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Effect of an oil heat treatment on the leachability and biological resistance of boric acid impregnated wood*

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Abstract – Vegetable oils provide boron retention of about 30% of initial amount depending on oil drying properties. Linseed oil is the most efficient, followed by soybean oil and rapeseed oil. Durability of *C. japonica* and *F. crenata* wood specimens has been enhanced by application of linseed oil alone but not enough to reduce termite's attack of *Coptotermes formosanus*. Treating wood with a 1.0% w/w boric acid solution prior to oil treatment protects *C. japonica* from termite and fungi degradations. Efficiency against termites is mainly due to boron retention by oil but hydrophobic oil also forms a barrier decreasing fungi penetration. Boron efficacy threshold around 0.7 kg/m³BAE, lower than classical boron treatments thresholds indicates that oil water-repellence reinforces boron biostatic effect.

boric acid / vegetable oil / wood preservation / termite test / fungi test

Résumé – Impact d'un traitement oléothermique sur la durabilité du bois imprégné à l'acide borique. La recherche en préservation du bois se tourne à présent vers des produits à faibles impacts environnementaux. Nous avons étudié l'opportunité d'associer borates et huiles végétales pour accroître la rétention du bore ainsi que la résistance du bois aux termites et aux champignons xylophages. Les huiles végétales permettent de retenir environ 30 % du bore initial selon leurs propriétés de séchage. Ainsi, l'huile de lin est plus efficace que les huiles de soja et de colza. La durabilité d'éprouvettes d'aubier de *C. japonica* et *F. crenata* est améliorée par l'ajout d'huile de lin mais trop peu pour éviter les attaques de termites *Coptotermes formosanus*. En revanche, la combinaison d'une imprégnation par une solution d'acide borique à 1.0 % m/m suivi d'une traitement à l'huile protège efficacement *C. japonica* contre les termites et les champignons. Alors que son efficacité contre les termites semble principalement due à la rétention du bore, l'huile semble former une barrière efficace contre la pénétration des champignons xylophages. Les seuils d'efficacité d'environ 0.7 kg/m³BAE, plus faible que ceux enregistrés pour les traitements classiques au bore indique que l'huile renforce l'effet biostatique du bore.

acide borique / huiles végétales / préservation / résistance aux termites / résistance aux champignons

1. INTRODUCTION

Because of environmental care, utilization of traditional preservatives like CCA for all applications is now discussed. Preservation policies are now directed towards fit-for-purpose to provide a preservative adapted to each situation of wood exposure [30]. For those reasons, boron-based preservatives are considered as a good alternative regarding their low mammalian toxicity [7, 8, 13, 29, 33]. Boron compounds have a broad spectrum effectiveness against wood degrading organisms like fungi and termites as well as a low environmental impact [1, 28]. However borates can easily be leached out from wood under outdoor exposure conditions because of their high solubility into water [3, 10, 21].

To reduce boron leaching different strategies are reported in literature like utilization of borates under low moisture condition, chemical combination to produce less leachable compounds like proteins [31], complex with tannins [24], ester borates [17] or amine [9, 20]. Addition of hydrophobic agents or coating like varnish or paint has also been considered with various efficiency depending on coating permeability [20]. Most authors consider that boron retention efficiency of such combinations is around 15 to 50% of initial boron retained [16, 17, 20].

We observe since a few years an increasing interest in the wood treatment involving vegetable oil [14, 15, 18] or natural-oils derived of fatty acids [12, 22, 23]. Following Rapp (2001) [25] this technique will be named here "oil heat treatment" Water repellence provided by hydrophobic oil reduces wood equilibrium moisture content and leaching of extractives like tannins, thus decreasing insects attacks and fungi decay [34]. This type of treatment also provides a good dimensional

* Tables I to III are only available in electronic form at <http://www.afs-journal.org>.

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stability [25–27] and when it is performed on green wood, allows to dry the wood with less peripheric cracking compared to air-drying [2,4]. However oil-heat treatments are sometimes inefficient to prevent insect attacks and fungi decay, especially for sapstain and mold fungi because of their non toxicity to those organisms [19].

This paper deals with the possibility of combining a boron impregnation and an oil-heat treatment to improve wood durability. Three vegetable oils varying by their drying properties and therefore permeability of their dry film are compared as boron retention agent. The impact of these combinations is evaluated for boron retention and biological resistance to termites and decay fungi.

2. MATERIALS AND METHODS

2.1. Wood material and chemicals

Sapwood specimens of Japanese cedar (*Cryptomeria japonica* D. Don) and beech (*Fagus crenata* Blume), have been prepared for vacuum-pressure impregnation using the following dimensions:

- Retention test: 50 (L) × 25 (T) × 15 (R) mm
- Termites test: 20 (L) × 10 (T) × 10 (R) mm
- Fungi test: 40 (L) × 20 (T) × 5 (R) mm

Those sizes based on Japanese Industrial Standard (JIS) for leaching, decay and termite tests are usually recommended for surface treatment. As our treatment combines both impregnation and surface treatments, we have decided to use surface-test samples and submit them to leaching procedure made for impregnation treatment. Thus specimens were oven-dried at 60 °C for 72 h and then weighted before being treated by a 30-min vacuum treatment at 88 kpa absolute pressure with boric acid aqueous solutions of 0.5, 1.0, 2.0 or 5.0% w/w. Specimens were then weighted in their saturated state to determine the boric acid uptake (kg/m³ boric acid equivalent. cf. formula below) and then reconditioned at 20 °C and 50% relative humidity during two weeks until stabilisation.

$$BA = (W_{imp} - W_0) \times \frac{\%_{sol}}{V}$$

BA: boric acid uptake (kg/m³ BAE) W_{imp} : impregnated weight (kg)
 W_0 : oven dried weight (kg) $\%_{sol}$: % H₃BO₃ of treatment solution w/w
 V: sample volume (m³).

Before being submitted to an oil-heat treatment, the transverse sections of the 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 oil layer. Thus treatment result is application of an oil layer of about 1.5 mm thick on tangential and radial surfaces. Inner part of wood pieces is free from oil.

Oil heat 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. Rapeseed oil, soybean oil and linseed oil were used as treatment oil separately. Linseed oil is considered as a drying oil due to its high content in polyunsaturated fatty acids like linolenic acid (45 to 70%) and linoleic acid (12 to 24%) or monounsaturated acid like oleic acid (10 to 21%). Soybean oil is a semi-drying oil presenting a lower unsaturation degree compared to linseed oil because of its lower content

in polyunsaturated fatty acids (40 to 50%). Rapeseed oil is the less drying oil with about 60% of oleic acid and 15% of saturated fatty acids. Impact of these drying properties has been investigated regarding boron retention and biological resistance of wood to degradation organisms.

2.2. Leaching procedure

Leaching procedure was conducted according to the Japanese industrial Standard (JIS) K 1571 (JIS 2004) [35] for impregnation treatment to reproduce the effect of weathering. This procedure involves a cycle of immersion-drying during ten days. Specimens were soaked in distilled water (10 vol. water for 1 vol. wood) at 27 °C under magnetic stirring at 400-500 rpm for 8 h and then oven dried at 60 °C for 16 h. Leaching water is replaced after keeping a sample for boron concentration analysis and this procedure is repeated ten times.

As boron is the preservative element of the mix, we followed the amount of leached boron along the leaching procedure by using Inductively Coupled Plasma analysis (ICP) of leachates, leached and unleached samples. Analyses of boron amount remaining before and after leaching were realized after specimen's extraction in hot water at 80 °C during 2 h.

2.3. Termite test

Untreated control, leached and unleached wood specimens were exposed to subterranean termites (*Coptotermes formosanus* Shiraki) in a no-choice feeding test according to the Japan Industrial Standard (JIS) K 1571 (2004) [35]. 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 barrier. Acrylic containers were then placed in large plastic boxes papered with humid cotton pieces and sealed to keep high moisture conditions for testing. One hundred fifty termite workers and fifteen soldiers were collected and placed with each specimen. The full system was kept at 28 °C and 85% RH for three weeks. Termite mortality was recorded every week and mass losses due to termite attack were calculated by weighting for each specimen after three weeks of exposure.

2.4. Decay test

Specimens were exposed in monocultural fungi test performed according to Japan Industrial Standard (JIS) K 1571 (2004) [35] using a brown-rot fungus *Fomitopsis palustris* (Berkeley et Curtis) Murrill (FFPRI 0507) and a white-rot fungus *Trametes versicolor* (L. ex Fr.) Quel. (FFPRI 1030). Liquid fungal culture have been prepared by inoculation of a solution containing 40 g of glucose, 3 g of peptone, 15 g of malt extract and 1 000 mL of distilled water. Those cultures were then shaken at 26 °C for 10 days at 100 rpm.

Glass jars containing 250 g of quartz sand were wetted with nutrient aqueous solution as follow and then sterilized by autoclave: for White rot fungi, 85 mL of a solution containing 40 g of glucose, 3 g of peptone, 15 g of malt extract and 1 000 mL of distilled water; for Brown rot fungi: 80 mL of a solution containing 20 g of glucose, 1.5 g of peptone, 7.5 g of malt extract and 1 000 mL of distilled water.

After inoculating glass jars with fungal cultures and waiting 10 days, three samples of the same condition sterilized using gaseous

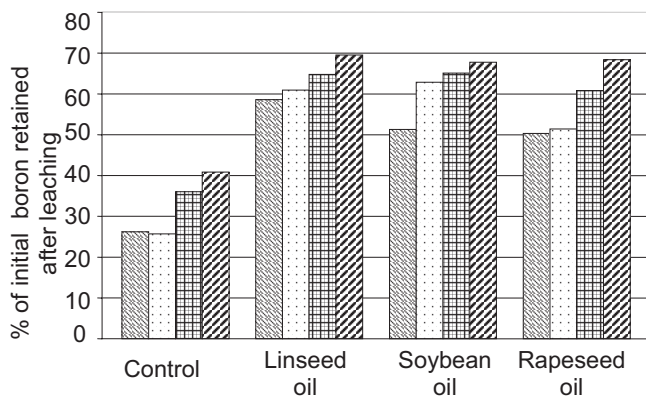


Figure 1. Boron retention after leaching provided by addition of linseed, soybean or rapeseed oil in *C. japonica* specimens treated with boric acid solution of 0.5, 1.0, 2.0 or 5.0 % w/w (cf. Tab. I at www.afs-journal.org).

ethylene oxide were placed in each jar. Tests jars were finally incubated at 26 °C for 12 weeks. Mass loss of specimens was calculated as the difference of final and initial oven-dried weight of each sample.

3. RESULTS AND DISCUSSION

3.1. Retention results

Oil penetration depth has been evaluated around 1.5 mm. Considering this depth, oil retention provided by this treatment is about 140 to 160 kg/m³ into the first millimetre from the surface. Figure 1 indicates that vegetable oils increase boron retention in wood of about 25 to 40% depending on the type of oil. Linseed and soybean oils are more efficient to prevent boron leaching than rapeseed oil. This fact may probably be explained by oil composition and especially their insaturation degree expressed through the iodine value (IV) which is the iodine quantity fixed on 1g of fat. The higher this iodine value, the higher the insaturation degree. When insaturation degree is high like for linseed oil (IV = 186), drying process is fast and fully completed to form a surface layer. When insaturation degree is low like for rapeseed oil (IV = 105), drying process is slow and the layer formed is less impermeable than in the case of linseed oil. Soybean oil (IV = 138) has an intermediate behaviour regarding drying and consequently regarding boron leaching.

Except a small increase of retention for wood treated with a 1.0% boric acid solution, boron concentration has no impact on amount of boron leached (Tab. I, available online at www.afs-journal.org).

3.2. Termite resistance

Mass losses after a three-weeks exposure to *Coptotermes formosanus* termites are indicated in Figure 2. Control samples have lost about 26% of their initial weight for *C. japonica* and 13% for *F. crenata*. Results for both species indicate

that the combination of boric acid and vegetable oil is the only solution to reach a durable state. Indeed we know that a treatment involving only boron could not protect wood because of leaching and results in Figure 2 show that application of vegetable oil alone reduces about half of mass losses in unleached state but just a few percent after leaching. An unexpected result occurs for rapeseed oil which seems to be a bit efficient in both leached and unleached states.

Boron retention around 30% of initial level for *C. japonica* and 20% for *F. crenata* provided by addition of vegetable oil makes the wood able to resist to termite attack when boric acid solution concentration is high enough. Resistance for both species is provided by application a boric acid solution of 1.0% w/w followed by an oil heat treatment. Retention level recorded after leaching and efficient against termites is around 1.8 kg/m³ BAE for *F. crenata* and *C. japonica*. Determination of this efficiency threshold is based on specimen mass loss which have to be lower than 3% compared to initial mass. This result a bit low but close to the bibliographic threshold for boric acid indicates a reduced impact of borate and vegetable oil combination regarding wood durability to termites. Efficiency is mainly due to boron retention by vegetable oil application and termite mortality is confirming this tendency even if for 0.5% boric acid / oil-treated *C. japonica* samples, low mortality percents compared to boron amount indicate a lack of accessibility of boron or weak termite attack due to oily aspect of wood (Tab. II, available online at www.afs-journal.org).

3.3. Decay resistance

Masses losses of *C. japonica* and *F. crenata* sapwood specimens after a 12-weeks exposure to white-rot fungus *Trametes versicolor* and brown-rot fungus *Fomitopsis palustris* are indicated in Figures 3 and 4 respectively.

The three vegetable oils seem efficient when used alone against *Trametes versicolor* on *C. japonica*. In the case of *F. crenata*, they reduce mass losses by about 80% compared to control sample but this improvement is not sufficient with about 8% of mass loss left (Tab. III, available online at www.afs-journal.org).

Addition of boric acid is then useless for *C. japonica* but becomes unavoidable for *F. crenata* and treating wood with a 1.0% w/w boric acid solution for *F. crenata* prior to oil treatment with one of the three oils reduces mass losses to reach durable state with less than 3% of mass loss after 12 weeks of exposure. Mass losses are following boric acid amount remaining in wood, consequently linseed oil provides a better protection than soybean oil. Rapeseed oil is the less efficient.

Results for *Fomitopsis palustris* are more complex than for *Trametes versicolor*. When used alone, vegetable oil provides a “pass or fail” behaviour to wood pieces. A number of samples can be considered as durable when others are highly non durable. This behaviour seems correlated with drying properties of oil.

In unleached state, 100% of *C. japonica* samples dipped in linseed oil are durable when only 55 % and 45% for soybean and rapeseed oil respectively. These percentages decrease with

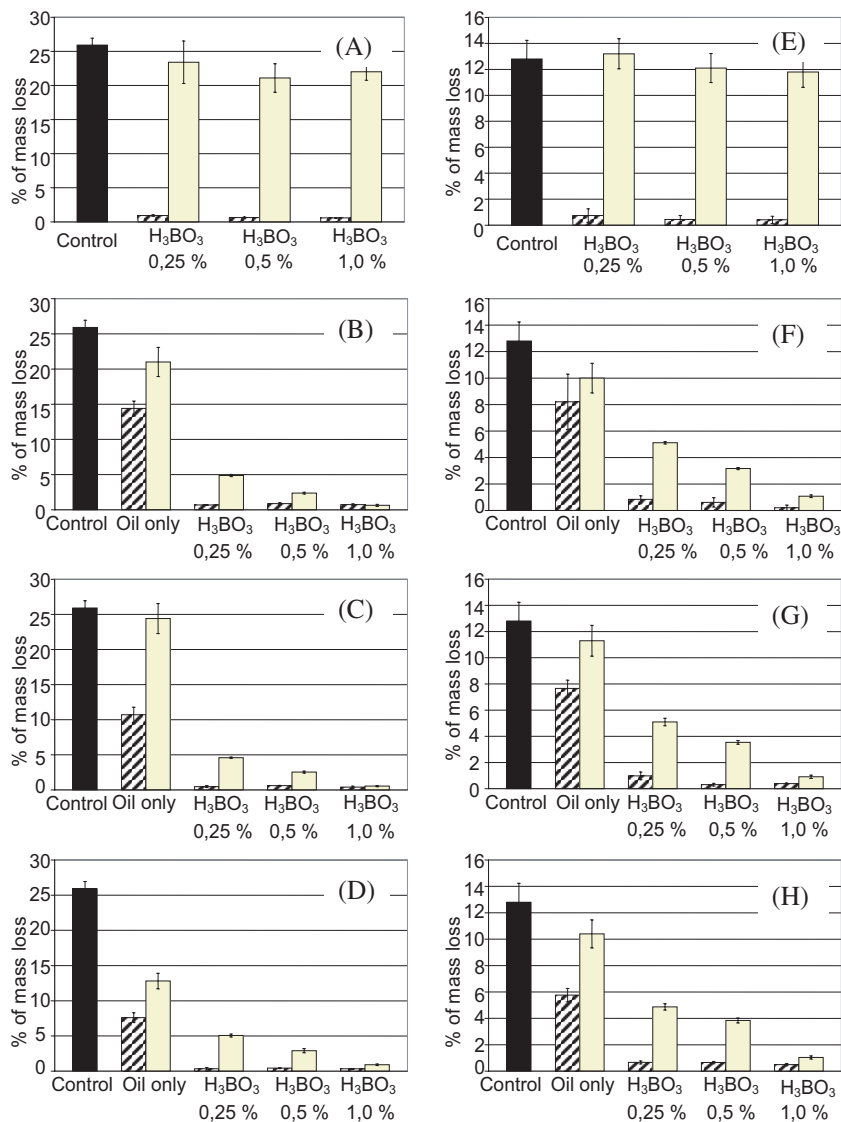


Figure 2. Mass losses of *C. japonica* (A, B, C, D) and *F. crenata* (E, F, G, H) sapwood specimens treated with combinations of boric acid in different amount alone (A,E) or combined with linseed oil (B, F), soybean oil (C, G) or rapeseed oil (D, H) exposed to termites *Coptotermes formosanus* in a three-weeks no-choice feeding test according to Japanese industrial standard (JIS) K 1571 (JIS 2004). Mass losses of control (■), unleached (▨) and leached (■) samples after exposure (cf. Tab. II at www.afs-journal.org).

leaching. Indeed, 66%, 22% and no samples are considered as durable after leaching for linseed, soybean and rapeseed oil respectively. In those cases, non durable samples reach the highest mass losses directly, indicating that oil probably acts as a barrier against fungi penetration. Efficiency of this barrier is then logically depending on oil drying properties. In the case of *F. crenata* samples, linseed oil alone seems able to provide durable sample. Soybean and rapeseed-oiled samples are always non durable even in their unleached state.

Addition of boric acid is then interesting to reduce influence of oil barrier permeability to *Fomopsis palustris*. For *C. japonica* samples, a 0.5% w/w boric acid solution is efficient enough to reach durable state even if boron retention is only around 0.7 kg/m³ BAE. This result a bit lower than classical threshold of 0.9 to 1.5 kg/m³ for decay fungi suggests a combined effect of boron and oil to provide durability. In

the case of *F. crenata*, efficiency threshold is still higher than for *C. japonica*. Addition of a 1.0% w/w boric acid solution increases resistance but not enough to provide durability.

Using a 0.5% w/w boric acid solution prior to oil-heat treatment is finally efficient to protect *C. japonica* against decay fungi. Linseed oil provides the best solution but soybean or even rapeseed oil can be efficient enough.

For *F. crenata*, combination of a 1.0% w/w boric acid solution prior to oil-heat treatment with linseed oil is the only solution to protect specimens against decay fungi. A solution for using soybean and rapeseed would be to increase boric acid content after leaching and protect *F. crenata* sapwood. Treating wood with a 1.5% boric acid solution instead of 1.0% could be efficient enough but we have to think about environmental impact of losing more than 60% of boron after leaching for highly concentrated samples.

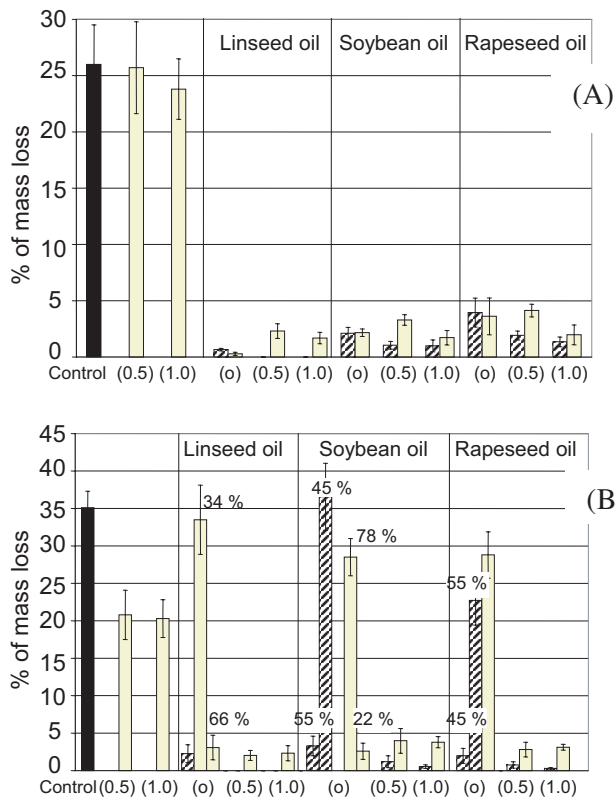


Figure 3. Mass losses of *C. japonica* (A) and *F. crenata* (B) sapwood specimens treated with combinations of boric acid alone or combined with linseed, soybean or rapeseed oil, after a 12 weeks exposure to white-rot decay fungus *Trametes versicolor* according to Japanese industrial standard (JIS) K 1571 (JIS 2004). Mass losses of control ■, unleached □ and leached samples ▨ after exposure. Percentages indicated are the % of specimens in the lot concerned by the column below. (o): Oil only. (0.5): 0.5 % H₃BO₃ w/w in treatment solution. (1.0): 1.0 % H₃BO₃ w/w in treatment solution (cf. Tab. III at www.afs-journal.org).

4. CONCLUSION

An oil heat treatment consisting in a hot bath around 130 °C followed by a cold bath around 80 °C provides a retention of about 30% of initial boron after wood leaching. This retention level is not depending on boron initial amount but on type of vegetable oil and can be compared to most coating products which present boron retention efficiency of about 15 to 50% [16, 17, 20]. Vegetable oils efficiencies seem to depend on their drying properties [16, 20]. High insaturation degree of linseed oil leads to a low permeability to water compared to other oils. This fact could explain the highest efficiency of linseed oil followed by soybean and rapeseed oil. After testing combinations of boric acid in different concentrations with the three vegetable oils, it appears that a treatment involving impregnation by a 1.0% w/w boric acid solution followed by an oil heat treatment with linseed oil is able to protect *C. japonica* and *F. crenata* sapwood specimens against *Coptotermes formosanus* termites as well as brown-rot fungus *Fomitopsis palustris* and white-rot fungus *Trametes versicolor*. Soybean

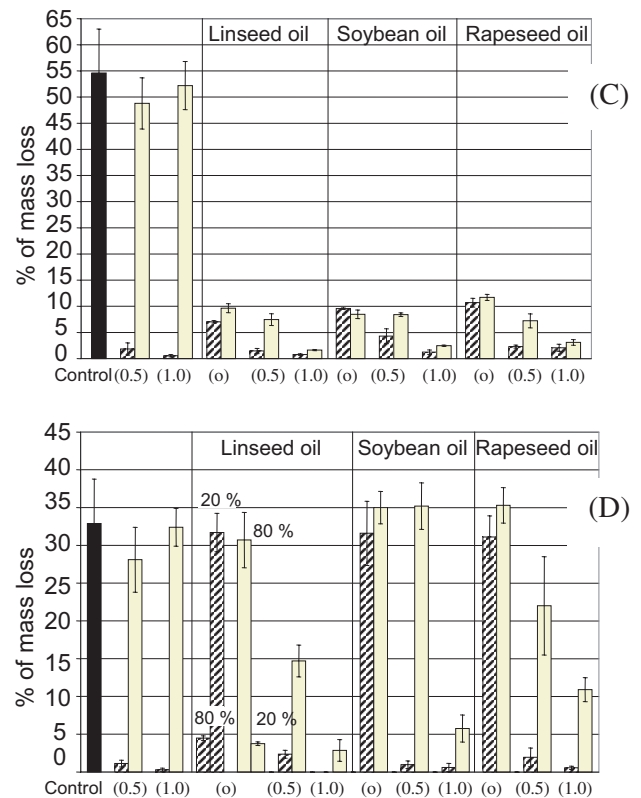


Figure 4. Mass losses of *C. japonica* (C) and *F. crenata* (D) sapwood specimens treated with combinations of boric acid alone or combined with linseed, soybean or rapeseed oil, after a 12 weeks exposure to brown-rot decay fungus *Fomitopsis palustris* according to Japanese Industrial standard (JIS) K 1571 (JIS 2004). Mass losses of control ■, unleached □ and leached samples ▨ after exposure (cf. Tab. III at www.afs-journal.org). Percentages indicated are the % of specimens in the lot concerned by the column below. (o): Oil only. (0.5): 0.5 % H₃BO₃ w/w in treatment solution. (1.0): 1.0 % H₃BO₃ w/w in treatment solution.

oil and rapeseed oil have proved to be efficient when combined with a 1.0% boric acid treatment except against *Fomitopsis palustris*.

Tests on *C. japonica* revealed an interesting combined effect of oil and boron. Indeed whereas termites are only kept away by high boron content close to those found in previous studies on *Coptotermes formosanus* [5, 6] combination of vegetable oil and boron is efficient against decay fungi for toxicity threshold of 0.7 kg/m³ BAE, a lower threshold compared to the 0.9–1.5 kg/m³ BAE found in previous studies on boron efficiency against those fungi [11, 32]. This effect may be due to hydrophobic effect of oil which reduces wood moisture content. High moisture requirement of decay fungi do not allowed them to grow in such conditions. Moreover, some authors report that fungi require a minimal free pore space for growing. Oil efficiency could then be explained by overfilling the lumen [27].

This succession of classical treatments providing durability and using products of weak toxicity for animals and plants

could be a good alternative to classical treatments presenting strong environmental impacts.

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