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Section 3 Wood protecting chemicals

Development of boron/linseed oil combined treatment as a low-toxic wood protection
Evaluation of boron fixation and resistance to termites according to Japanese and European standards

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ABSTRACT

Combinations of boric acid as a first step of treatment and linseed oil as a second step have been performed in order to enhance boron retention to leaching and wood resistance to termites. Classic leaching and termites resistance standards are inappropriate to evaluate this combination which can be considered as both a wood core preservation treatment and a coating.
Japanese Industrial Standard (JIS K1571, 2004) on Japanese cedar (*Cryptomeria japonica*) Japanese beech (*Fagus crenata*) exposed to subterranean termite *Coptotermes formosanus*, and, European standards EN84 and EN117 on Pine (*Pinus sylvestris*) exposed to termite *Reticulitermes santonensis* have been performed for the same boric acid/ linseed oil treatments.
Addition of oil as a water repellent to boron treated wood gave promising results with about 30% of initial boron retained. Termite mortality rates and efficiency thresholds using the different standards are determined and compared. Moreover, the relevance of Japanese mass loss indicator and European visual evaluation are discussed in the case of unconventional wood protection system such as boron/linseed oil combination.

Keywords: Wood preservation, Boric acid, Linseed oil, Leaching, Termite tests.

1. INTRODUCTION

Scientists and populations are more and more aware of the need to reduce impact of human activities on environment. Utilization of traditional wood preservatives with high environmental impact like chromated copper arsenate (CCA) is now discussed. Boron-based preservatives are considered as a good alternative for a modern and respectful wood preservation because of their low mammalian toxicity (Jansen et al. 1984, Maier and Knight 1991). Those compounds such as boric acid or borates have proved their efficiency as low-toxic wood preservatives to fungi and termites (Lloyd 1997, Drysdale 1994, Grace et al. 1992, 1994a,b). Borates are highly soluble in water and can be applied by inexpensive impregnation methods like dip-diffusion or vacuum-pressure treatments (Byrne and Morris 1997, Higley et al. 1996, Lebow and Morrell 1988). Nevertheless, borate's water-solubility makes them unsuitable to weathering, borates are easily

leached out under outdoor exposure (Cockroft and Levy 1973, Lloyd and Manning 1995, Peylo and Willeitner 1997), so they are never used without an additional protection.
Attempts to increase boron retention are investigated for few years and many methods or processes have been proposed. Many authors proposed to combine boron with other chemicals compounds to produce less leachable compounds like proteins (Thevenon et al. 1997, 1998, 2003, Polus-Ratajczak and Mazela 2004), complexes with tannins (Pizzi and Baecker 1996, Thevenon 1999), amine (Kartal and Imamura 2004, Petric et al. 2001) or fatty acids (Lyon et al. 2007a, Pizzi 1993 a,b). The simplest way to reduce boron leaching is to keep the wood under low moisture exposition, addition of hydrophobic agents or coating like varnish or paints (Homan and Militz 1995, Pavlic et al. 2001) have then proved to be efficient with some differences depending mainly on coating impermeability. Reduction of wood moisture content and mechanical barrier by oil treatment appeared to be sufficient to protect in most of the fungi (Welzbacher and Rapp 2002), exposition cases but failed to reduce termites attacks (Dumonceaud 2001, Lyon et al. 2007b). Regarding this very risk, addition of borates appeared to be efficient and necessary. Exposition to termites according to Japanese standards based on mass loss criteria made these combinations considered as efficient. European standard EN 117 is based on a visual examination of the attack level. This difference could be essential regarding borates/oil treatments lack of termites repellence. Indeed termites have to start consuming the wood for being stopped, that is not a problem considering the mass loss criteria but it could be problematic with the visual examination of EN 117.

This paper proposes a comparison between Japanese and European standards of exposition to termites for evaluation of a non conventional wood treatment including both core and surface treatment. Boron/linseed oil combinations are used for treatment of Japanese and European species, samples are weathered and exposed to termites according to the two standards testing methods.

2. EXPERIMENTAL METHODS

2.1 Preparation of wood specimens

2.1.1 Japanese Standard

Sapwood specimens of Japanese cedar *(Cryptomeria japonica* D. Don) and Japanese beech (*Fagus crenata* Blume), were prepared for vacuum-pressure impregnation at size of 20 (R) X 20 (T) X 10 (L) mm which is the size respecting the Japanese Industrial Standard (JIS) for leaching and termite tests. Specimens were oven-dried at 60°C for 72 hours and then weighed before being treated by vacuum-pressure.

2.1.2 European standard

Sapwood specimens of Pine (*Pinus sylvestris* L.) were prepared according to the European standard EN 117 requirements. Samples sizes are 15 x 25 x 50 (R, T, L) mm. Samples were oven-dried at 103°C for 24 hours to determine their anhydrous weight and then treated by vacuum-pressure.

2.2 Treatment

Samples were treated by a 30-minutes vacuum treatment at 88 kpa absolute pressure with boric acid aqueous solutions of 0.25, 0.5, 1.0 or 2.0% w/w. Specimens were then weighted in their

saturated state to determine the boric acid uptake (kg/m^3 BAE) and then reconditioned at 20°C and 50% relative humidity during two weeks until their stabilisation.
Before being submitted to an oil treatment, transversal sections of specimens were coated with epoxy resin to forbid exchanges in longitudinal direction as we want to study radial and tangential retention. Moreover, we know that longitudinal exchanges are usually blocked by a three centimetres thick oil layer. Thus, the treatment result is an application of an oil layer of about 1.5 mm thick on tangential and radial surfaces, the inner part of wood pieces staying free from oil.
Oil treatment consisted in a double dipping of one hour each; first in a hot bath at 130°C and second in a cold bath at 80°C. Linseed oil, soybean oil and rapeseed oil are evaluated regarding boron retention increase provided.

2.3 Leaching procedure

2.3.1 Japanese Standard

Leaching test was conducted according to the Japanese Industrial Standard K 1571 (JIS 2004) to reproduce the effect of weathering. This procedure involves a cycle of immersion-drying during ten days. Specimens were soaked in distilled water (10 volumes of water for 1 volume of wood) at 27°C under magnetic stirring at 400-500 rpm during 8 hours and then oven-dried at 60°C during 16 hours. Leaching water is replaced after keeping a specimen for boron concentration analysis and this procedure is repeated ten times.
We followed the amount of leached boron along the leaching procedure using Inductively Coupled Plasma analysis (ICP) of leachates. Analyses of remaining boron amount before and after leaching test were realized after wood specimens'extraction in hot water at 80°C for 2 hours

2.3.2 European standard

Leaching procedure for European specimens was conducted according to the European Standard EN 84. Specimens were soaked in distilled water (5 volumes of water for 1 volume of wood) under 15-minutes vacuum at 4 kpa absolute pressure. After returning to atmosphere pressure, water was changed nine times within the next 14 days including one change at the end of first and second day, and, without more than 3 days between two water changes.

2.4 Termite resistance tests

2.4.1 Japanese Standard

Untreated control, leached and unleached specimens were exposed to subterranean termites (*Coptotermes formosanus* Shiraki) in a no-choice feeding test according to the Japan Industrial Standard K 1571 (JIS 2004). Specimens were placed at the bottom of an acrylic cylindrical container (8 cm diameter and 6 cm high) blocked at one end with dental plastone to form a strong 5 mm thick barrier. 150 termite workers and 15 soldiers were collected and placed with each wood specimen. Those containers were then placed in large plastics boxes papered with humid cotton pieces and sealed to keep high moisture conditions for testing. Those boxes were kept at 28 °C and 85% RH during three weeks. Termites'mortality was recorded every week and mass losses due to termite attack were calculated by weighting each sample after three weeks of exposure. A mass loss under 3.0% is enough to consider the treatment as effective.

2.4.2 European standard

Untreated control, leached and unleached specimens were exposed to termites *Reticulitermes santonensis* (Oleron island, France) according to European Standard EN117. Each specimen was placed in a glass jar containing 4 centimetres of sand and a small plastic cylinder as a sample support to keep the specimens up on the sand. 250 termite workers, 3 nymphs and 3 soldiers were introduced with each specimen. Glass jars were kept at 27°C and 80% RH for 8 weeks. Termites' activity was checked every week and termites mortality calculated after 8 weeks.
Degree of attack was determined after 8 weeks by using the EN117 visual value ranging from 0 to 4 based on the following categories :

0: No attack. 1: Attempted attack 2: Slight attack. 3: Average attack. 4: Strong attack.

3. RESULTS AND DISCUSSION

3.1 Retention of boron

Results in table 1 indicate the boron retention of boric acid and combined treated specimens after leaching test performed according to the Japanese standard. Addition of oil to boric acid treated wood increases boron retention of 16.7 to 33.5 % of initial boron introduced compared to the specimens of same boron concentration without oil.
Boron retention provided by oil is higher when initial boron concentration increases with a difference of about 7% for example for linseed oil between 0.25 and 1.00% of boric acid.
Type of oil has also an impact on boron retention. Linseed oil seems more efficient to prevent boron leaching than soybean oil and much better than rapeseed oil. Boron retention by linseed oil is about 5% higher than for rapeseed oil. This fact could be explained by the permeability of oil film. Poly-unsaturated linseed oil forms a more impermeable layer after oxidative drying than more unsaturated soybean and rapeseed oil.

There are also some differences between C. *japonica* and F. *crenata*. Initial retention loads are lower for F. *crenata* than for C. *japonica* and it decreases percentages of boron retained after leaching. This difference of initial retention load is highlighting the impact of specimens' dimensions. Retention of specimens treated with boric acid only are close to zero after leaching so we can not really evaluate the impact of addition of oil which could be higher for bigger wood pieces. This could also be a source of differences with specimens required for European standard which are bigger than those for Japanese standard.

Table 1: Amount of boric acid (BAE kg/m^3) leached after a 10-days leaching test based on Japan Industrial Standard K 1571 (JIS 2004) on boric acid/oil treated sapwood specimens (L = linseed oil, S = soybean oil, R = rapeseed oil)

Cryptomeria japonica	Retention load of unleached samples (Kg/m^3 BAE) (1)	Boron retained after leaching (Kg/m^3 BAE) (2)	% increase compared to control specimens
Treatment			
0.25% control	2.15 (0.28)	0.14 (0.11)	-
0.25 % / L	2.02 (0.14)	0.67 (0.22)	26.7
0.25 % / S	1.91 (0.13)	0.61 (0.14)	25.4
0.25 % / R	2.09 (0.20)	0.55 (0.09)	19.8
0.50 % control	4.25 (0.11)	0.13 (0.11)	-
0.50 % / L	4.13 (0.17)	1.48 (0.10)	32.8
0.50 % / S	4.39 (0.15)	1.44 (0.08)	29.8
0.50 % / R	4.23 (0.09)	1.27 (0.10)	27.0
1.00 % control	8.03 (0.09)	0.21 (0.04)	-
1.00 % / L	8.36 (0.18)	3.02 (0.11)	33.5
1.00 % / S	7.94 (0.21)	2.80 (0.12)	32.6
1.00 % / R	8.07 (0.16)	2.54 (0.07)	28.8

Fagus crenata	Retention load of unleached samples (Kg/m^3 BAE) (1)	Boron retained after leaching (Kg/m^3 BAE) (2)	% increase compared to control specimens
0.25% control	1.74 (0.12)	0.07 (0.08)	-
0.25 % / L	1.68 (0.21)	0.40 (0.18)	19.8
0.25 % / S	1.80 (0.09)	0.39 (0.07)	17.6
0.25 % / R	1.69 (0.14)	0.35 (0.11)	16.7
0.50 % control	3.55 (0.24)	0.11 (0.12)	-
0.50 % / L	3.55 (0.09)	0.82 (0.24)	20.0
0.50 % / S	3.37 (0.12)	0.79 (0.11)	20.4
0.50 % / R	3.58 (0.20)	0.72 (0.15)	17.1
1.00 % control	7.16 (0.07)	0.22 (0.11)	-
1.00 % / L	6.97 (0.13)	1.70 (0.09)	21.3
1.00 % / S	6.92 (0.09)	1.65 (0.15)	20.8
1.00 % / R	7.14 (0.14)	1.59 (0.10)	19.2

(1) Average of 20 specimens
(2) Average of 10 specimens

As the linseed oil showed to be more efficient than soybean oil and rapeseed oil to prevent boron leaching, we performed termite resistance test according to Japanese and European standards on combined boric acid/linseed oil treated wood.

3.2 Termite resistance tests

3.2.1 Japanese Standard

Figure 1 indicates mass losses of specimens treated with combined boric acid/linseed oil after exposition to *Coptotermes formosanus* termites according to Japanese Industrial Standard.

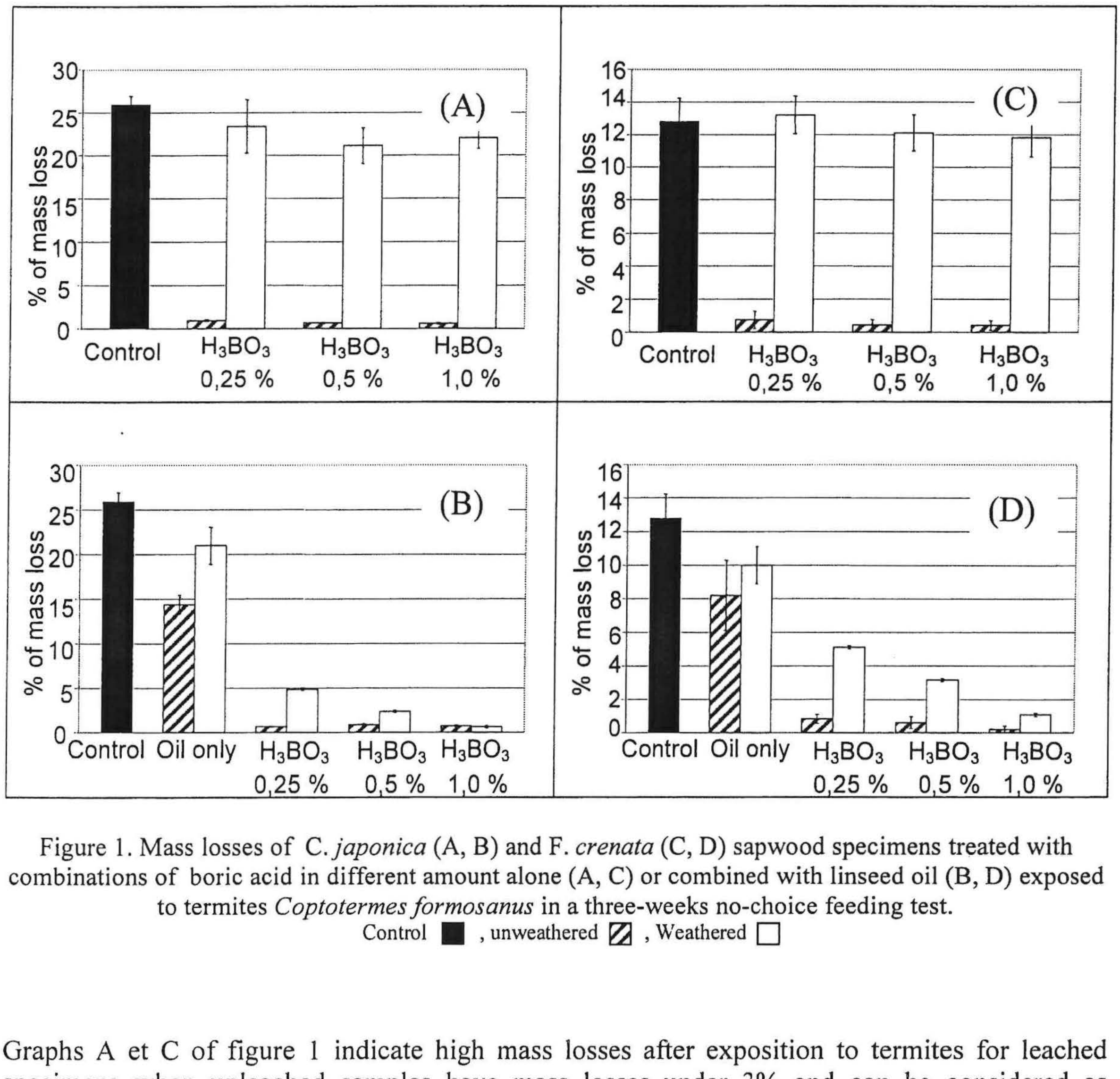

Figure 1. Mass losses of C. *japonica* (A, B) and F. *crenata* (C, D) sapwood specimens treated with combinations of boric acid in different amount alone (A, C) or combined with linseed oil (B, D) exposed to termites *Coptotermes formosanus* in a three-weeks no-choice feeding test.
Control ■ , unweathered ▨ , Weathered □

Graphs A et C of figure 1 indicate high mass losses after exposition to termites for leached specimens when unleached samples have mass losses under 3% and can be considered as resistant according to standard. This result confirms high leachability of boron compounds due to their water solubility. Specimens treated with linseed oil only have mass losses of 14.4 to 21.0 % for C. *japonica* and 8.21 to 10.0 for F. *crenata* when control are respectively 25.9 and 12.8 % for those species. Oil treatment reduces mass losses but is enable to protect the wood against termites. This confirms retardant effect of oil but vegetable oils are non toxic and non repellent to termites (Nunes et al. 2006).

Addition of linseed oil to boric acid treated wood decreases mass losses of specimens as initial boron content increases. Those observations and weak efficiency of oil-alone treatment show that efficiency to termites is due to boron retention by linseed oil.

Table 2 indicates termites'survival rates after exposure. Those results confirm the non toxicity of linseed oil with about 82.1 % of survival after exposure when this rate is 87.9 % for control specimens. Survival rates decrease as boron concentrations increase.

Table 2: Termites survival in boric acid/linseed oil treated *C. japonica* and F. *crenata* sapwood specimens exposed to a termite-resistance test of three weeks based on Japan Industrial Standard (JIS) K 1571 (2004) .

Cryptomeria japonica	**Termites' survival rate (%), mean (SD) (1)**	
Treatment	**Leached**	**Unleached**
Linseed oil	82.1 (3.15)	54.5 (4.71)
0.25% control	81.3 (4.24)	24.6 (3.01)
0.25 % / Linseed	62.7 (2.20)	23.4 (4.21)
0.50 % control	67.3 (5.41)	0.00 (0.00)
0.50 % / Linseed	52.1 (1.20)	0.00 (0.00)
1.00 % control	70.1 (4.11)	0.00 (0.00)
1.00 % / Linseed	1.90 (0.33)	0.00 (0.00)
Untreated control	87.9 (3.28)	

Fagus crenata	**Termites' survival rate (%), mean (SD) (1)**	
Treatment	**Leached**	**Unleached**
Linseed oil	78.6 (2.25)	61.0 (2.13)
0.25% control	74.3 (3.00)	59.8 (2.12)
0.25 % / Linseed	68.2 (2.20)	51.8 (6.05)
0.50 % control	86.6 (2.23)	0.00 (0.00)
0.50 % / Linseed	70.2 (5.10)	0.00 (0.00)
1.00 % control	73.3 (2.21)	0.00 (0.00)
1.00 % / Linseed	50.9 (3.29)	0.00 (0.00)
Untreated control	90.1 (4.42)	

(1)Average of 5 replicate specimens
Values in parenthesis are standard deviations.

3.2.2 European standard

Table 3 shows results of termite test performed according to European method EN117.

Evaluation of combined boric acid/linseed oil treatment effect according to European standard confirms results of Japanese test for some aspects. Linseed oil treated specimens without boric acid, leached or unleached, at retention load of 180 to 230 kg/m^3 present severe attacks. Attack level is 4 any time. Termites survival is about 45 to 50% when it is 67.4 % for control untreated specimens. This confirms the non toxicity of linseed oil to termites but there is a first difference with Japanese experiments where the survival rates were almost equal between oil treated and control samples. We explain this difference by oil retention load higher for *Pinus sylvestris* in conditions of European standard (180-230 kg/m^3) than for *Cryptomeria japonica* in Japanese standard (140-160 kg/m^3).

Attack level decreases as boron retention increases. Attack level is 3 (80%) to 4 (20%) for boric acid concentration of 0.5% and 2 (20%) to 1 (80%) for a concentration of 2.00 %. There is also a difference with Japanese standard in this case. Efficient boric acid concentration is about 2.00% for European standard when mass loss of Japanese cedar and Japanese beech are below 3% for a boric acid concentration of 0.25%. Efficient retention load is about 1.5 to 2.5 kg/m^3 BAE for Japanese standard and about double, 5.0 kg/m^3 BAE for European standard. Leached specimens are severely attacked like for Japanese experiments.

Addition of linseed oil to boric acid treated specimens reduces attack level and increases termites'survival rates but an initial boric acid concentration of 2.00% is enable to prevent termite attack after weathering. Efficiency of combined treatment is lower according to European standard than Japanese standard whereas we could expect a better efficiency to due higher boron retention of big sized samples.

Table 3: Boron retention load, maximal and minimal attack levels and termites survival in boric acid/linseed oil treated *Pinus sylvestris* sapwood specimens exposed in a no-choice test to *Reticulitermes santonensis* according to EN117

Treatment	Retention load of unleached samples (kg/m^3 BAE)	Minimal attack level (% frequency)	Maximal attack level (% frequency)	Termites' survival rate (mean %)
H_3BO_3 0.5 %	1.21	3 (80)	4 (20)	0
H_3BO_3 0.5 % leached	-	4	4	64.8
H_3BO_3 1.0 %	2.57	2 (40)	3 (60)	0
H_3BO_3 1.0 % leached	-	4	4	55.5
H_3BO_3 2.0 %	5.07	1 (80)	2 (20)	0
H_3BO_3 2.0 % leached	-	4	4	38.4
Linseed oil	-	4	4	45.0
Linseed oil leached	-	4	4	48.1
Linseed oil + H_3BO_3 0.50 %	1.19	2 (40)	3 (60)	0
Linseed oil + H_3BO_3 0.50 % leached	-	3	3	32.2
Linseed oil + H_3BO_3 1.0 %	2.55	2 (60)	3 (40)	0
Linseed oil + H_3BO_3 1.0 % leached	-	3	3	15.1
Linseed oil + H_3BO_3 2.0 %	5.21	0 (20)	1 (80)	0
Linseed oil + H_3BO_3 2.0 % leached	-	2 (20)	3 (80)	0
Control	-	4	4	67.4

This difference comes mainly from the objectives of the two standard testing methods. Japanese method evaluates degradation with potential damages for mechanical resistance of wood whereas European method considers esthetical aspects as well. A slight erosion of 1.5 mm on a 10th of the total surface of a sample is considered as a moderate attack according to EN117 and treatment is then considered as inefficient. This degradation represents a mass loss of about 1 to 2 % and could probably been considered as negligible to Japanese standard.

4. CONCLUSIONS

Combined boric acid/linseed oil treatments have shown a good potential as low toxic wood preservatives with increase of boron retention of about 30% depending on the type of oil and decreases of mass losses after exposition to termites compared to both acid boric or linseed oil treatment performed separately. However efficiency thresholds for those treatments can be

severely discussed. By using Japanese standard, toxicity threshold appeared for utilization of a 1.0% boric acid concentrated solution whereas a solution of 2.0% was not efficient enough according to EN117 because of esthetical aspects taken into accounts in European standard.
Those experiments adapted from different standards indicate the lack of reliable standard procedure for testing biocide core treatment and coating at the same time. Indeed EN 559 classifying performances of wood preservative considers that leaching test is unnecessary if biocide is under a coating without considering coating permeability.
Preservation efficiency, low environmental impact and easy transfer to industry of these treatments already performed separately constitute a great potential for their utilization on wood preservation market.

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