.79%) are lower than those presented in the laboratory oven (125.94%), exemplifying its slower drying process.

TABELA 2 - Initial moisture content (TU_i), final moisture content (TU_f) and drying time in an air-conditioned chamber.

Concentrations of NP's Ag (mg L ⁻¹)	Initial moisture content (%)	Elapsed time of drying (h)	Final moisture content (%)
Controle	127.38	962	18.03
40	150.34	962	19.98
100	140.85	962	18.20
Averages	139.53	-	18.74

In a similar study on open-air drying, Talgatti et al. (2018) found average initial moisture content of 107% and average final moisture content of 18% for *Eucalyptus grandis* W. Hill wood. França et al. (2019) carried out open-air drying during the fall in the state of Espírito Santo and obtained an average initial moisture content of 83.56% and an average final moisture content of 15.95%, in 3,528 h. In this study, the average initial moisture content was 139.53% and the average final moisture content was 18.74% in 962 h, obtained at an average temperature of 20°C and a relative humidity of 65% in an acclimatized chamber. It should also be noted that the concentration of 40 mg L⁻¹ showed the greatest loss of moisture (130.36%) during the 962 h of drying, followed by the concentration of 100 mg L⁻¹

(122.65%), with TUi and TUF slightly higher than those of the control.

Table 3 shows the drying rates above and below the FSP, as well as the overall average for laboratory oven drying. It can be seen that the wood with a drying rate above the FSP, with 40 mg L⁻¹ impregnation, differed significantly from the control and that with 100 mg L⁻¹ impregnation, showing that there was a greater loss of moisture in the wood compared to the others. When compared to kiln drying, with moisture content below FSP, it was concluded that there was no significant difference, however, it was found that the control showed greater moisture loss at this stage.

TABELA 3 - Drying rate of *Eucalyptus grandis* W. Hill wood in a laboratory oven.

Concentrations of NP's Ag (mg L ⁻¹)	Drying rate (g cm ⁻¹ h) 10 ³		
	Above the FPS	below the FPS	Averages
Control	2.4775 b*	0.2025 a	2.0761 b
40	3.5447 a	0.1945 a	2.9535 a
100	2.6135 b	0.1991 a	2.1874 b

*Means followed by the same letter in the column do not differ from each other, by the Tukey test, at 5% probability of error.

The average values of the kiln drying rate ranged from 2.07 to 2.95 (g cm⁻² h) 10³, with the piece impregnated with 40 mg L⁻¹ having the highest drying rate of 2.95 (g cm⁻² h) 10³, differing significantly from the others. Zen et al. (2019) reported that the average value in conventional drying of green wood up to 30% (capillary water) was 0.7468%, from 30% to 9% (impregnation water) was 0.1715% and green, up to 9%, was 0.2845%, showing that there was a reduction in moisture loss from the piece when below the FPS. Luis et al. (2017) found a reduction in the hygroscopic range of the drying rate when studying the

effect of vaporizing eucalyptus wood for two treatments.

As for drying in an air-conditioned chamber, Table 4 shows that the average drying rate values ranged from 0.2284 to 0.3172 (g cm⁻² h) 10³, with no significant difference. It can also be seen that the wood treated with Ag NP's impregnated with 40 mg L⁻¹ and below the FPS, although showing no significant difference from the other treatments, showed greater moisture loss compared to the others; however, the highest overall average occurred with the concentration of 100 mg L⁻¹.

TABELA 4 - Drying rate of *Eucalyptus grandis* W. Hill wood in an acclimatized chamber.

Concentrations of NP's Ag (mg L ⁻¹)	Drying rate (g cm ⁻¹ h) 10 ³		
	Above the FPS	below the FPS	Averages
Control	0.3749 a*	0.0129 a	0.2284 a
40	0.2784 a	0.0213 a	0.2373 a
100	0.2694 a	0.0142 a	0.3172 a

*Means followed by the same letter in the column do not differ from each other, by the Tukey test, at 5% probability of error.

According to Zen et al. (2019), in a similar analysis using *Eucalyptus* spp. wood, the drying rates obtained under the moisture content conditions of green up to 30%, 30% up to 22% and green up to 22% were 1.5, 0.25 and 1.00 (g cm⁻² h) 10³, respectively, lower than those found in this study. Batista et al. (2015) also found average values for drying *Eucalyptus* spp. in an air-conditioned chamber were 0.128 to 0.157 (g cm⁻² h) 10³, lower than those found in this

study, and the author classified the drying of his research as slow. Talgatti et al. (2018), using a species of *Eucalyptus grandis* W. Hill, dried in the open air in the region of Santa Maria - RS and at a similar temperature and relative humidity to this study, obtained an average drying rate of 0.1977 (g cm⁻² h) 10³.

After the experiment, the pieces showed only the defects of curling and twisting. From this, we can see in

Figure 3 that the boards dried in an air-conditioned chamber and impregnated with nanoparticles (40 mg L^{-1} and 100 mg L^{-1}) showed a reduction in defect values compared to the control of approximately 0.6 mm, although this did not show a significant difference.

Kiln drying saw an increase in defect values compared to drying in an air-conditioned chamber. It can be seen that the timber impregnated with Ag NP's at 40 mg L^{-1}

and 100 mg L^{-1} had 2.61 and 1.76 mm defects respectively, lower than the control, which had 3.30 mm. However, there was no significant difference between the two. In general, the boards dried in an air-conditioned chamber showed the best results, and were classified as medium magnitude, according to NBR 14.806 (ABNT, 2002). However, kiln drying showed values of more than 4 mm and the boards were classified as strong.

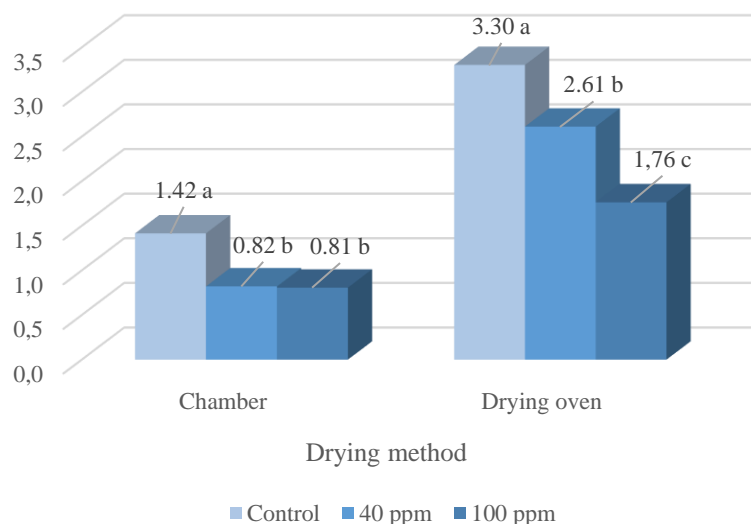


FIGURA 3 - Wood curling defect of *Eucalyptus grandis* W. Hill. *Means followed by the same letter in the column do not differ according to Tukey's test at 5% probability of error.

According to research by Batista et al. (2015), the *Eucalyptus grandis* W. Hill species is prone to high values of curl, with the author finding 60.49 mm, a value higher than those obtained in this research. In addition, Batista et al. (2010) explain that the wood of this species can be very unstable in size, causing this defect. Also, according to Caixeta et al. (2002) in a study of *Eucalyptus* between 13 and 17 years old, after assessing the quality of the wood subjected to drying in the open air, average values for curling were 3.12 mm, and the curling found by the aforementioned author was similar to the control. In addition, Stangerlin et al. (2009) reported that curling can result from the difference between transverse contractions that can occur when only one side of the board is exposed to drying. Dittmann et al. (2017) states that eucalyptus wood is susceptible to the defect of curling during drying, especially if it is carried out under severe conditions and at high temperatures.

The presence of twisting in the pieces was observed, caused by the presence of spiral grain, which is characteristic of wood of the *Eucalyptus* sp. genus, i.e. a slight inclination in the samples. Because it is a very serious

defect, twisting is not accepted and, for this reason, only the boards that showed this defect were quantified in percentage and there was no measurement.

Spiral grain is defined as a deviation of the fibers, being the angle between the longitudinal elements of the wood and the axis of the trunk, and can occur as a result of cutting the pieces or by natural existence. The presence of twisting can be caused by the spiral grain contained in trees, due to the orientation of the fibers out of alignment with the longitudinal axis of the wood. Some species of the *Eucalyptus* spp. genus are prone to this characteristic (PINKOWSKI et al., 2016).

Table 5 shows that the control with climatized chamber drying had the highest number of boards with twisting, followed by the one with 100 mg L^{-1} impregnation dried in a laboratory oven. According to Pinkowski et al. (2016), twisting is caused by a combination of different contractions and grain deviations, such as spiral grain. In addition, Freitas et al. (2016), reports that dimensional variation in the radial and tangential directions, differences between juvenile and adult wood, as well as the difference between early and late wood, help this warping to appear.

TABELA 5 - Percentages of boards with a twisting defect.

Cultivation environments	Concentrations of NP's Ag (mg L ⁻¹)		
	Control	40	100
Percentages of boards with a twisting defect			
Drying oven	33.33	33.33	66.66
Acclimatized chamber	83.33	50.00	33.33

During the course of this work, it was possible to see the importance and need for new studies relating wood drying to the impregnation of nanomaterials, always seeking to find methods that are more efficient and capable of reducing their environmental impact. In addition, exploring new approaches, such as the use of innovative technologies and the development of more sustainable methods, is essential to promote the continuous evolution of the sector.

It is also possible to see the importance of this line of research for Brazilian scientific development and for the development of the forestry sector, since the application of this innovative technology, using silver nanoparticle impregnation, results in important benefits for wood drying.

CONCLUSION

Based on the results of this research, it was possible to conclude that impregnation with Ag NPs has benefits in terms of drying rate, enabling greater loss of moisture from the wood. In addition, the treatment reduces warping defects. These effects are found mainly in wood impregnated with 100 mg L⁻¹.

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