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# APPLICATION OF NANOMETRIC ACRYLICS TO REDUCE BORON LEACHING

***Claudia M IBAÑEZ****
Prof.dr.eng. – Sede Tacuarembó, Universidad de la República
Address: Campus Interinstitucional, Ruta 5 km 386.500, Tacuarembó, Uruguay
E-mail: marcela.ibanez@cut.edu.uy

***Álvaro CAMARGO***
Prof.MSc. – Sede Tacuarembó, Universidad de la República
Address: Campus Interinstitucional, Ruta 5 km 386.500, Tacuarembó, Uruguay
E-mail: alvaro.camargo@cut.edu.uy

***Damián GHERSCOVIC***
Prof.Chem. – Instituto Nacional de Tecnología Industrial. Madera y Muebles
Address: Juana Gorriti 3520, Buenos Aires, Argentina
E-mail: damian@inti.gob.ar

***Ricardo CAMERA***
Chem. – Instituto Nacional de Tecnología Industrial. Madera y Muebles
Address: Juana Gorriti 3520, Buenos Aires, Argentina
E-mail: ricardocamera@hotmail.com

***Abstract:***

*Boron compounds are widely used for their fungicidal, insecticidal, and flame retardant properties; they are also of low toxicity. However, in highly humid environments boron leaches easily from the wood, hence the numerous researches that seek to reduce this effect. In this work, disodium octaborate tetrahydrate (DOT) mixed with an organic polymer emulsion based on nanometric acrylics was applied in two concentrations to Pinus taeda and Populus sp wood. The effects of the organic polymer emulsion on the reduction of boron leaching were evaluated. The treatment was tested against brown and white rot fungi according to the EN113 standard. After leaching, samples of both wood species treated with the organic polymer showed decreased weight losses. The organic polymer decreased boron loss by leaching, even in highly humid conditions, making it potentially suitable for outdoor use.*

***Key words:*** *boron; Gloeophyllum trabeum; leaching; organic polymer; Trametes versicolor*

## INTRODUCTION

Simple borates such as boric acid, borax [$Na_2B_8O_{13}.4H_2O$] or sodium octaborate tetrahydrate (DOT) combine a unique set of properties that make them very suitable for wood protection. Their fungicidal and insecticidal properties are renowned, being considered superior to those of copper or zinc, although the latter fixate better to wood (Lloyd *et al*. 2001, Kartal *et al*. 2020). They are effective fire retardants, easy to handle and treat wood (Marney and Russell 2007, Uner *et al*. 2016). Their mobility and water solubility allow them to be applied to species otherwise impossible to treat with other preservatives; they can even be used to treat *Eucalyptus grandis* heartwood with high water content (Caldeira, 2010, Ibáñez *et al*. 2017). They are low cost, odorless and colorless. As water soluble products, they can be applied to wood by conventional methods such as immersion, diffusion - even to dry wood with or without incisions - vacuum-pressure or as steam, with or without temperature (Caldeira, 2010). In addition, they are of low toxicity to humans and their environmental impact is minimal (Teshima *et al*. 2001, Bolt *et al.* 2017, 2020). For over fifty years they have been used in commercial products, mostly for industrial use.

However, their water solubility makes them easily leachable; in high humidity environments they are almost completely leached from the wood, and thus are not suitable for exterior use or in contact with soil (AWPA 2016). Ramos *et al*. (2006) argues that physical rather than chemical adsorption is the preferential mechanism by which boron binds to wood. In this regard, many researches have been carried out in order to partially fixate boron to wood without losing its biocidal properties.

Obanda *et al.* (2008) reviewed this topic presenting the different strategies that have been developed, from surface treatments -such as the application of varnishes, resins and hydrophobic waxes- to treatments that either combine more than one compound or precipitate as organ-soluble salts -using metalloborates, ammoniacal metalloborates or amines and their oxides. They have also been combined with proteins,

* *Corresponding author*

tannins, siloxane, silicates and silicone (Kartal *et al*. 2009, Palanti *et al.* 2012, Thevenon *et al.* 2008, Tondi *et al*. 2017, Ratajczak and Mazela 2007).

More recent studies report that boron lixiviation can be reduced by covalent bonds formed with tannins (Efhamisisi *et al*. 2017), combining disodium octaborate tetrahydrate (DOT) with a wax emulsion (Wang *et al*. 2018) or through treatments in stages -applying quaternary ammoniums and sodium fluoroborate, which also give hydrophobic properties to the treated wood (Huang *et al*. 2018).

The future of boron as a wood protector depends on its fixation to wood; however, it must also allow its mobility, in order to preserve its ability to form complexes with molecules of biological importance. Boron biocidal efficacy is related to the formation of complexes between the tetrahydroxyborate ion $[B(OH)]^{4-}$, and the polyols of molecules with biological importance for decay fungi or wood xylophagous insects (vitamins, coenzymes etc) (Freeman *et al.* 2009). Combinations of boron or metals with acrylics are primarily used in coatings and adhesives (Dulgar *et al*. 2019, Eren and Kaplan 2019). In this work a combination of boron and acrylic polymer is deeply applied to solid wood in one step, reducing the operating costs of the impregnation process. This polymer is formed from nanometric acrylic compounds to facilitate its distribution inside wood. Disodium octaborate tetrahydrate (DOT) is used as the boron source as it is significantly more soluble in water than other boron compounds and contains more boron per unit mass. The aim is to determine whether the acrylic polymer can prevent boron leaching from wood without decreasing its fungicidal properties, even when used in high humidity environments.This work seeks to demostrate the potential commercial use of this formulation and method, making boron salts more effective and widespread for outdoor uses.

## MATERIALS AND METHODS

### *Wood selection, conditioning and impregnation*

Samples of *Pinus taeda* L. and *Populus* sp sapwood (500 per species), of (50 ± 2.0 [L] x 25 ± 2.0 [T] x 15 ± 2.0 [R]) mm, free of knots, defects and fungal or mould action were used. Samples were oven dried at 103 ± 2°C for 24 hours, and allowed to cool at room conditions in a diseccator, in order to determine initial dry weight and density; those that differed by less than 15% from the average density value were selected (460 samples per species). Disodium octaborate tetrahydrate (DOT, 99.5% purity) from Bórax Argentina S.A. (Salta, Argentina) was used at two concentrations, 1.4 and 2.1% (boric acid equivalents), named B1 and B2 respectively.

The anti-leaching product, used in two concentrations -named F0 (7% w/w) and F1 (14% w/w) - was developed by an Argentinian chemical company; it is a low toxicity organic polymer formed from nanometric acrylic monomers, with no fungicidal properties. The product was provided by the manufacturer already polymerized and ready for application with boron. Each DOT concentration (B1 and B2) was applied to the wood, both alone and mixed with each anti-leaching concentration (F0 and F1) - resulting in six treatments, plus two control treatments with CCA (Chromated copper arsenate) and CCB (Copper chrome borate), both at a concentration of 1.4% w/w. CCA and CCB were used as control treatments as they both are commercial products with renowned fungicidal properties, high efficacy and resistance to leaching.

Each treatment was applied to 28 samples per species (224 treated samples per species; the remaining untreated samples were used as control) by the full cell method (vacuum-pressure-vacuum) in the laboratory. The stages of the cycle were the following: 30 minutes of vacuum at 0.6 bar - 60 minutes at 10 bar of pressure - 30 minutes of vacuum at 0.6 bar. Table 1 shows the treatment applied in each group of samples.

*Table 1*

**Treatments applied to the *P. taeda* and *Populus* sp samples**

| Used terminology | Treatments* |
|---|---|
| B1 | 1.4 % B |
| B2 | 2.1 % B |
| B1F0 | 1.4% B + F0 |
| B1F1 | 1.4% B + F1 |
| B2F0 | 2.1% B + F0 |
| B2F1 | 2.1% B + F1 |

*boron concentration (%) applied to the wood, and anti leaching product at two concentrations, F0 and F1.

Once the samples were treated, retentions in each treatment were determined according to the following formula:

$$R\ (kg/m^3) = (w_2 - w_1) * (C) / v$$

where: R is retention of impregnating treatment ($kg/m^3$); $w_1$: initial anhydrous weight of each sample (kg); $w_2$ weight of each treated sample after treatment (kg); C: boron concentration (%); v: volume of each sample ($m^3$).

The samples were then put in an oven at 60°C in trays covered with polyethylene for 26 hours in order to promote fixation according to indications provided by the manufacturer. Those samples that did not differ by more than 15% from the average retention value were selected for the subsequent tests.

***Leaching test***

This pre -treatment emulates long-term use conditions by accelerated ageing of the samples in a desiccator. After the fixation period and before exposing the samples to decay fungi, half the samples of each treatment (14 per species) were leached according to the standard EN 84:1997. The samples were immersed in more than five times their volume of deionized water and then maintained under vacuum (0.6 bar) for 20min. Then, the samples were soaked for 2h and finally maintained in fresh deionized water, changed ten times within 14 days.

***Boron quantifications***

The boron lost in the leaching water was quantified on days 1 and 9 by a potentiometric method: a complex is formed between the borates present in the sample and mannitol (Merck Com. Darmstadt, Germany); this complex acidified the solution, which was titrated with a dilute alkaline solution of NaOH (0.025M) (Dawson *et al*. 1990). The amount of boron in the sample is equivalent to the amount of NaOH required to reach the initial pH of the leaching water (Wilson 1958). Before the test, the methodology was validated so that acrylic or wood extractive residues did not affect the results.

***Fungal resistance test***

The essays were carried out according to the standard EN113:1996. Two strains of decay fungi were used; *Trametes versicolor* (BW001 FCST) (white rot) and *Gloeophyllum trabeum* (BB001 FCST) (brown rot). Both strains belong to the fungi collection of the Forestry Laboratory of Sede Tacuarembó, Universidad de la República del Uruguay. The strains were maintained in a malt extract (12.5g/L) - agar (20g/L) medium (AM), both from Oxoid Ltd (Basingstoke, UK).

Flasks containing AM were sterilized in an autoclave at 121°C for 15 minutes and inoculated with the fungi. They were then incubated at 28°C and 75% relative humidity (RH) until the surface of the culture medium was completely covered by the fungi.

The wood samples (leached, non leached, and control) were conditioned at 22°C and 65% RH for two weeks, then steam- sterilized for 10 minutes (each 24 hours for three days), and finally exposed to the fungi.

A treated sample and an untreated control sample were placed in each flask -resulting in 224 flasks per wood species, plus 2 more flasks with two untreated control samples each (without DOT or acrylic). After incubation, the samples were oven dried at 103 ± 2°C until constant weight (dry final weight) and the efficiency of each treatment was determined through:

$$\% \text{ weight lost} = (\text{initial dry weight} - \text{final dry weight} / \text{initial dry weight}) \times 100.$$

***Statistical analysis***

Results were analyzed by the STATISTICA software (version 7.1 2005; Statsoft Inc., Tulsa, OK, USA). ANOVA and the Duncan multiple range test with a significance level of $\alpha = 0.05$ were performed on the fungal resistance results of the treated wood.

**RESULTS AND DISCUSSION**

Table 2 shows the average retention values of the mixed chemicals (DOT and anti leaching polymer at different concentrations) for each treatment with their respective deviations. In all cases, *P.taeda* had higher retentions than *Populus* (c. 10%) due to its better penetrability. The lowest concentration of boron, B1 had low retention values, even when applied with the anti -leaching products. In this regard, neither F0 nor F1 effected retention significantly, and had little difference between each other.

*Table 2*

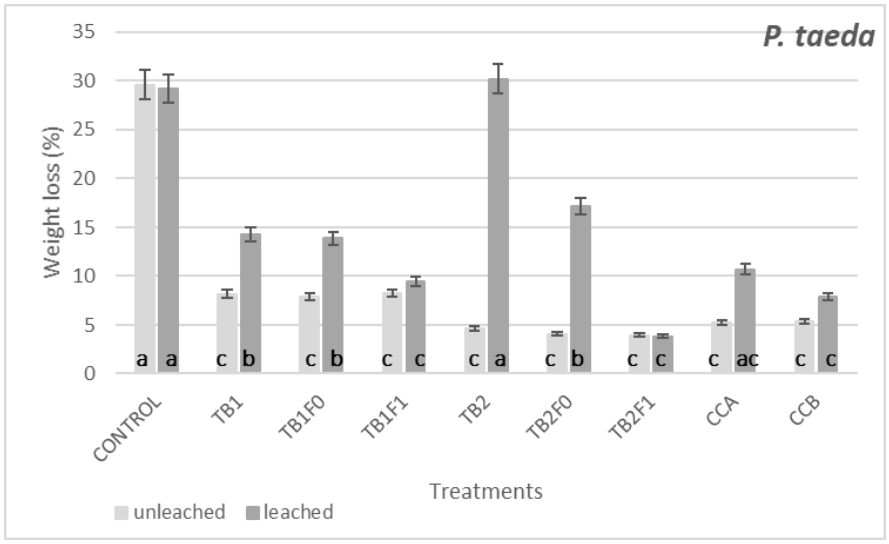
**Preservative retentions for each treatment - DOT and antileaching polymer – in *Populus sp* and *Pinus taeda***

| Retention of mixed chemicals (kg/m$^3$ wood) (DS) | | | |
|---|---|---|---|
| ***Populus sp*** | | ***Pinus taeda*** | |
| PCCA | 6.4 (0.3) | TCCA | 7.7(2.1) |
| PCCB | 6.5 (0.3) | TCCB | 7.6(0.7) |
| PB1 | 6.6 (0.5) | TB1 | 7.6(0.8) |
| PB1F0 | 6.4 (0.5) | TB1F0 | 8.5(1.0) |
| PB1F1 | 6.2 (0.6) | TB1F1 | 8.7(0.9) |
| PB2 | 9.3 (0.8) | TB2 | 10.7(1.2) |
| PB2F0 | 9.1 (1.0) | TB2F0 | 11.6(1.2) |
| PB2F1 | 9.7 (0.8) | TB2F1 | 11.1(1.2) |

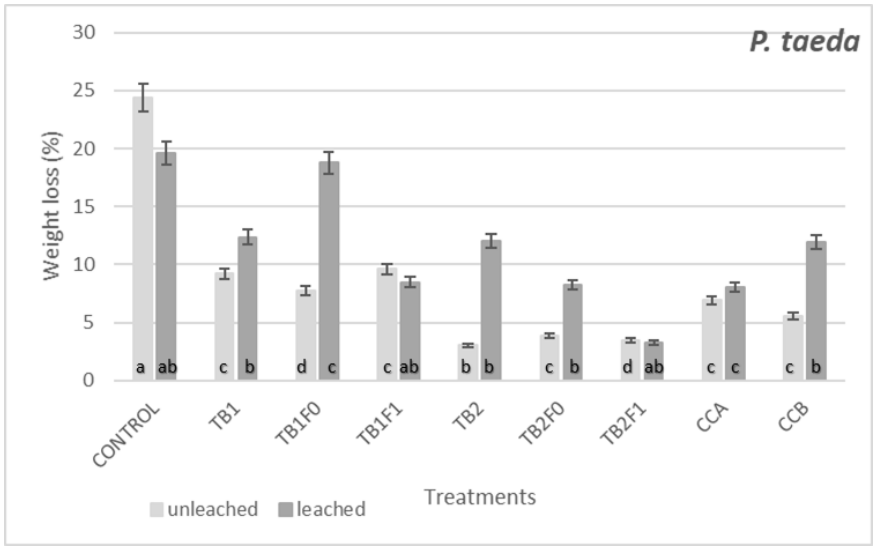
Average values with their respective standard deviations DS. P: *Populus sp* T*: P taeda.*
B1: 1.4 % B; B2: 2.1 % B; B1F0: 1.4% B+ F0; B1F1: 1.4% B + F1; B2F0: 2.1% B+ F0; B2F1: 2.1% B + F1.

Fig. 1 shows fungal resistance (average weight loss values). The weight loss of the untreated control samples is around 20%, validating the decay capacity of the fungi according to the standard EN 113 (1996).

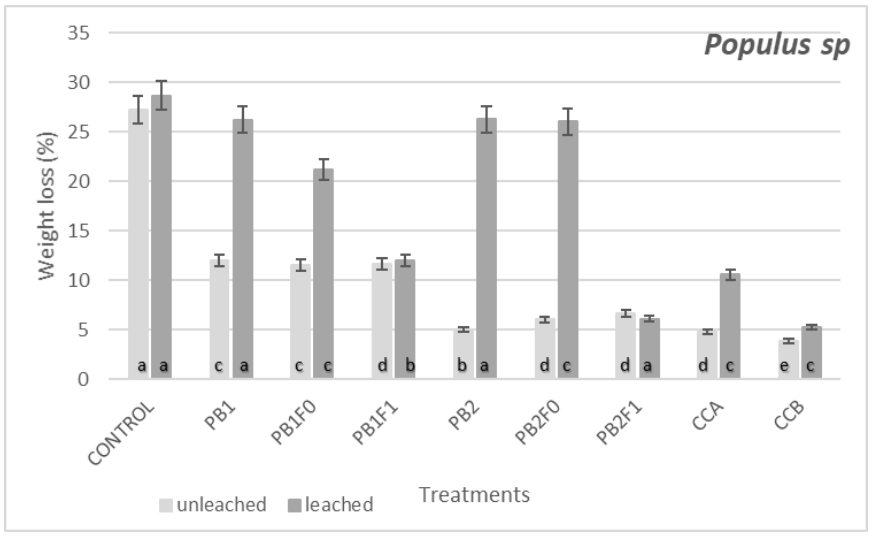
a




b




c



d

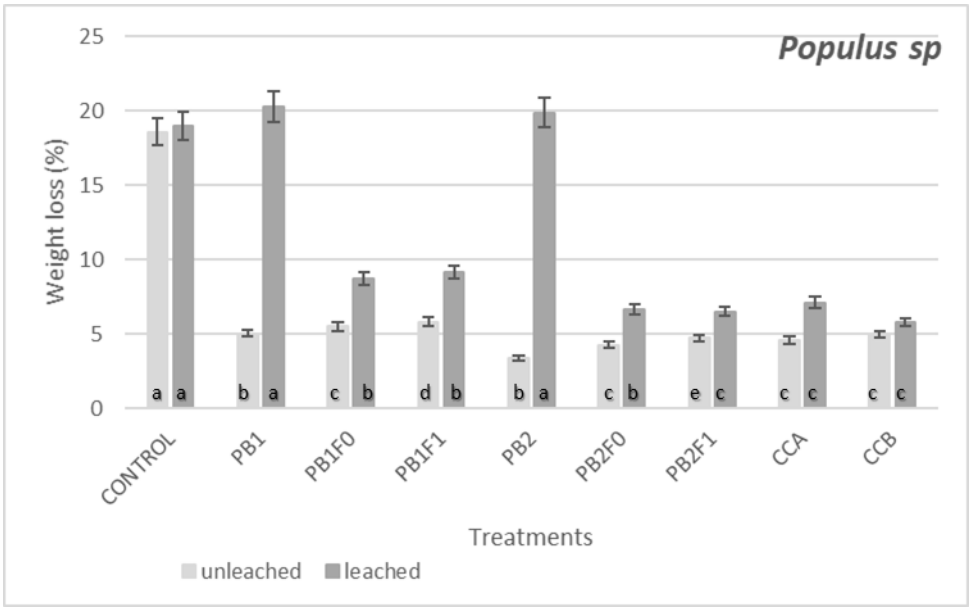



***Fig. 1.***
***Weight loss (%) of treated, untreated and control P. taeda and Populus sp. samples by fungal decay: a) and c) by Trametes versicolor; b) and d) by Gloephyllum trabeum***
***Equal letters indicate there is no significant difference between treatments (Duncan test).***
***B1: 1.4 % B; B2: 2.1 % B; B1F0: 1.4% B + F0; B1F1: 1.4% B + F1; B2F0: 2.1% B + F0; B2F1: 2.1% B + F1.***

Weight losses on wood treated only with DOT (B1 and B2) were below 10%, with the exception of B1 on *Populus* sp (PB1), which reached 12%; however, in all cases weight loses were above 3% - limit value by which the wood is considered protected according to EN 113 standard. This result was unexpected since all retention levels were above the DOT toxicity threshold for basidiomycetes; Freeman *et al.* (2009) report that boron retention above 2.5kg/m$^3$ are enough to protect many wood species. Despite this, in Fig. 1 it can be observed that in no case an increase in DOT- anti leaching product retention decreased weight loss.

When applying F0 and F1 the behaviour of the wood differed slightly between species. For all treatments *P. taeda* weight losses were between 3 and 9%, whereas for *Populus* sp, were higher and in a wider range (between 4 and 12%).

After leaching, B2F1 on *P. taeda* was the only treatment that maintained mass losses below 3% and on *Populus sp* was 7%; all other samples reached weight losses of around 15% and more. These results match those presented in the literature when using other anti leaching products for boron; Mohareb *et al* (2010) reported that when applying monoglyceride as an anti - leaching in a vacuum-vacuum two stage treatment, weight losses against *Poria placenta* were around 15% and 20%.

From a statistical point of view, for both wood and fungal species, there are significant differences between the treatments with and without the anti - leaching products with a confidence level of 0.05. Leached samples treated only with DOT reached weight losses similar to those of the untreated control samples. The same happened with leached samples treated with B1F0 in both wood species, although in lower proportion. This indicates that the anti leaching products improve the wood's performance in humid environments, and that F1 is more efficient than F0.

Fig. 1 also shows that neither F0 or F1 affected the fungicidal properties of boron. Obounou-Akong *et al*. (2015) report something similar about hydrogels as anti leaching products.

CCA and CCB retention values were around those used commercially - 6.8Kg/m$^3$, minimum for a 50-year useful life (Jankowsky *et al*. 2012) - which is reflected by the low mass losses (below 3%, Fig. 1). Despite this, samples treated with CCA and CCB were sensitive to leaching, with weight losses reaching 30% (for PCCA). The curation and conditioning period (4 weeks) may not have been sufficient for the chromium to bind to the lignin, so the actives were always below their toxic thresholds for basidiomycetes, or, as many authors claim(Kartal and Lebow 2000, Shibata *et al*. 2007), because CCA tends to release its components into the environment when under unfavourable conditions.

*Table 3*

**Boron concentration (µg/mL) in the leaching water at days 1 and 9**

| | [B] µg/mL | | | [B] µg/mL | |
|---|---|---|---|---|---|
| ***Populus sp*** | **Day 1** | **Day 9** | ***P. taeda*** | **Day 1** | **Day 9** |
| CONTROL | 0.01 | 0.01 | CONTROL | 0.01 | 0.01 |
| PB1 | 119.56 | 0.78 | TB1 | 113.10 | 5.81 |
| PB1F0 | 116.31 | 0.01 | TB1F0 | 121.72 | 5.18 |
| PB1F1 | 97.96 | 1.22 | TB1F1 | 82.25 | 3.65 |
| PB2 | 169.51 | 1.01 | TB2 | 189.23 | 6.03 |
| PB2F0 | 158.58 | 1.27 | TB2F0 | 175.01 | 2.30 |
| PB2F1 | 95.82 | 0.46 | TB2F1 | 107.46 | 2.89 |
| PCCB | 16.91 | 0.01 | TCCB | 19.10 | 0.01 |

*P: Populus sp* T*: P taeda.*
**B1**: 1.4 % B; **B2**: 2.1 % B; **B1F0**: 1.4% B + F0; **B1F1**: 1.4% B + F1; **B2F0**: 2.1% B + F0; **B2F1:** 2.1% B + F1.

Mitsuhashi *et al*. (2007) claim accelerated leaching to be an insufficient method to predict preservative losses in a water diffusible system such as this one; this work verified that boron loss on day 1 was much higher than on day 9, mostly as a consequence of dilution, as expected. Baysal *et al*. (2006) report something similar when evaluating the gradual decrease of boron leached from the wood over 5 days.

Table 3 shows that on day 1, for both wood species boron loss – measured in water leaching - was ten times lower for CCB than for all the DOT treatments. This result shows that boron improves the ability of chromium to fixate the preservative compunds. For both wood species, when applying F0 with B2 (2.1% concentration) (PB2F0 and TB2F0), boron losses in the leaching water were above those of the samples without the anti- leaching product (PB2 and TB2). Boron leaches easily from the wood when the polymer is applied in low concentrations. However, the mass losses due to fungal action were below expectations.

*Table 4*

**Percentage of boron lost (%)(*) in the leaching water in day 1 of leaching**

| | % B lost | | % B lost |
|---|---|---|---|
| **PR1** | 9.1 | **TR1** | 7.4 |
| **PR1F0** | 9.1 | **TR1F0** | 7.2 |
| **PR1F1** | 5.7 | **TR1F1** | 5.3 |
| **PR2** | 9.3 | **TR2** | 7.2 |
| **PR2F0** | 10.2 | **TR2F0** | 10.9 |
| **PR2F1** | 5.5 | **TR2F1** | 4.8 |

P: *Populus sp* T*: P. taeda.*
**B1**: 1.4 % B; **B2**: 2.1 % B; **B1F0**: 1.4% B + F0; **B1F1**: 1.4% B + F1; **B2F0**: 2.1% B + F0; **B2F1:** 2.1% B + F1.
(*) Boron loss percentage was calculated as initial boron retained in the wood minus boron in the leaching water

Table 4 shows boron losses were below 10% (of initial boron) for all cases. As the boron concentration in the leaching water at day 9 was below 5µg/ml, the % of boron lost was calculated considering only day 1. The decrease of boron loss by leaching is analogous to what Tondi (2012) reports on tannins applied as an anti leaching, which is attributed to their incomplete polymerization; this suggests that the tested organic polymer (F0 and F1) may be involved in some chemical or physical interaction with the constitutive polymers of the wood, or it may simply bind to the fibres and restrict wetting.

The results show the anti leaching polymer potential at the higher concentration (F1); not only it reduces boron loss by leaching, but also can be applied mixed with boron in a single stage, reducing the operative costs of impregnation, since whenever acrylics are used, the cost of post-impregnation heat treatment will be taken for curing. Wood treated with boron and F1 could be used in environments of high biological risk (but not in contact with soil) since generally a slow and low boron loss suggests better long-term protection (Baysal *et al*. 2006). The formulation of this compound will be researched further, especially on the physical and chemical interaction between the anti-leaching F1 and the boron.

## CONCLUSIONS

This work presents preliminary results on the development of an anti leaching product that can be applied to solid wood along with borates. The organic polymer applied at the lower concentration (F1)

improved the permanence of boron inside the wood even in highly humid conditions. When combined, DOT and the anti leaching product present remarkable fungicidal properties, making it potentially suitable for outdoor use.

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