








ORIGINAL ARTICLE

Effect of moisture and active ingredient concentration on CCA-C retention

Efeito da umidade e concentração de ingrediente ativo na retenção de CCA-C

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Abstract

The strategy of wood preservative treatment companies is to control the factors that influence retention, such as the concentration of the preservative solution and wood moisture, to reach values higher and closer to the standard minimum. The objective of this study was to reduce the consumption of Chromated Copper Arsenate type C (CCA-C) without affecting the quality of the treatment by determining the most efficient wood moisture interval for two genotypes of *Eucalyptus* sp. at different concentrations of active ingredients. A total of 54 fence posts of the species *E. cloeziana* and the hybrid *E. camaldulensis* x *E. urophylla* were treated. The pieces were randomly selected from three drying piles with different storage times in the yard (long, intermediate, and short). The active ingredient concentrations tested were 1.2, 1.4, and 1.6%. Retention analysis was performed to evaluate the quality of the preservative treatment. For both analyzed materials, the lowest and highest average retention values were observed in short and long storage piles and concentrations of 1.2% and 1.6% A.I. CCA-C, respectively. For *E. cloeziana*, only posts with a long storage time and treated with 1.6% of CCA-C were considered suitable for use in contact with the soil, according to the Brazilian standard, while for the clonal hybrid, posts with intermediate and long storage times, treated with 1.6% of CCA-C were considered suitable for use in contact with the soil. It was observed that CCA-C retention in the studied fence posts was directly influenced by the concentration of active ingredient in the preservative solution and by the storage time of the materials in the yard.

Keywords: Preservative treatment; Storage time in the yard; *Eucalyptus*.

Resumo

A estratégia das empresas de tratamento preservativo da madeira é controlar os fatores que influenciam a retenção, como a concentração da solução preservante e a umidade da madeira, para atingir valores mais altos e próximos do mínimo padrão. O objetivo deste trabalho foi reduzir o consumo de Arseniato de Cobre Cromatado tipo C (CCA-C) sem afetar a qualidade do tratamento, determinando o intervalo de umidade da madeira mais eficiente para dois genótipos de *Eucalyptus* sp. em diferentes concentrações de ingredientes ativos. Foram tratados 54 moirões da espécie *E. cloeziana* e do híbrido *E. camaldulensis* x *E. urophylla*. As peças foram selecionadas aleatoriamente a partir de três pilhas de secagem com diferentes tempos de estocagem no pátio (longo, intermediário e curto). As concentrações de ingrediente ativo testadas foram 1,2, 1,4 e 1,6%. A análise de retenção foi realizada para avaliar a qualidade do tratamento conservante. Para ambos os materiais analisados, os menores e os maiores valores médios de retenção foram observados nas pilhas de curto e longo tempo de estocagem e concentrações de 1,2% e 1,6% I.A. CCA-C, respectivamente. Para o *E. cloeziana*,

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apenas os moirões com longo tempo de estocagem e tratados com 1,6% de CCA-C foram considerados aptos para uso em contato com o solo de acordo com a norma brasileira, enquanto que para o híbrido clonal, moirões com tempo de estocagem intermediário e longo, tratados com 1,6% de CCA-C foram considerados aptos para uso em contato com o solo. Observou-se que a retenção de CCA-C nos moirões estudados foi influenciada diretamente pela concentração do princípio ativo na solução conservante e pelo tempo de armazenamento dos materiais no pátio.

Palavras-chave: Tratamento preservativo; Tempo de estocagem no pátio; *Eucalyptus*.

INTRODUCTION

Companies in the wood preservation sector must meet the norms and quality standards regulated by law for the commercialization of their products. The penetration and retention parameters of preservative products in the wood are the main factors in these regulations. Regarding penetration, the NBR 9480 standard (Associação Brasileira de Normas Técnicas, 2009) considers products to be suitable only when the sapwood or treatable zone is 100% impregnated. Regarding retention, the standard stipulates minimum values according to the degree of exposure of the intended use category for each product.

Chromated Copper Arsenate type C (CCA-C) is a product widely used for protecting wood against attack from xylophagous organisms such as fungi, insects, and marine agents. Despite the restriction of its use in some countries, it is still widely and exclusively industrially used in Brazil and can be used only by wood preservation plants registered with the Brazilian Institute of the Environment and Renewable Resources (Arantes et al., 2017).

CCA-C reacts with wood to form insoluble complexes; therefore, it presents good resistance to leaching and satisfactory results in protecting against xylophagous agents. However, it presents a high risk of environmental contamination and is classified by the Brazilian Institute of the Environment and Renewable Resources as Class 1.

According to Pavlak et al. (2013), CCA-C corresponds to 53% of the total variable cost for wood preservation companies. Thus, the strategy is to use less of this product and to provide satisfactory wood protection by working with retention values as close as possible to the minimum values required for each product sold. This is a constant challenge addressed in the yards of the treatment plants since there are many factors that directly affect the retention of the active ingredients in the wood.

Variables inherent to the raw wood material such as moisture, permeability, density, anatomical structure, and amount of heartwood are the main factors that influence the retention of preservatives in the wood (Vidal et al., 2015). Knowledge regarding specific ideal wood moisture values and operationally viable methodologies for their reliable determination in the yard are incomplete. The NBR 9480 standard (Associação Brasileira de Normas Técnicas, 2009) considers industrial preservative treatments performed on wood with moisture equal to or less than 35%, but not totally dry, to be ideal.

Regarding the preservative solution, the concentration of active ingredient is the main factor influencing retention, and usually, the higher the concentration of the product, the greater the retention obtained (Lopes et al., 2017; Nicholas & Siau, 1973). However, a better understanding of the other factors can facilitate a reduction in CCA-C consumption by obtaining satisfactory retentions with lower concentration in the treatments. Thus, it is necessary to verify the main factors that affect retention to better understand the process and to obtain the desired results.

Given the above, the objective of this study was to determine the most reliable moisture range of two genotypes of *Eucalyptus* to obtain efficient retention of CCA-C, using concentrations of preservatives lower than those used in the industry.

MATERIALS AND METHODS

Biological material

The material used was selected from the yard of a forestry company located in the city of Martinho Campos in the state of Minas Gerais, Brazil. A total of 54 fence posts

were selected. The posts had diameters between 8 and 10 cm and lengths of 2.20 m and were made from six-year-old trees of the species *Eucalyptus cloeziana* and of the clonal hybrid *Eucalyptus camaldulensis* x *Eucalyptus urophylla*.

Fence posts were randomly selected from piles that had been stored for different lengths of time in the yard to obtain moisture heterogeneity (Table 1). The wood stored for up to 20 days corresponded to the short storage time. Wood stored between 20 days and one year corresponded to the intermediate storage time. Wood with a storage time greater than one year corresponded to the long storage time. The fence posts that had been in the yard for more than one year were fit for preservative treatment; they had not been deteriorated by biotic agents due to the environmental conditions of the storage yard, which included high temperature and low relative humidity. Nine posts were selected for each storage time and for each genetic material.

Table 1. Variation in the moisture of the genetic materials at each storage time.

Genetic material	Variation in moisture at each storage time (%)		
	Short	Intermediate	Long
<i>E. cloeziana</i>	36.18–44.58	27.26–34.89	13.76–18.64
Clonal hybrid	52.60–64.86	27.45–48.64	13.03–23.71

To evaluate the influence of wood moisture and active ingredient concentration on treatment quality, the fence posts were subdivided into three treatment concentrations: 1.2%, 1.4%, and 1.6% of CCA-C active ingredient.

Processing of the fence posts (preservative pretreatment)

In the company's carpentry, the fence posts were processed, and two 3-cm-thick discs were removed 30 cm from the ends of each fence to determine the mean moisture of the fence posts. After this processing step, the length of the fence posts was approximately 150 cm, and they were used in the preservative treatment and for the analysis of treatment quality.

Preservative treatment

Immediately after the specimens were processed, their wet masses were obtained, and the central portion of the fence posts was subjected to preservative treatment so that the moisture sampled was not altered.

The posts were subjected to the normal production process of the forestry company, which consisted of i) 20 minutes of vacuum to reach 515 mmHg and 30 minutes to maintain 515 mmHg; ii) injection of the preservative solution under pressure of 12 kgf.cm⁻² for 45 minutes; and iii) 10 minutes of vacuum to reach 200 mmHg. After the treatment, the fence posts were transported to a covered storage yard with air circulation.

The product used was LIFEWOOD 60 composed of 28.5% hexavalent chromium (CrO₃), 11.10% bivalent copper (CuO), 20.40% pentavalent arsenic (As₂O₅), and 40% water supplied by Koppers Performance Chemicals Brasil Comércio de Preservantes Ltda.

Determination of the mean moisture of the fence posts

Immediately after the 3-cm-thick discs were cut, they were sectioned into specimens measuring approximately 1.5 cm x 1.5 cm x 3.0 cm, which were identified (Figure 1) and immediately weighed to obtain the wet mass. The discs were sectioned to control the humidity of their entire section, including the heartwood. The moisture content of each specimen was determined according to the NBR 7190 standard (Associação Brasileira de Normas Técnicas, 2010). The overall mean moisture of each fence post studied was determined through the arithmetic mean of the moisture content of each specimen.

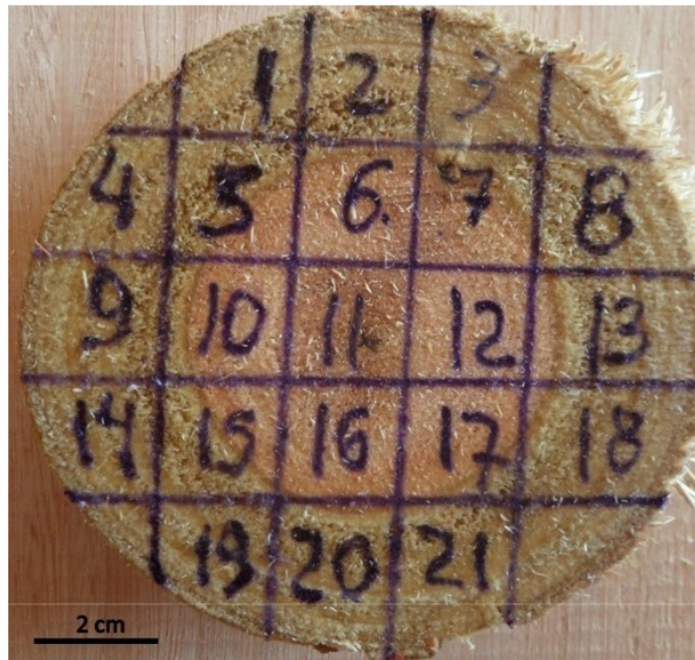


Figure 1. Schematic of the removal of the specimens to determine the moisture of the fence posts before treatment.

CCA-C retention test

For the retention test, 3 equidistant specimens were removed from each fence post (Figure 2). Then, they were dried in an oven at $103 \pm 2 \text{ }^\circ\text{C}$ for 6 hours. They were weighed (dry mass obtained on a precision scale), and the length and diameter (0.01 mm) were measured with a digital caliper to determine the volume for the calculation of the anhydrous density (g cm^{-3}). Subsequently, the specimens were ground and sieved. The samples were filtered in 40 and 60 mesh sieves and dried in an oven ($103 \pm 2 \text{ }^\circ\text{C}$) until they reached constant mass. Then, they were transferred to plastic cuvettes and mounted on Mylar TF-160 film manufactured by Premier Lab Supply located in the city of Port St. Lucie, FL, USA, for X-ray analysis. The CCA-C retention and the concentration of each active ingredient (copper, chromium, and arsenic) of the product in the analyzed samples were obtained by a Lab-X 3500 spectrometer manufactured by Oxford Instruments, located in the city of Witney, Oxfordshire, United Kingdom, by using the anhydrous density as the input variable. The retention of CCA-C and the concentrations of chromium, copper, and arsenic were indicated by spectrometer readings.

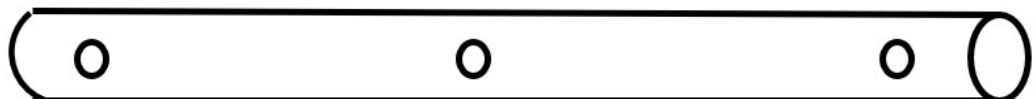


Figure 2. Locations in the fence posts from where the samples were removed.

Statistical analysis

Statistical analyses were performed using the software Sisvar, version 5.6. The results were subjected to analysis of variance by the F test at 5% significance to determine whether there were differences between treatments. When significant differences were found between the quantitative treatments, regression equations were fitted. Tukey's test was performed at 5% significance for qualitative treatments when significant differences were observed.

RESULTS AND DISCUSSION

The minimum, mean, and maximum values of the mean moisture of the fence posts are shown in Table 2.

Table 2. Mean moisture values of the fence posts.

Storage time	Moisture (%)					
	<i>Eucalyptus cloeziana</i>			Clonal hybrid		
	Min.	Mean	Max.	Min.	Mean	Max.
Short	36.18	38.80 (2.80)	44.58	52.60	58.05 (4.26)	64.86
Intermediate	27.26	31.74 (3.04)	34.89	27.45	38.40 (6.17)	48.64
Long	13.76	16.22 (1.81)	18.64	13.03	17.86 (3.57)	23.71

The values in parentheses are the standard deviations (SDs).

CCA-C retention test in wood

The lowest (3.8 kg m⁻³) and the highest (8.1 kg m⁻³) retention values for *E. cloeziana* were observed for the short and long storage time groups and concentrations of 1.2% and 1.6% A.I. CCA-C, respectively. The lowest (3.4 kg m⁻³) and the highest (8.4 kg m⁻³) retention values for the clonal hybrid were observed for the short and long storage time groups and concentrations of 1.2% and 1.6% A.I. CCA-C, respectively. The lowest variability in retention values for *E. cloeziana* was observed for the long storage time group at a concentration of 1.4% A.I. CCA-C, and the highest variability for *E. cloeziana* was observed for the long storage time group at a concentration of 1.2% A.I. CCA-C. For the clonal hybrid, the concentrations of 1.2% and 1.6% A.I. CCA-C had the lowest and highest variability, respectively, both from the long storage time groups (Table 3).

Table 3. Mean retention values at different concentrations of active ingredients for *E. cloeziana* and clonal hybrid.

Storage time	CCA-C retention (kg m ⁻³)/Active Ingredient Concentration (%)					
	<i>Eucalyptus cloeziana</i>			Clonal hybrid		
	1.2	1.4	1.6	1.2	1.4	1.6
Short	4.3 a (0.51)	4.4 a (0.34)	5.0 a (0.37)	3.9 a (0.86)	5.6 a (0.92)	5.4 a (1.09)
Intermediate	5.6 b (0.71)	5.1 ab (0.23)	5.6 a (0.52)	4.7 a (0.71)	5.0 a (0.51)	6.7 ab (0.64)
Long	5.5 ab (1.05)	6.0 b (0.19)	7.2 b (0.84)	5.4 a (0.19)	5.9 a (0.43)	7.1 b (1.12)

The values in parentheses are the standard deviations (SDs). Means followed by the same letters in the column do not differ by Tukey's test at 5% significance.

The lowest mean retention value (4.3 kg m⁻³) for *E. cloeziana* was observed in the fence posts from short storage time group treated with a concentration of 1.2% A.I. CCA-C, while the highest mean retention value (7.2 kg m⁻³) was observed in fence posts from the long storage time group treated with 1.6% A.I. CCA-C, and only the latter were considered fit for use because they reached the minimum retention recommended by NBR 9480 standard (Associação Brasileira de Normas Técnicas, 2009) of 6.5 kg m⁻³ A.I. CCA-C for pieces in contact with soil. The fence posts treated with the other concentrations and storage times did not reach the minimum retention required for use in contact with the soil but were considered suitable for use in structures without contact with the soil according to the same standard, which recommends that the minimum retention for this type of use is only 4.0 kg m⁻³.

For the clonal hybrid, the lowest mean retention value (3.9 kg m⁻³) was observed in the fence posts from the short storage time group treated with 1.2% A.I. CCA-C, while the highest mean retention value (7.1 kg m⁻³) was observed in the fence posts from the long storage time group treated with 1.6% A.I. CCA-C. Only fence posts from the intermediate and long storage time groups treated with 1.6% A.I. CCA-C were suitable for use in contact with the soil. The fence posts treated with the other concentrations and drying times were considered suitable only for use as suspended parts not in contact with the soil; the exception was the fence posts

with a short storage time treated with 1.2% CCA-C, which exhibited values below 4.0 kg m^{-3} – according to the standard, their use is not recommended.

The results of this study confirm the results of authors such as Amaral et al. (2014), who observed a significant increase in mean CCA-C retention with increasing drying time in *Eucalyptus urophylla* fence posts. In turn, authors such as Arantes et al. (2017) did not find satisfactory retention results for use in contact with soil in *Eucalyptus urophylla* x *E. grandis* fence posts treated with CCA-C concentrations higher than those used in this study due to the mean moisture of the pieces used, which were slightly below the fiber saturation point.

Evangelista (2011) worked with the same genotypes or with the parents of the hybrid in question at concentrations of 1.5 and 1.7%, respectively, and obtained values below those recommended by NBR 9480 standard (Associação Brasileira de Normas Técnicas, 2009) for fence posts used in contact with the soil. Schneid et al. (2013) presented satisfactory retention results according to NBR 9480 standard (Associação Brasileira de Normas Técnicas, 2009) for *E. cloeziana*: 7.20; 9.23; 9.10 kg m^{-3} for pressure times of 60, 90, and 120 minutes, respectively, and a concentration of 2%. Valle et al. (2013) also evaluated the retention of CCA-C in *E. urophylla* fence posts treated at 2% concentration and showed results between 8 and 9 (kg A.l.). m^{-3} . Lima (2012) also evaluated the CCA-C retention of fence posts treated at a concentration of 2% and presented 7.3, 10.6 and 9.9 kg m^{-3} for *E. urophylla*, *E. camaldulensis*, and *E. urophylla* x *E. camaldulensis*, respectively.

The analyses of the retention values found for the studied genetic materials clearly shows that increasing the concentration and drying time of the materials is essential to achieving satisfactory retention values. However, as the CCA-C product is responsible for a large part of the costs of wood treatment, it would be more economically viable for preservation plants to optimize the moisture control of the materials to be treated. In addition, there may be differences between genetic materials, and it is not ideal from the business point of view to perform treatments without genotype control.

For both genetic materials, the retentions below the minimum stipulated by NBR 9480 standard (Associação Brasileira de Normas Técnicas, 2009) may be due to poor distribution or penetration of the preservative in the fence posts.

It is noteworthy that the results presented refer to the influence of the overall moisture conditions of the fence posts; that is, they consider both the sapwood (surface) and heartwood (internal) moisture. Electric resistance meters, which are widely used in wood treatment plants, reach only the sapwood; thus, the moisture supplied refers to the surface moisture of the pieces, which quickly enters equilibrium with the environment, thus differing from the overall moisture. Therefore, given the importance of the reported overall moisture, further studies should be conducted to develop operationally viable methodologies for determining the overall moisture of wood pieces in the yards of treatment plants.

The analysis of the influence of the storage time and different concentrations of CCA-C for *E. cloeziana* showed that at a concentration of 1.2% CCA-C, the retentions of the fence posts with long storage times were statistically equal to the retentions of the pieces with short and intermediate storage times and that the retentions for the latter two were significantly different from each other. For the concentration of 1.4%, the retentions of the intermediate time were statistically equal to the retentions of the short and long times, while the retention values of the latter two were significantly different. For the concentration of 1.6%, the retentions of the short and intermediate times were statistically equal and differed from the retentions of the long storage time (Table 3).

For the clonal hybrid, there was no significant difference between the retentions at the different storage times at concentrations of 1.2% and 1.4% CCA-C. For the concentration of 1.6%, it was observed that the intermediate time was statistically equal to the short and long times, while the latter two were significantly different from each other (Table 3).

The data presented by Amaral et al. (2014) confirm those presented in this study, where the highest CCA-C retention (6.41 kg m^{-3}) occurred for the lowest tested moisture (19.24%) and the retentions for the other moisture contents (25.53, 33.87, and 40.45%) were the same (5.70 kg m^{-3}). Similarly, Evangelista (2011) observed CCA-C retention values below that

The control of factors that influence the chemical treatment process is essential for companies. Separating the batches by genetic materials, controlling the humidity of these materials, and defining the concentration of active ingredients to be applied in the autoclave allows for better retention of the chemical and, consequently, a better quality of the treated material.

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