

## ***Eucalyptus* wood treatment and leaching behavior of CCB (Chromated Copper Borate): a field test in Brazilian Midwest**

Tratamento da madeira de *Eucalyptus* e lixiviação de CCB (Borato de Cobre Cromatado): teste de campo no Centro-Oeste brasileiro

Tratamiento de la madera de *Eucalyptus* y lixiviación de CCB (Borato de Cobre Cromado): ensayo de campo en el Medio Oeste brasileño

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### **Abstract**

The use of Chromated Copper Borate (CCB) for wood treatment is known with several studies on a laboratory scale. However, there is a lack of field studies to analyze the effect of the CCB over time. This study aimed to evaluate the wood properties of *Eucalyptus urophylla* S.T. Blake x *Eucalyptus grandis* W. Mill ex Maiden (called E. urograndis), treated with CCB as well evaluate the leaching of chromium, copper and bore (Cr/Cu/B) in field test. The field experiment, with wood treated and untreated (no CCB application), was installed in 2016 and remained until 2018. Wood physico-mechanical properties were evaluated for each condition (treated and untreated) and at three different time: at 0, 1 and 2 years of field exposure. The elements (Cr/Cu/B) losses (leaching) were determined by the difference in the quantification of each element retained in the wood (retention), from year 0 (amount of original elements) in relation to years 1 and 2 of field exposure. The preservative treatment of E. urograndis wood with CCB was efficient to maintain its physical and mechanical properties (mass loss, basic density, rupture and elasticity modulus) during the 2 years of field exposure. The E. urograndis wood without CCB treatment showed reductions in the physical-mechanical properties, indicating their low natural durability. High leaching (close to 100%) for boron was observed. In addition, the total of CCB retention has not changed (statistically) after 2 years.

**Keywords:** Wood properties; Wood deterioration; Wood preservation.

## Resumo

O uso do Borato de Cobre Cromatado (CCB) para o tratamento da madeira é conhecido através de diversos estudos em escala laboratorial. No entanto, faltam estudos de campo para analisar o efeito do CCB ao longo do tempo. Este estudo teve como objetivo avaliar as propriedades da madeira de *Eucalyptus urophylla* S.T. Blake x *Eucalyptus grandis* W. Mill ex Maiden (E. urograndis), tratado com CCB, bem como analisar a lixiviação de cromo, cobre e boro (Cr/Cu/B) em teste de campo. O experimento de campo, com as madeiras tratadas e não tratadas (sem aplicação de CCB), foi instalado em 2016 e permaneceu até 2018. As propriedades físico-mecânicas da madeira foram avaliadas para cada condição (tratada e não tratada) e em três momentos distintos: 0, 1 e 2 anos de exposição em campo. As perdas dos elementos (Cr/Cu/B) (lixiviação) foram determinadas pela diferença na quantificação de cada elemento retido na madeira (retenção), a partir do ano 0 (quantidade de elementos originais) em relação aos anos 1 e 2 de exposição em campo. O tratamento preservativo da madeira de E. urograndis com CCB foi eficiente para manter suas características físicas e mecânicas (perda de massa, densidade básica, ruptura e módulo de elasticidade) durante os 2 anos de exposição em campo. A madeira de E. urograndis sem tratamento CCB apresentou redução significativa nas propriedades físico-mecânicas, indicando a sua baixa durabilidade natural. Foi observada alta lixiviação (perto de 100%) para o boro. Além disso, a retenção total de CCB não mudou (estatisticamente) após 2 anos.

**Palavras-chave:** Propriedades da madeira; Deterioração da madeira; Preservação da madeira.

## Resumen

El uso de Borato de Cobre Cromatado (CCB) para el tratamiento de la madera se conoce a través de varios estudios a escala de laboratorio. Sin embargo, faltan estudios de campo para analizar el efecto de CCB a lo largo del tiempo. Este estudio tuvo como objetivo evaluar las propiedades de la madera de *Eucalyptus urophylla* S.T. Blake x *Eucalyptus grandis* W. Mill ex Maiden (denominada E. urograndis), tratada con CCB, además de analizar la lixiviación de cromo, cobre y boro (Cr/Cu/B) en pruebas de campo. El experimento de campo, con madera tratada y sin tratar, se instaló en 2016 y se mantuvo hasta 2018. Se evaluaron las propiedades físico-mecánicas de la madera para cada condición (tratada y sin tratar) y en tres momentos diferentes: 0, 1 y 2 años exposición de campo. Las pérdidas de elementos (Cr/Cu/B) (lixiviación) fueron determinadas por la diferencia en la cuantificación de cada elemento retenido en la madera (retención), desde el año 0 (número de elementos originales) en adelante en relación a los años 1 y 2. El tratamiento conservante de la madera de E. urograndis con CCB fue eficaz para preservar sus propiedades físicas y mecánicas (pérdida de masa, densidad básica, ruptura y módulo elástico) durante los 2 años de exposición en campo. La madera de E. urograndis sin tratamiento CCB mostró una reducción de las propiedades físico-mecánicas, lo que indica una baja durabilidad natural. Se observó una alta lixiviación (cercana al 100%) de boro. Además, la retención total de CCB no cambió (estadísticamente) después de 2 años.

**Palabras clave:** Propiedades de la madera; Deterioro de la madera; Preservación de la madera.

## 1. Introduction

Wood becomes vulnerable to the action of xylophagous agents when in direct contact with the soil. The wood is attacked by fungi and insects, which can deteriorate it and reduces service life, with significant economic losses (Bi et al., 2019).

To define the degree of resistance offered by wood to the action of degrading organisms, field test involving partial burial of wood pieces followed by their periodic health assessments are fundamental (Ncube et al., 2012; Brischke et al., 2013; Kleindienst et al., 2017; Torres-Andrade et al., 2019). It is considered extremely important to conduct field deterioration tests with small wood pieces to allow collection of information regarding wood resistance in a short time (Ribeiro et al., 2014; Tarvainen et al., 2020).

To extend the service life of wood, preservatives frequently are employed to prevent wood deterioration from fungi and insects damage (García-Valcárcel and Tadeo, 2006; Panigrahi and Tripathy, 2019; Noll et al., 2019; Akcay et al., 2020). Wood preservation industries across the globe produced many effective synthetic chemicals that help in protecting wood products under different service conditions against deteriorating agents for many years (Adebawo, 2020). The CCB (Cu/Cr/B) is one of commercial preservative used to treat wood fence posts. It is composed of hexavalent chromium in the form of CrO<sub>3</sub> (63.5%); copper as CuO (26.0%) and boron (10.5%), with the features of fixation of elements fungicidal and insecticidal, respectively (Teleginski et al., 2016).

Commercialization of CCB began in Germany in the early 1960s with a view to replace CCA (Cu/Cr/As) due to the arsenic toxicity (Shanu et al., 2015) and can constitutes in environmentally friendly methods for the treatment of the wood than

the CCA (Teacă et al., 2019). The wood preservative industry is interested in finding environmentally friendly methods for the treatment of the wood (Teacă et al., 2019) and the public increasingly sensitive to the use toxic products, like arsenic, because of their potential risks to the environment as well as human health (Bi et al., 2019).

However, the use of CCB has a resistance due to their higher leaching as compared to CCA. Despite that, the literature offers no consensus on the study of the CCB-treated wood behavior in a field experiment. Thus, study of the behavior of wood treated with CCB in a field test is important to define the preservative product leaching. Also, it is important to determine mass loss, basic density, and mechanical strength of wood in the field tests (Quintilhan et al., 2018; Torres-Andrade et al., 2019). It is necessary to accumulate critical data regarding the reduction of these properties when the wood is exposed in the field conditions (Trevisan et al., 2007). The results of analyzes will be important to establish the feasibility of using CCB treated *Eucalyptus* wood.

The present study aimed to evaluate the physico-mechanical properties of *Eucalyptus urophylla* x *Eucalyptus grandis* (E. urograndis) wood treated with Chromated Copper Borate (CCB) as well as evaluate the leaching of chromium, copper and bore (Cu/Cr/B) in field test.

## 2. Methodology

The methodology applied was experimental research, according to the classification of Pereira et al. (2018), with data collection and analysis techniques through wood sampling.

### 2.1 Study area

The field experiment was installed in the Midwest region of Brazil (16°18'28.85"S; 49°13'3.80"W), from July 2016 to August 2018. The climate of the region is Aw, koppen classification (Alvares et al., 2013) with humid and rainy summer; and dry winter. The average annual precipitation is 1,432 mm and average temperature 23.0 °C. The soil was characterized by the presence of red latosol, average texture, low organic matter content, and moderate acidity, as determined by Al<sup>3+</sup>, H+Al and aluminum saturation.

### 2.2 Wood sample collection, preservative treatment and field test installation

The wood samples were collected from a forest plantation at seven-year-old. The forest species was the *Eucalyptus urophylla* S.T. Blake x *Eucalyptus grandis* W. Mill ex Maiden (E. urograndis). Ten trees were selected and then felled, debarked and cut into fence posts, with length of 2.20 meters and diameters ranging from 9.2 to 15.4 cm. The E. urograndis wood fence posts were air dried for ninety days until reaching 25% moisture content.

Thirty fence posts were obtained (3 pieces per tree). Fifteen were submitted to preservative treatment and fifteen were not treated. Thus, two conditions of evaluation treated and untreated wood were constituted.

The fence posts treatment was in an industrial preservative company with Chromated Copper Borate (CCB), with 50% active ingredients and at concentrations of 2.0 %. An autoclave was used in a full-cell process (Bethell method) and pressure of 12 kgf cm<sup>-2</sup> per 60 min.

The concentration, pressure and time used in this study were defined according to the parameters commonly used in the wood treatment plants in Brazil (Lopes et al., 2017). After the preservative treatment, the fence posts were air dried for 30 days to the preservative fixation.

The treated and untreated wood fence posts were cut into samples with dimensions of 2.0 x 2.0 x 30 cm (radial, tangential and longitudinal). Four wood samples were taken by each fence posts in a total of 120 samples from the sapwood region (wood treatable region): 60 treated and 60 untreated samples.

The wood samples were placed in a climate chamber (21°C and 65% Relative Humidity) to reach 12% moisture content then weighed. These wood samples were buried upright up to half their size (15 cm) in the experimental area (item 2.1) with a distance of 30 cm between them. The field experiment was installed in June 2016 (year 0) and remained until June 2018 (year 2), completing two years of field exposure.

### 2.3 Wood properties and leaching

Wood properties and leaching evaluations were performed at three different time points: (i) year 0 - samples before being subjected to the field experiment; (ii) year 1 - samples after 1 year in field experiment and (iii) year 2 - samples after 2 years in field experiment.

For the evaluation of wood properties, twenty (20) samples for each condition (treated and untreated) were collected randomly per year (0, 1 and 2 years). The treated and untreated wood samples were dug up and cleaned (removing all soil accumulation) after one and two years, respectively.

The wood samples were submitted to a climate chamber (21 °C and 65% Relative Humidity) to reach 12% moisture content and weighed. The calculation of mass loss and determination of the degree of resistance to field degrading agents were performed as described in ASTM D-2017 (ASTM, 2005). The loss of mass was obtained from the difference between the initial mass (before the installation of the experiment) and after its removal (1 and 2 years).

Subsequently, the samples were subjected to mechanical testing on a universal testing machine with a maximum load capacity of 500 kgf at a speed of 1 mm min<sup>-1</sup> as suggested by Melo and Del Menezes (2016). The elasticity and rupture modules in static flexion were calculated according to Pan American Standards Commission - COPANT 30:1-006 (COPANT, 1971).

After the determination of mass loss and mechanical properties, half of the samples (10 samples per year and condition; treated and untreated) were used to determine the wood basic density by hydrostatic balance method according Brazilian standard NBR 11941 (ABNT, 2003) and the other half (10 samples per year; only treated condition) was used to determine CCB leaching.

To assess CCB leaching, the wood samples treated were crushed. Then the wood material obtained in 60 mesh granulometry was used to digestion, and to determine the amount of each element (copper, chromium and boron) per wood cubic unit (retention) in the Aqualiti Tecnologia em Saneamento laboratory, according to the methodology proposed by NBR 6232 (ABNT, 2013) and Paes et al. (2014).

The technique of inductively coupled plasma optical emission spectrometry (ICP - OES, PerkinElmer, Optima 7800 DV), equipped with a concentric nebulizer and cyclonic nebulization chamber, was used to quantify the elements copper, chromium and boron (Cu/Cr/B) retained in the wood. This technique is one of the most used to verify metals retained in wood, as suggested by Vivian et al. (2012) and Lima et al. (2020). The obtained values were converted into retention units (kg of active ingredient per m<sup>3</sup>).

The elements (Cr/Cu/B) losses (leaching) were determined by the difference in the quantification of each element retained in the wood (retention), from year 0 (amount of original elements) in relation to years 1 and 2, as proposed by García-Valcárcel et al. (2004).

### 2.4 Statistical analysis

Outliers were measured by the Box-Plot method, normal distribution of data was evaluated by the Shapiro-Wilk test, and heterogeneity of variance by the Bartlett and Levene tests ( $p < 0.05$ ). The data presented normality of distribution and homogeneity of variance. The analysis of variance (ANOVA), Student's t-test and Tukey's test were performed in order to verify the effect of the condition of the samples (treated and untreated) and the exposure time of the samples in the field

experiment (years 0, 1, and 2), respectively. The values obtained for each year of evaluation were used to calculate the average of the treated and untreated condition.

### 3. Results

#### 3.1 Wood properties

The *E. urograndis* wood properties were affected by the condition (treated and untreated) and time of exposure in the field (years) (Table 1). In addition, deterioration by fungi was observed in the untreated samples (Figure 2).

**Table 1:** Mass loss (ML), Basic density (BD), Rupture (RM) and Elasticity (EM) modulus of treated and untreated *E. urograndis* wood within 2 years of evaluation.

Condition	Year	ML (%)	BD (kg m <sup>-3</sup> )	RM (MPa)	EM (MPa)
Treated	0	0.00	570.00 a	107.51 a	10,363.20 a
	1	0.49 b	580.00 a	103.71 a	9,852.41 a
	2	1.04 a	580.00 a	100.33 a	9,818.38 a
Mean		0.77 *	577.00 *	103.85 *	10,011.33*
Untreated	0	0.00	560.00 a	99.53 a	10,326.47 a
	1	37.87 b	390.00 b	20.66 b	3,568.86 b
	2	57.13 a	250.00 c	-	-
Mean		47.50	400.00	60.10	6,947.66

Averages followed by the same lowercase letter by year do not differ at 5% probability. (-) untreated samples after 2 years of field experiment could not be analyzed; see Figure 2B. \*difference significative between conditions ( $p < 0.05$ )

Source: Authors.

The wood untreated showed greater mass loss when compared to wood treated with CCB (47.50 % against 0.77 %). The results showed that after 2 years of field exposure, the wood treated lost only 1.04% of their initial mass, while the untreated lost 57.13% (more than half of its initial mass).

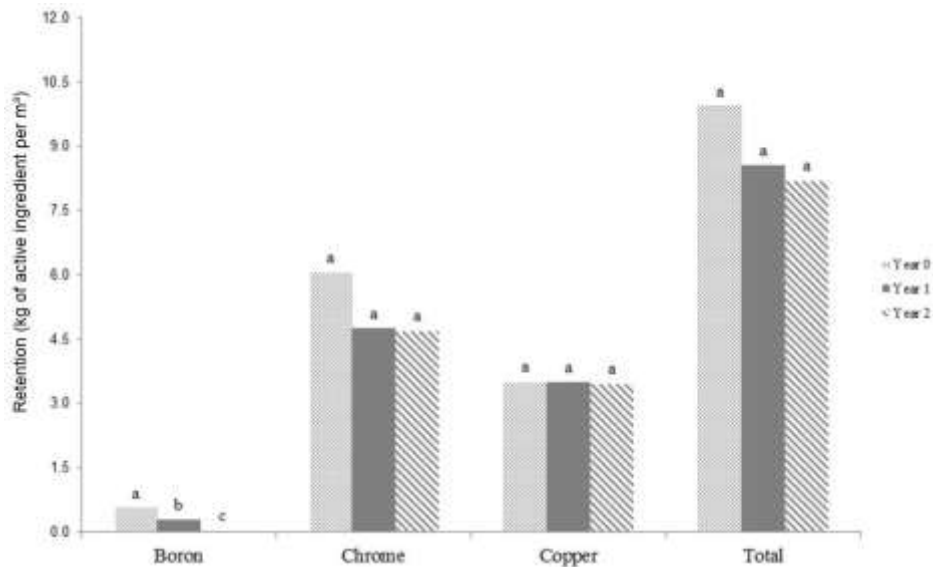
The basic density in the untreated wood showed a reduction of 310 kg m<sup>-3</sup> (560 kg m<sup>-3</sup> - before buried - to 250 kg m<sup>-3</sup>) after 2 years of exposure in the field. In the same way, the untreated wood samples reduced significantly their RM and EM from 99.53 to 20.66 MPa and 10,326.47 to 3,568.86 MPa after 1 year in the field test. The untreated wood taken in the second year did not have adequate dimensions to perform the mechanical tests (see Figure 2B); therefore, the data is not shown.

Regarding wood subjected to preservative treatment with CCB: there was no reduction in physical and mechanical properties (BD, RM and EM) with the time of exposure (years).

#### 3.2 CCB Leaching

Losses of elements from CCB-treated wood were highest for boron (B); from 0.56 to 0.04 kg of active ingredient per m<sup>3</sup> and lowest for Cr and Cu (Figure 1). The reduction in the amount of B compared to the original treated wood samples was almost 100%.

**Figure 1:** Retention of Boron, Chrome, Copper and Total (B+Cr+Cu) in the *E. urograndis* wood within two years of evaluation. Means followed by the same letter, per element and total retention, did not differ statistically (Tukey's test,  $p < 0.05$ )



Source: Authors.

## 4. Discussion

### 4.1 Wood properties

The reduction in the properties in untreated (without CCB) *E. urograndis* wood exposed to the field test per 2 years (Table 1; Figure 2 a, b) indicated their low natural resistance to the biodeterioration. These reductions are related to the development of xylophages organisms (Quintilhan et al., 2018; Oliveira et al., 2019). Fungi are amongst the organisms involved in the wood biodegradation (Kleindienst et al., 2017).

Basidiomycete fungi, which are responsible for white and brown rot, are the main fungi responsible for the wood biodegradation (Pawlik et al., 2019; Mattila et al., 2020). The white rot fungi are able to degrade the cellulose, hemicellulose, and lignin, resulting in a severe reduction in wood material. The brown rot degrades only the polysaccharides (i.e., cellulose and hemicellulose) (Kim et al., 2019).

The action of fungi decomposes the wood cell wall biopolymers (cellulose, hemicelluloses, lignin), causing changes in the anatomical structure and, consequently, decreasing the wood properties (Pawlik et al., 2019).



**Figure 2:** Untreated and treated *E. urograndis* wood samples after one (a, c) and two (b, d) years of field test.



Source: Authors.

The results indicated that untreated wood lost almost half of its initial mass (47.50%), resulting in non-resistant class according to the American Society for Testing Materials D2017 (ASTM 2005) classification. The stronger reductions in basic density in untreated wood were also observed and indicate that the fungi acted by degrading the cell wall of wood.

According to Oliveira et al. (2019), the wood density of *Eucalyptus* species may be an indicator of resistance to the attack of xylophagous agents since low wood density tends to be more easily decayed. The changes in the physical properties in untreated wood had an impact on the mechanical properties. The rupture modulus (RM) and elasticity modulus (EM) values were reduced by 40% and 30% (average values by conditions), respectively.

Studies have shown that mechanical properties of wood has a reduction with the action of decay fungi (Singh and Singh 2014; Witomski et al., 2016; Silva et al., 2019) and termites (França et al., 2017; Gallio et al., 2018). When external organisms consume of a wood component, an effective loss of material and therefore a reduction in mechanical properties (Kim et al., 2019).

The longer the exposure time of untreated wood in the field, the greater the changes in the wood properties. The reduction in the initial mass and basic density, from year 0 to year 2, was greater than 50% (0.00 to 57.13% and 560 to 250 kg m<sup>-3</sup>, respectively). Wood deterioration can be observed in *E. urograndis* samples buried after 2 years (Figure 2b). In addition, due to degradation, it was not possible to carry out mechanical tests in untreated samples (2 years).

On the other hand, the wood treated with CCB did not change its physical-mechanical properties (Table 1 and Figure 2 c, d) up to the second year of soil exposure and indicate the importance of preservative treatment in the wood resistance to field biodeterioration. The effectiveness of the preservative process through the action of chemical elements applied to wood (such as insecticide and fungicide) has been proven by comparing treated and untreated woods (Humar et al., 2011; Mattos et al., 2013; Quintilhan et al., 2018).

Although the mass loss of treated wood increased from year 1 to year 2, the resistance of *E. urograndis* wood to the attack of xylophages was not affected; remained highly resistant according to the American Society for Testing Materials D 2017 (ASTM, 2005) classification. The values for RM and EM in treated wood at all evaluation years were sufficient to meet the minimum values required for the use of wood in the form of fence posts, according to Brazilian standards NBR 9480 (ABNT, 2018), which requires the minimum resistance of 52 MPa, for woods with a moisture content of 12%.

#### 4.2 CCB Leaching

The B (boron) applied in the *E. urograndis* wood during preservative treatment was almost completely leached after 2 years of field testing. Some studies also confirmed the greater boron leaching when subjected to tests that determined the migration of wood elements to other environments (García-Valcárcel et al., 2004; Kaur et al., 2016).

High B leaching may be related to the inadequate formation of Cr borate, which fixes B in wood (Pizzi and Baecker, 1996). In addition, boron is considered to be the most mobile element of CCB, especially when wood is in direct contact with soils (Lebow, 1996).

Several factors may affect the greater or lesser B leaching, such as the time and exposure factors, wood properties, (Kaur et al., 2016). The leaching resistance of preservative components in wood depends on the physical and chemical properties of the preservative product. Also, depends on the way it attaches to wood as well as it being insoluble in water or being capable of forming insoluble complexes through chemical reactions with the cell wall components (Panigrahi and Tripathy, 2019).

The total retention of CCB in *E. urograndis* wood did not change statistically from year 0 to 2 and the values obtained are in accordance with the minimum recommended by the Brazilian standard (6.5 kg kg of active ingredient per m<sup>3</sup>) for the application of fence posts in contact with the soil (ABNT, 2018). The Brazilian standard does not consider isolated element retention. The results showed that B was completely leached in 2 years and the wood was still preserved (Figure 2 c, d).

## 5. Conclusion

- The treatment of *Eucalyptus urophylla* x *Eucalyptus grandis* wood with Chromated Copper Borate (CCB) was efficient to maintain its physical and mechanical properties during the 2 years of field exposure.
- The *Eucalyptus urophylla* x *Eucalyptus grandis* (7 years old) wood without CCB treatment showed significant reductions in the physical-mechanical properties, indicating their low natural durability to the action of xylophagous agents.
- High leaching (close to 100%) for boron was observed. Even though the total CCB retention has not changed (statistically) in 2 years and the values of retention are in accordance with the minimum recommended by the Brazilian standard to the fence posts in contact with soil.
- Supplementary studies, such as the exposure the CCB treated *Eucalyptus urophylla* x *Eucalyptus grandis* wood in a field test longer (more than 2 years) and with other edaphoclimatic characteristics, are recommended.

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