Australian Government
Forest and Wood Products Research and Development Corporation

MANUFACTURING & PRODUCTS

PROJECT NUMBER: PN04.2007

Combining fire retardant and preservative systems for timber products in exposed applications

JUNE 2006

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Combining fire retardant and preservative systems for timber products in exposed applications

Prepared for the

Forest and Wood Products
Research and Development Corporation

by

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The FWPRDC is jointly funded by the Australian forest and wood products industry and the Australian Government.

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Final report received by the FWPRDC in June 2006

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EXECUTIVE SUMMARY

The primary aim of this project was to develop a 'proof of concept' for a dual fire retardant/wood preservative (FR/WP), one-step treatment technology for softwood and/or hardwood species that were of low natural durability, with the focus on outdoor, above ground applications. The treatment should satisfy AS 3959 – 1999, Construction of buildings in bushfire-prone areas and at the same time comply with the preservation standard, AS 1604.1 – 2000, Specification for preservative treatment part 1: sawn and round timber.

A cost effective combined fire retardant and preservative treatment for timber that provides resistance to both fire and biodegradation and can be applied using existing technology, would deliver a substantial economic benefit to the forestry and forest products industry and is currently not available within Australia. Such a treatment would enable timber to maintain a competitive market share against alternative timber-substitute materials, and would open up new markets for timber products. In addition, such a treatment would deliver a significant social benefit through the reduced loss of dwellings in times of bush-fires.

The limitations on potential fire retardants were a function of the co-application with current preservatives (which have already attained H3 status); we considered both aqueous and LOSP type systems.

Overall the results have demonstrated that it was possible to achieve excellent fire retardancy and wood preservation in a single step treatment process prior to weathering. However, the inability of some fire retardants to be impregnated into timber at sufficiently high loadings to achieve adequate fire retardation and the inability to co-solvate a number of fire retardants with preservatives in a wide range of solvents, proved to be a limiting factor.

The weathering regime called for in AS 3959 proved to be a stumbling block, as none of the fire retardant / wood preservative systems studied in this project were capable of passing this test. It should be noted however that the amount of water applied to the specimens during the 1000 hours of exposure as per the AS 3959 standard, is equivalent to approximately 20,000 mm of rain. This corresponds to 2,000 mm of rain per annum (or 170 mm per month) for 10 years; under most Australian conditions this is somewhat excessive and the question has to be asked as to the relevance of this test for Australian conditions.

The results have revealed that some of the fire retardants demonstrated anti-termite and anti-fungal properties; however none of the wood preservatives considered in this study demonstrated any fire retardancy properties. When the two types of additives were combined, some of the fire retardants enhanced the performance of the wood preservatives by improving the dispersion and penetration of the wood preservative actives in the timber.

An indicative costing of a best case scenario for implementing a single step FR/WP system was between \$450-750/m^3 above current preservative system costs.

The overview discussion is provided in the body of this report, and where results have not been presented previously, detailed discussion and results are provided in the appendices.

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1. INTRODUCTION

The purpose of this project was to develop a 'proof of concept' for a dual fire retardant/wood preservative (FR/WP) treatment technology for softwood (*P. radiata*) and/or hardwood (*E. regnans*) species that were of low natural durability, to satisfy AS 3959 – 1999.[1] The focus was on timber that found use in outdoor, above-ground applications.

In order to develop the technology in the most time efficient manner, the approach adopted here was to focus on combining an existing preservative, as outlined in Hazard Class 3 of AS 1604.1 – 2000,[2] with a known fire retardant.

As a result of consultation with industry, Hazard Class 3 applications were identified as the highest priority for fire retardant treatment. The preservatives selected for study in this project were those accepted for Hazard Class 3 applications; they included the waterborne preservatives: chromated copper arsenate (CCA), ammoniacal copper quaternary (ACQ), and copper azole (CuAz) and the light organic solvent preservatives (LOSP): copper napthenate (CuN), tebuconazole/propiconazole (TPP) and tributyltin napthenate (TBTN), all co-formulated with a synthetic pyrethroid insecticide (permethrin). This consultation also concluded that the fire retardant should be applied at the same time as the preservative, *i.e.*, a single step process, as it was unlikely to be economically viable to treat the product twice.

Several fire retardant systems that appeared suitable in terms of their chemical and physical properties were identified. There was however, no data available that suggested the levels of fire retardant required for satisfactory performance in timber; nor was there any data relating to the resistance of these fire retardants to weathering when impregnated into timber. One of the key challenges of this project was to understand how the fundamental properties of the two systems could be combined to produce a novel fire retardant/preservative system without compromising the performance of each component.

In this project we chose to consider halogenated, phosphorus and inorganic based fire retardants. Halogenated fire retardants generally act in the gas phase by trapping free radical species formed during combustion of a material.[3] This considerably slows or prevents the burning process, thus reducing heat generation as well as the production of further gaseous flammable material that act to propagate the flame.[4] The free radical species in question include hydrogen and hydroxyl which are primarily responsible for continuing the flame reaction, as well as other radicals which contribute to bond breaking within the timber matrix.

Phosphorus containing fire retardants are active mainly in the solid (or condensed) phase of burning materials and are particularly effective in materials with high oxygen content, such as cellulosics. The fire retardant is converted by thermal decomposition to

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[1] *AS 3959 – 1999, Construction of buildings in bushfire-prone areas.*
[2] *AS 1604.1 – 2000, Specification for preservative treatment part 1: sawn and round timber.*
[3] *Alaee, M., Arias, P., Sjodin, A. and Bergman, A. (2003). An overview of commercially used brominated flame retardants, their applications, their use patterns in different countries/regions and possible modes of release.* Environmental International, **29**, 683-689.
[4] *European Flame Retardants Association (EFRA) website – www.cefic-efra.com Last accessed 30-5-06*

phosphoric acid, which in the condensed phase extracts water from the pyrolysing substrate causing it to char and, in the case of wood, enhancing its already substantial charring properties. Phosphorus-based fire retardants act on a burning situation in more than one way: they coat the condensed combustible layer, thereby cooling the condensed phase and excluding the access of oxygen and thus delaying ignition. In addition, they form or help form a carbon char on the surface, thereby protecting the remaining condensed combustible layer from escaping into the flame and combining with oxygen to propagate the flame.[5] In the gas phase, phosphorus based fire retardants stop the free radical oxidation process of the carbon at the carbon monoxide stage, preventing the highly exothermic reaction of carbon dioxide formation.[6]

Inorganic fire retardants interfere in the burning process via various physical processes, *i.e.,* (a) release of water or non-flammable gases that dilute the gases feeding the flames, thus cooling the fire, (b) absorption of heat energy (in the gas-release reactions) thus cooling the fire and (c) production of a non-flammable and resistant layer on the surface of the material.[4]

The literature suggested that halogenated fire retardants were not broadly used in timber for commercial applications. Phosphorus based fire retardants on the other hand were commonly used in timber but often as compounds that were easily hydrolysable and in this form presented a threat to the timber longevity in terms of saponification. In addition, their use in combination with an H3 class wood preservative has never occurred. The inorganic fire retardant that we used was an experimental proprietary product and contained large quantities of calcium cations as well as other inorganic and metal ions. The exact fire retardant mechanism of this calcium based product was unknown, however, analysis of our data suggested that it acted primarily in the condensed phase to enhance char formation and slow the formation of, and subsequent release of gases.

For a fire retardant/preservative system to have widespread industrial applicability, the technology needed to meet a number of requirements at the proof of concept stage:

1. The fire retardant had to be compatible with a preservative from Hazard Class 3, AS 1604-1 – 2000.
2. To meet the requirements of the building code the combined fire retardant/preservative had to be applied via vacuum-pressure impregnation.
3. To be economically viable the combined fire retardant/preservative had to be applied in a single treatment using existing infra-structure.
4. The fire retardant and preservative needed to have sufficient permanence in wood to withstand the accelerated weathering regimes specified in AS 3959 – 1999.
5. The fire retardant/preservative needed to be environmentally benign.
6. The overall cost of the treatment process had to be such that the treated timber was cost competitive with alternative materials.

To address these issues, a review of the literature was conducted to provide an indication of which preservatives and fire retardants were reasonable for consideration in this work. The review also provided a guide as to which methodologies would suit our aims.

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[5] *Milestone 2 report - State of the Art Review; Doc CMIT-(C)-2004-272*

[6] *Bourbigot, S. and Le Bras, M. (2004). 'Flame Retardant Plastics. In Plastics Flammability Handbook: Principles, Regulations, Testing and Approval' 3rd ed, Ed J.Troitzsch*

Both softwood (*P. radiata*) and hardwood (*E. regnans*) timbers were examined in this project. The experimental program consisted of two stages:

1. The establishment via an iterative process, of a short list of possible combinations of fire retardants and preservatives that were most promising in terms of their fire performance, resistance to weathering and ease of application.

2. Having identified the systems that showed promise, an experimental program was undertaken. This involved a more detailed investigation of performance using the various test methodologies outlined in the appropriate standards and protocols.

To take this work from proof of concept to the next stage, (*i.e.*, commercialisation) an assessment of economic feasibility was conducted based on chemical and process costs and the loadings required to achieve a specified level of performance. Costs were benchmarked with industry partners and compared with existing commercial systems.

Finally, as part of the next stage of development, a series of projects were proposed to address some of the questions raised in this work. These projects will be followed by large scale field assessments.

2. PROJECT DETAILS

Project Number: PN04.2007

Project Title: Combined Fire-Retardant and Preservative Systems for Timber Products in Exposed Applications

Project Manager: CSIRO/Robert Peile

Principal Investigator: Vince Dowling

Personnel working on this project:

NAME	POSITION ON PROJECT	ORGANISATION	% TIME
Vincent Dowling	Project Leader	CSIRO MIT	5
Donavan Marney	Scientist: chemistry, combustion	CSIRO MIT	30
Lee Russell	Scientist	CSIRO MIT	30
Laurie Cookson	Scientist: mycology, applied preservation	CSIRO FFP	5
Rob Mann	Scientist: chemistry, communication, industry liaison	CSIRO FFP	30
Andrea Hunt	Scientist: chemistry	CSIRO FFP	50
Narelle Chew	Scientist: mycology and entomology	CSIRO FFP	20

Table 1: Nomenclature

APVMA	Australian Pesticides and Veterinary Medicines Authority
AWPC	Australian wood preservation committee
TTI	Time to ignition
PHRR	Peak heat release rate
LOS	Light organic solvent
LOSP	Light organic solvent preservative
FR/WP	Fire Retardant / Wood Preservative
e.m.c.	Equilibrium moisture content
ACQ	Ammoniacal Copper quaternary
CCA	Copper chromium arsenate
CuN	Copper naphthenate and permethrin
TPP	Tebuconazole/Propiconazole and permethrin
Reofos RDP	Resorcinol bis diphenyl phosphate
FR-513	Tribromoneopentyl alcohol
Burn-X	Aqueous mixture containing mainly calcium cation and chloride anion
Cereclor AS 65	Chlorinated paraffin
PE-68	Tetrabromobisphenol A bis (2,3-dibromopropyl ether)
LOSP	Light organic solvent preservative
FR-370	Tris (tribromoneopentyl) phosphate
Deca	Decabromo diphenyl ether
FR-1206	1,2,5,6,9,10-Hexabromocyclododecane
BE 51	Tetrabromobisphenol A bis (allyl ether)
PHT 4-Diol	Tetrabromo phthalic anhydride-diol
TMB(I)	Trimethyl borate in proprietary solvent formulation
TMB(II)	Trimethyl borate in methanol
Reofos 35	Isopropylated triaryl phosphate ester
TBTN	Tributyltin naphthenate
CuAz	Copper Azole
LHS	Left hand side
RHS	Right hand side

3. IDENTIFICATION OF CANDIDATE FIRE RETARDANTS

A literature review was carried out to identify suitable fire retardants for combination with preservatives to protect timber from both fire and biodegradation when used in external applications.[5]

The major findings of the literature review were:

- There are no commercially available combined fire retardant / wood preservative impregnation systems for exterior timbers that comply with both fire and durability standards.
- The only fire retardants currently approved for exterior use are those based on heat-cured polymeric systems. Timber treated with these systems was not considered to be preservative treated, although it was shown that this timber was more durable than if it were untreated.
- Extensive studies on combined preservative and fire retardant treated timber by the USDA Forest Products Laboratory in Wisconsin found that timber treated with amino-plastic resins and quaternary ammonium salts performed well in fire tests, even after accelerated weathering. These systems were covered by a patent.[7]
- Some inorganic fire retardants that claimed to be suitable for timber in exterior applications could be found within the patent literature; however, these were generally applied via a two step treatment.
- There were organic soluble fire retardants that appeared to be compatible with solvent or oil-borne preservatives. However, very few systems of this type had been examined in timber, and none of these had been assessed in exterior applications or in combination with preservatives.
- The permanence of the candidate fire retardants in timber exposed to outdoor, above ground weathering trials was still to be established.

In summary, there was no prior art to suggest that the approach proposed for this project, as outlined in the original proposal, did not have merit.

Upon consultation with industry collaborators, it was suggested that in the first instance timber used in Hazard Class 3 applications was the most likely to come under scrutiny in areas that were designated as being of high bushfire hazard. Hence the focus of the project was on identifying fire retardants that were compatible with Hazard Class 3 preservatives. The Hazard Class 3 preservatives included in this study were: the waterborne preservatives, chromated copper arsenate (CCA), ammoniacal copper quaternary (ACQ) and copper azole (CuAz) and the light organic solvent preservatives (LOSP), Copper napthenate (CuN), Tebuconazole/Propiconazole (TPP) and Tributyltin napthenate (TBTN) - all co-formulated with a synthetic pyrethroid insecticide.

After consultation with industry collaborators and as a result of the literature review, the criteria for fire retardant selection was established and this is included in Table 2 below.

[7] LeVan, S. L. and DeGroot, R. C. (1993). *One step process for imparting decay resistance and fire retardancy to wood products.* United States Patent No. 5,185,214.

Table 2: Criteria for fire retardant selection

Criteria[a]	Description	Comments
Efficacy in wood	Ability of the fire retardant treated product to meet the relevant performance standards.[b]	Can be established via an experimental program.
Permanence in wood in exposed applications	The fire retardant must be resistant to leaching and stable towards ultraviolet exposure.	Some insight can be gained by considering the solubility of the candidate fire retardant. Accelerated weathering trials provide an indication of the relative permanence of compounds in wood. $K_{o/w}$ may be a useful parameter to consider when comparing the permanence of fire retardants.[c]
Solubility	The fire retardant must be able to be co-formulated with the preservative.	For fire retardants that are not soluble in water, they may be applied from light organic solvents, oils or as oil-in-water emulsions (the feasibility of forming formulating emulsions will be explored as part of the project).
Availability	Commercial sources	Fire retardant must be available in commercial quantities.
Cost	The cost and loading are the most important factors in estimating process costs.	Costs are currently being sought. In many instances the cost of sourcing large quantities of chemicals is substantially different to that of small quantities.
Toxicity	Short and long term effects of the fire retardant on health and the environment	There is a push away from the use of halogens in fire retardants because of possible chronic health and environmental effects.

[a] *Not listed in order of importance.*
[b] *Both fire and preservative standards must be met. It is critical to establish if the fire retardant influences the behaviour of the preservative, and vice-versa. Ideally, the combination of both fire retardant and preservative may lead to synergism, rather than antagonism.*
[c] *Partition co-efficients ($K_{o/w}$), e.g. octanol/water.*

The types of compounds included in this project were:

i. Halogenated aliphatic compounds
ii. Halogenated aromatic compounds
iii. Phosphorus based compounds - phosphates
iv. Halogenated phosphate esters
v. Boron compounds

The consultation with industry also suggested that the fire retardant be applied at the same time as the preservative, *i.e.*, a single step process, as it was unlikely to be economically viable to treat the product twice.

It should be noted that, at the commencement of the project an LOSP based treatment regime was considered by industry collaborators to be the most preferred outcome. But after the APVMA[8] decision in March 2005 to limit the use of CCA to areas other than human contact situations, the market response resulted in more plants transferring to the aqueous ACQ and copper azole systems rather than LOSP. This changed the focus of the project and resulted in a stronger interest in aqueous based systems.

Based on the criteria listed in Table 2, the fire retardants detailed in Table 3 were chosen.

[8] *http://www.apvma.gov.au/media/mr0501.shtml*

Table 3: Candidate fire retardants (FR)

Compound name or trade name	FR classification	Physical description
Tribromoneopentyl alcohol (FR-513)	Brominated aliphatic alcohol	White solid, mp = 65 ^{o}C
Hexa-bromocyclododecane (FR-1206 or HBCD)	Brominated Aliphatic	White to off-white, crystalline,free flowing powder, mp = 175-185 °C
Tris(tribromoneo-pentyl)phosphate (FR-370)	Brominated phosphate ester	White powder, mp = 181 ^{o}C
Decabromo-diphenyl ether	Brominated aromatic	White to off-white solid, mp = 303-307 ^{o}C
Chlorinated paraffins (Cereclor AS 65)	Chlorinated alkane	Varies, liquid to waxy solid
Triaryl phosphate ester (Reofos 35)	Isoproylated triaryl phosphate	Clear liquid
Triaryl phosphate ester Reofos® RDP	Biphosphate-ester of resorcinol.	Clear liquid
Tetra-bromobisphenol A bis (allylether) BE-51	Aromatic bromine	White powder, mp = 115-120 °C
Tetra-bromobisphenol a bis (2,3-dibromopropyl ether) PE-68)	Aromatic and aliphatic bromine	Off-white powder, mp = 106-120 °C
Tetra-bromophthalate diol reactive FR intermediate (PHT 4-Diol)	Aromatic brominated alcohol	Light brown viscous liquid
Trimethyl borate(I)	Boron ester	Clear liquid
Trimethyl borate(II)	Boron ester	Clear liquid

4. ESTABLISH BASELINE DATA[9]

In this section of work the fire performance of untreated and preservative treated *P. radiata* and *E. regnans* specimens was measured according to a modified version of *AS/NZ 3837*[10] using a mass loss calorimeter. The irradiance level used was 25 kWm^{-2} as mentioned in Section 1.5.6 of AS 3959. The parameters of interest were peak heat release rate (PHRR) - a measure of the materials ability to spread a flame or fire, and time to ignition (TTI) - the time at which the material ignites using a piloted ignition source. This data was essential to determine the level of fire performance displayed by the untreated and preservative treated timber and establish a baseline.

The fire performance of a commercially available fire retardant timber treatment system (FRX) was also to be examined to establish the baseline for this potentially competing option.

A summary of the fire performance results for preservatives is presented in Table 4. More detailed results and discussion can be found in the Milestone 3 Report.[9] These results are representative of data collected throughout the project and are based on specimens with an exposed surface area of 25 cm^2 (single face exposed to the radiation source). In the early stages of this work smaller specimens were used which had a surface area of 4 cm^2. However it was found that the signal-to-noise ratio of the output data introduced too much variability into the results, so the decision was made to continue the work with the larger specimens.

Summary of results from preservative-treated *P. radiata* and *E. regnans*

- With respect to the untreated timber and timber treated with the solvent controls, the preservatives did not impart any additional fire retardancy to either timber.
- *P. radiata* and *E. regnans* treated with TMB(I) ignited early, burnt with the greatest intensity but with the slowest rate of mass loss and left the highest proportion of residual char. This presents some contradictions and it can be assumed that the first two characteristics are a function of the TMB carrier system (*i.e.,* methanol, pale boiled linseed oil and high flash kerosene), whilst the last two are probably a function of the improved fire performance brought about by the boron ester.[13]
- In general there was little difference in the burn performance of the two timbers (taking into consideration time to ignition, total burn time and total heat release) when treated with the same preservative.

[9] *Milestone 3 Report, CMIT-(C)-2004-447*
[10] *AS/NZS 3837 "Method of test for heat and smoke release rates for materials and products using an oxygen consumption calorimeter."*

Table 4: Preservative burn data extracted from Mass Loss Calorimeter for *P. radiata* (and *E. regnans*) exposed to a radiation of 25 kW m^{-2}

Treatment	No.	TTI (s)	TBT (s)	PHRR (kWm^{-2})	THR (MJm^{-2})
Untreated	16A	115(137)	315(337)	282(299)	70.7(76.2)
White Spirit	7A	127(120)	332(274)	256(287)	59.3(67.7)
Water	8A	123(116)	329(303)	277(253)	61.2(64.6)
CCA	1A	110(113)	295(307)	250(240)	63.1(65.4)
CuAz	2A	95(118)	300(297)	223(270)	58.7(69.8)
ACQ	3A	109(109)	306(324)	274(309)	70.1(75.9)
TBTN	4A	125(134)	327(298)	287(331)	77.8(73.0)
CuN	5A	111(136)	332(319)	275(342)	69.4(82.1)
Teb/Prop	6A	115(118)	306(285)	255(284)	62.0(63.9)
TMB(I)	9A	90(94)	541(785)	379(377)	118.0(113.0)

Commercial fire retardant alternative for timber

It was originally intended to examine the commercially available system FRX as part of this project. The FRX product is generated via a multi-step process involving curing of material after application to the timber, and as such is not directly comparable to the single step methodology. The FRX system as we understand it is based upon the use of western red cedar. This timber has advantages over *P. radiata* and *E. regnans* in that in its untreated state, it has sufficient durability to not require H3 level preservative treatment, and it has inherent fire resistant properties.[11]

This system was a potential competitor against which the performance of any single step system was to be measured. However, both the manufacturer of FRX (Arch Wood Protection, Georgia, USA), and the Australian distributor (Koppers Arch Australia Pty Ltd) were unable to make any of the active material or final treated timber available for comparative testing.

At this stage of the project, the FRX was replaced by another commercially available (but still experimental) fire retardant, Burn-X. This product does not require a heat or cure step so it was decided to continue with it as a candidate fire retardant for co-formulation and its performance data was examined in the next section.

The Burn-X was sourced via the internet and a quantity was supplied by the manufacturer (VEGA Chemical Ltd, Turkey) for inclusion in this work. The

[11] *Western Wood Products Association USA (http://www.weyerhaeuser.com/coastalwood/wycedar/cedar_characteristics.htm)*

manufacturer claimed that this product would form a protective layer (*i.e.*, char) around the timber when exposed to a fire. It was also claimed that the product would provide protection to the timber against fungi and insects. In its proposed use as a fire retardant for external exposed applications, the manufacturer suggested that the timber be coated with a water repellent coating after impregnation of the FR.

An attempt was made to obtain timber treated with some of the compounds invented by the CRC for Wood Innovation. It has been claimed that these compounds have fire retardant activity.[5] This work however did not eventuate, due to an inability to obtain material from the CRC. It should be noted however that none of these compounds have reported leach resistance, and all involve multi-step application processes.

5. PRELIMINARY SCREENING FOR FIRE AND PRESERVATIVE PERFORMANCE[12,13]

Experiments were conducted to investigate the suitability of candidate fire retardants for the purpose of combining with an established wood preservative in a single step treatment process, and still meet the fire performance requirements of AS 3959 – 1999. The full set of results can be found in the Milestone 4[12] and Milestone 5[13] Reports.

- The solubility of each fire retardant in various solvents was analysed to establish the best means of assisting the impregnation of the timber.
- The fire performance of fire retardant treated *P. radiata* and *E. regnans* specimens was measured before and after accelerated leaching according to the AWPC protocols,[14] using a mass loss calorimeter. This data was necessary to determine the approximate loadings of each fire retardant required to give an acceptable level of fire performance.
- Combinations of fire retardants at highest available loadings with selected preservatives were tested for stability.
- Interactions between fire retardants and preservatives during thermal decomposition were analysed using thermo-gravimetric analysis (TGA) and differential thermal analysis (DTA).

The ability of each fire retardant to dissolve in a carrier solution is summarised in Table 5.

Summary of solubility results

- While the solubility of the FRs in white spirit varied substantially, most of the FRs could be formulated at 1% w/w with the inclusion of a co-solvent.
- For the majority of the FRs, formulation at concentrations of 5% or more required neat chlorinated solvent which would be unlikely to be acceptable in an industrial context.
- Cereclor AS 65 was able to be dissolved in white spirit alone.
- 5% FR-370 and Deca proved insoluble to any useful extent in anything other than chlorinated solvent. Even in neat chlorinated solvent, these FRs were only slightly soluble.

[12] Milestone 4 Report, CMIT-(C)-2005-032-Revised
[13] Milestone 5 Report, CMIT-(C)-2005-246
[14] *Australian Wood Preservative Committee (AWPC), (1997). Protocols for assessment of wood preservatives.*

Table 5: Solubility of fire retardants

Fire Retardant	No.	Conc. (% w/w)	Solvent (%)
Water control	8A	-	Water
White spirit control	7A	-	White spirit (WS)
FR-370	12A	1	WS (50) & dichloromethane (50)
FR-370	12B	5	Not sufficiently soluble
FR-513	13A	1	WS (75) & toluene (25)
HBCD	14A	1	WS (80), toluene (10) & acetone (10)
Reofos 35	10A	1	WS (90) & toluene (10)
Reofos 35	10B	5	WS (85) & toluene (15)
Reofos RDP	11A	1	WS (90) & toluene (10)
Reofos RDP	11B	5	WS (70), toluene (15) & dichloromethane(15)
Deca	N/A	-	Not sufficiently soluble
Cereclor AS 65	17A	10	White Spirit
Cereclor AS 65	17B	20	White Spirit
Cereclor AS 65	17C	40	White Spirit
Burn-X	15A	10	Water
Burn-X	15B	50	Water
Burn-X	15C	20	Water
Burn-X	15D	30	Water
Trimethyl borate(I)	9A	8	Proprietary solvent-based formulation incorporating methanol, pale boiled linseed oil and high flash kerosene
Trimethyl borate(II)		8	Methanol / white spirit

A summary of the fire performance results are presented in Table 6. *P. radiata* was treated with all fire retardants whereas *E. regnans* was treated with only a few. At this stage of the project there were problems with the supply of *E. regnans*, so rather than wait and delay the project, it was decided to base the selection of the fire retardants that would be carried forward, primarily on the *P. radiata* results. This was considered to be appropriate as the trends in the fire performance results from *E. regnans* were consistent with those from *P. radiata.*

Summary of results from fire retardant treated *P. radiata* and *E. regnans*

- In general, the FR treatments had much the same effect upon both timbers and all FRs were suitable for both timbers. In general, fire performance was equivalent on a loading per m^3 basis.
- PHT4-diol was not acceptable as it enhanced burning of the wood.
- Burn-X at the dosage rate of 50% was a stand-out as the best performing fire retardant.

Table 6: Fire retardant burn data extracted from Mass Loss Calorimeter for *P. radiata* (and *E. regnans*) exposed to a radiation of 25 kW m^{-2}

Treatment	No.	TTI (s)	TBT (s)	PHRR (kWm^{-2})	THR (MJm^{-2})
White Spirit	7A	127	332	256	59.3
Water	8A	123	329	277	61.2
Untreated	16A	115(137)	315(337)	282(299)	70.7(76.2)
1% Reofos 35	10A	120(145)	304(362)	279(352)	70.1(93.8)
5% Reofos 35	10B	154	320	269	66.3
1% Reofos RDP	11A	130	363	276	75.4
5% Reofos RDP	11B	100	894	249	71.4
1% FR-370	12A	110	336	249	66.0
1% FR-513	13A	116	289	160	40.1
5% FR-513	13B	121	314	275	69.0
1% FR-1206	14A	102	299	248	56.5
10% Burn-X	15A	128	869	218	54.1
50% Burn-X	15B	876	111	18	13.0
20% Burn-X	15C	123	877	204	61.8
30% Burn-X	15D	89	911	126	55.8
10% Cereclor AS 65	17A	92	392	276	76.0
20% Cereclor AS 65	17B	87(82)	423(477)	267(350)	76.7(101)
40% Cereclor AS 65	17C	78	920	254	84.9
5% BE-51	18A	70(138)	313(346)	241(333)	61.9(90.2)
2% PE-68	19A	96	311	260	72.3
10% PHT4-diol	20A	9	433	342	104

Combinations of fire retardants (at highest possible loadings) and preservatives (at H3 retention levels) were tested for stability. Combinations were assessed for visible signs of precipitation or degradation. The results after 12 weeks standing are summarised in Table 7 (aqueous systems) and Table 8 (LOSP systems), with red indicating that the combination was unsuitable, amber denoting some signs of unsuitability and green indicating a successful combination.

Table 7: Stability indicators of aqueous systems after 12 weeks standing

Fire Retardant	Solvent	Cu Azole	ACQ	CCA
Burn-X	50 % w/w in water			

Table 8: Stability indicators of light organic solvent systems after 12 weeks standing

Fire Retardant	Solvent	CuN	TPP	TBTN
FR-370	1% w/w in 60:40 White spirit/dichoromethane			
FR-513	1% w/w in 75:25 White Spirit / Toluene			
FR-1206	1% w/w in 80:10:10 White Spirit/Toluene/Acetone			
TMB(I)	32% w/w in Pale boiled linseed oil/ high flash kerosene (8% Boron)			
Cereclor AS 65	20% w/w in White Spirit			
Reofos 35	5% w/w in 85:15 White Spirit/Toluene			
PE-68	2% w/w in 80:20 White Spirit / Toluene			
BE51	5% w/w in 80:20 White Spirit / Toluene			
PHT-4 diol	10% w/w in 66:33 White Spirit / Acetone			

Summary of stability results after 12 weeks

- The combination of Burn-X and CCA proved to be the only compatible aqueous system. There were some indications after 4 weeks that pH modification may assist in co-formulation with ACQ. However, subsequent attempts to co-formulate with ACQ or copper azole with pH adjustment were unsuccessful.
- CuN and TPP were compatible with all fire retardants except TMB(I) and FR-370.
- TBTN was only compatible with 3 of the 9 fire retardants used.

The potential interactive effect of the preservative on the fire retardant performance was explored using TGA-DTA. The ability of any given preservative to interact with any given fire retardant during thermal decomposition is summarised in Table 9. Clear interaction was indicated by an ‘O’, questionable interaction was indicated by a ‘?’ and no interaction was indicated by an ‘X’.

Table 9: Summary of interactions between preservatives and fire retardants during thermal decomposition

Fire Retardants	Preservatives[15]			
	ACQ	*CuAz*	*Teb/Prop*	*TBTN*
Burn-X	O	X	X	X
FR-513	X	X	?	X
Reofos RDP	?	?	X	X
Reofos 35	O	O	?	?
Cereclor AS 65	O	X	O	X
FR-1206	N/A	?	X	?
FR-370	O	X	?	X
TMB(I)	O	?	X	?

Summary of interaction results

- ACQ interacted with a number of the fire retardants, including the aqueous based Burn-X, whereas CuAz only interacted with Reofos 35 an LOSP based fire retardant.
- The only fire retardant that TPP interacted with was Cereclor AS 65.
- TBTN did not interact with any of the fire retardants.

A small number of better performing fire retardants were chosen for further evaluation based upon their fire performance, their compatibility with preservatives and their interaction with preservatives during thermal degradation. These included FR-513, Reofos 35, Reofos RDP, Burn-X and Cereclor AS 65. *P. radiata* was treated with these fire retardants and exposed to the AWPA leaching protocol[2], and then tested for fire performance using the mass loss calorimeter. The results are presented in Table 10.

Summary of results from fire retardant treated *P. radiata* and *E. regnans* after leaching

- The LOSP based fire retardants were able to resist water leaching and in fact their fire performance was improved after leaching.
- Water leaching had a detrimental effect on the fire performance of the aqueous based systems.

[15] *It needs to be noted that at this stage of the project, CCA was assessed to be an unsuitable preservative for industry in the medium to long term, due to concerns regarding its environmental/toxicity impact; it was therefore not included in this section of the work. This was before the APVMA decision of March 2005.*

Table 10: Fire retardant burn data extracted from Mass Loss Calorimeter for *P. radiata* - unleached (and - leached) exposed to a radiation of 25 kW m^{-2}

Treatment	No.	TTI (s)	TBT (s)	PHRR (kWm^{-2})	THR (MJm^{-2})
White Spirit	7A	127	332	256	59.3
Water	8A	123	329	277	61.2
Untreated	16A	115(142)	315(321)	282(257)	70.7(68.8)
5% Reofos 35	10B	154(168)	320(342)	269(240)	66.3(63.7)
5% Reofos RDP	11B	100(102)	894(898)	249(233)	71.4(66.6)
1% FR-513	13B	121(110)	314(328)	275(239)	69.0(63.8)
10% Burn-X	15A	128(126)	869(341)	218(239)	54.1(66.1)
20% Burn-X	15C	123(120)	877(340)	204(291)	61.8(80.1)
10% Cereclor AS 65	17A	92(85)	391(399)	276(246)	76.0(58.9)
20% Cereclor AS 65	17B	87(84)	423(405)	267(240)	76.7(64.8)
40% Cereclor AS 65	17C	78(62)	920(735)	266(211)	76.7(68.4)

Selected fire retardants and wood preservatives

It was at this point that a decision was made as to which fire retardant and preservative systems to carry forward for further evaluation. Based on all the data presented up to this time, the following combinations were chosen:

Aqueous based system

- Burn-X and CCA[16]

Light organic solvent based systems

- Cereclor AS 65 and copper naphthenate
- Cereclor AS 65 and tebuconazole/propiconazole
- Reofos 35 or Reofos RDP and copper naphthenate
- Reofos 35 or Reofos RDP and tebuconazole/propiconazole
- FR-513 and copper naphthenate
- FR-513 and tebuconazole/propiconazole
- PE-68 and copper naphthenate
- PE-68 and tebuconazole/propiconazole

[16] *It was decided to use a Burn-X /CCA combination instead of a Burn-X /ACQ combination because of its superior performance in the compatibility study.*

6. FIRE AND PRESERVATIVE PERFORMANCE EVALUATION OF COMBINED SYSTEMS[17]

After deciding which systems to carry forward for further evaluation, it was important to establish the depth of penetration and distribution of chemicals within the timber after impregnation. It was also necessary to evaluate the impact of the chosen fire retardants upon the performance of the chosen wood preservatives (via termite and fungi bioassays) and vice-versa (via fire tests). The impact of weathering (using the regime called for in AS 3959) upon the fire performance of both the FR and WP and the combination FR/WP treated timber was assessed. In addition, the gases evolved during combustion were quantified to better understand the interactive effects between FRs and WPs. Finally, the addition of activators was explored to ascertain whether the fire performance could be improved. This area of work is reported in full in the Milestone 6 Report.[17]

The range of tests involved at this stage required a substantial amount of each target material. Availability of these quantities was crucial to the timely performance of the full test series. Initial treatment of the bulk of the samples was to be followed by lengthy test sequences. At this point in the project, however, it became impossible to source quantities of either Reofos 35 or Burn-X. As both these materials were crucial to the next stage due to their promising behaviour, a decision was made to wait as long as possible before proceeding. In the end, it was necessary to wait 6 months prior to availability of Burn-X, and ultimately Reofos RDP had to be substituted for Reofos 35 as supply could not be guaranteed.

6.1 Fire retardant and preservative distribution

The depth of penetration and distribution of the additives within the timber after impregnation was evaluated using a Field Emission Scanning Electron Microscope (FE-SEM). We also evaluated whether a fire retardant impacted upon the distribution of the preservative within the timber, along with the effects of weathering upon additive concentration.

We conducted SEM imaging at three locations along the edge (left hand side-LHS, middle and right hand side-RHS) and in the centre (left hand side-LHS, middle and right hand side-RHS) of each sectioned specimen (six locations in all, as shown in Figure 1) – further detail of this is shown in Appendix A1.1. The elements of interest that were monitored from the treatments were bromine in BE-51, FR-513 and PE-68; chlorine in Cereclor AS 65, calcium in Burn-X, phosphorus in Reofos 35 and Reofos RDP, and copper in CCA and CuN. The percentage area coverage of each element at each of the six locations on the specimen before and after weathering according to AS 3959, is presented in Table 11 and Figure 12.

[17] *Milestone 6 Report, CMIT-(C)-2006-077*

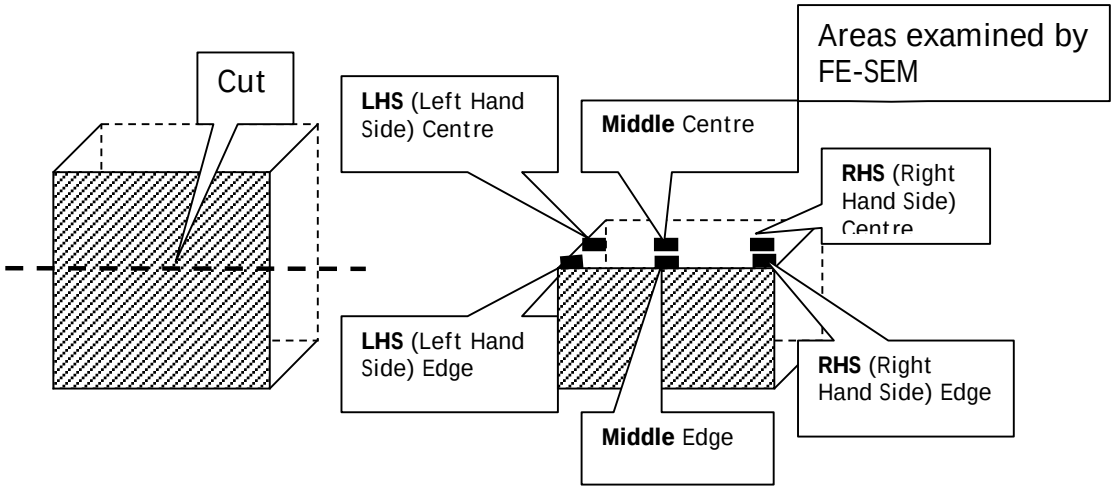


Figure 1: Profile of FE-SEM examination points

Table 11: Distribution of Fire Retardants and Wood Preservatives after Separate Treatment (and after weathering)

Treatment	Element of Interest	% Area Coverage & Location of Analysis Area					
		LHS Edge	Middle Edge	RHS Edge	LHS Centre	Middle Centre	RHS Centre
BE-51 in *P. radiata*	Bromine	8.5%	14.3%	14.5%	30.5%	28.3%	24.1%
Burn-X in *P. radiata*	Calcium	10.3%	12.0%	19.4%	N/A	3.4%	9.5%
Cereclor AS 65 in *P. radiata*	Chlorine	76.1%	50.2%	39.6%	36.4%	50.2%	57.0%
Reofos 35 in *E. regnans*	Phosphorus	3.0%	13.2%	6.3%	10.3%	6.3%	8.7%
CCA in *P. radiata*	Copper	0.47 (0.48)	0.21 (0.17)	0.63 (0.50)	0.93 (0.19)	0.31 (0.90)	0.45 (0.74)
CuN in *P. radiata*	Copper	0.15 (0.06)	0.07 (0.14)	0.13 (0.04)	0.09 (0.03)	0.21 (0.02)	0.33 (0.01)

Table 12: Distribution of wood preservatives after co-treatment with fire retardants (and after weathering)

Treatment	Element of Interest	% Area Coverage & Location of Analysis Area					
		LHS Edge	Middle Edge	RHS Edge	LHS Centre	Middle Centre	RHS Centre
CCA + Burn-X in *P. radiata*	Copper	1.9 (0.06)	2.6 (0.04)	1.6 (0.02)	0.29 (0.15)	0.15 (0.05)	0.19 (0.10)
CuN + FR513 in *P. radiata*	Copper	0.34 (0.24)	0.19 (0.35)	0.55 (0.49)	0.51 (0.27)	0.17 (0.11)	0.53 (0.34)
CuN + Reofos RDP in *P. radiata*	Copper	0.20 (0.02)	0.06 (0.01)	0.05 (0.02)	0.10 (0.02)	0.06 (0.01)	0.15 (0.01)
CuN + Cereclor AS 65 in *P. radiata*	Copper	5.2 (1.7)	1.8 (2.1)	6.7 (2.7)	3.0 (3.7)	2.8 (0.86)	2.2 (3.8)
CuN + PE 68 in *P. radiata*	Copper	3.0 (4.5)	2.5 (20.5)	5.3 (7.5)	1.7 (1.0)	1.5 (7.7)	6.5 (10.7)

This work has not appeared in any of the previous milestone reports so a detailed analysis of the results along with tables and figures are presented in Appendix 1.

Summary of additive distribution and penetration results

- The two halogenated fire retardants, BE-51 and Cereclor AS 65, were well distributed within the timber. In addition a relatively high amount of FR penetrated to the centre of the timber specimen.
- The CCA treatment delivered more additive to the timber than CuN.
- All of the LOS based fire retardants studied except for the Reofos RDP, enhanced the uptake of CuN in the timber. On the other hand, Burn-X acted to limit penetration of the CCA into the timber.
- The halogenated FRs seemed to fix the copper in the timber thus making the preservative stable to weathering.
- The presence of Burn-X resulted in a substantial loss of CCA during weathering according to AS 3959. The questions arising from this are:
    - Is the preservative still active after this weathering regime?
    - Is AS 3959 relevant to preservatives?

6.2 Fire performance

The impact of weathering upon the fire performance of the fire retarded treated timber was assessed using a modified version of the bushfire standard AS 3959. This calls for a weathering regime that complies with ASTM D2898, Method B[18], before fire testing using the modified version of AS 3837. A modified version of ASTM D2898 was undertaken in this work. A Q-Panel QUV was used instead of the equipment outlined in the standard and the exposure regimes were set as close to the standard as possible.

A summary of the fire performance results in terms of time to ignition and peak heat release rate of treated *P. radiata* before and after weathering is detailed in Figure 2 and Figure **3** respectively.

Summary of fire performance results before and after weathering

LOSP Systems

- The combination of FR and WP resulted in a reduced time to ignition relative to the FR. Exposure to the ASTM 2898 weathering regime did little to change this.
- In terms of the peak heat release rate (PHRR), the CuN / Cereclor AS 65, TPP / Cereclor AS 65 and CuN / PE-68 treatment combinations resulted in an improved fire performance before weathering relative to the FR.
- After accelerated weathering, the fire performance of CuN / Cereclor AS 65 and TPP / Cereclor AS 65 remained unaffected.

Aqueous System

- *P. radiata* treated with the Burn-X / CCA system demonstrated a similar peak heat release rate to the timber treated with only Burn-X.
- After accelerated weathering, the Burn-X / CCA system demonstrated an elevated peak heat release rate with respect to the unweathered counterpart, which indicated that a significant amount of the fire retardant active had been removed during the UV and water cycling process.

[18] *ASTM D2898-1999, Standard Test Methods for Accelerated Weathering of Fire-Retardant-Treated Wood for Fire Testing.*

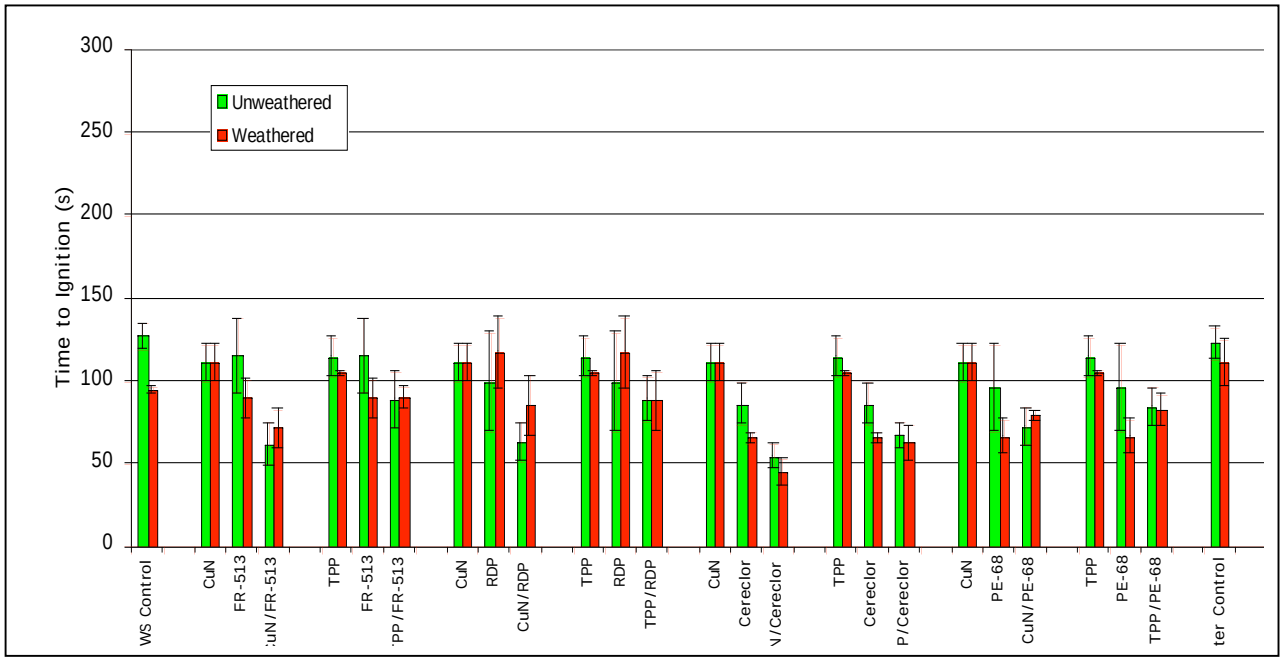


Figure 2: Comparison of time to ignition data of unweathered and QUV weathered *P. radiata* treated with preservative, fire retardant and a combination of both.

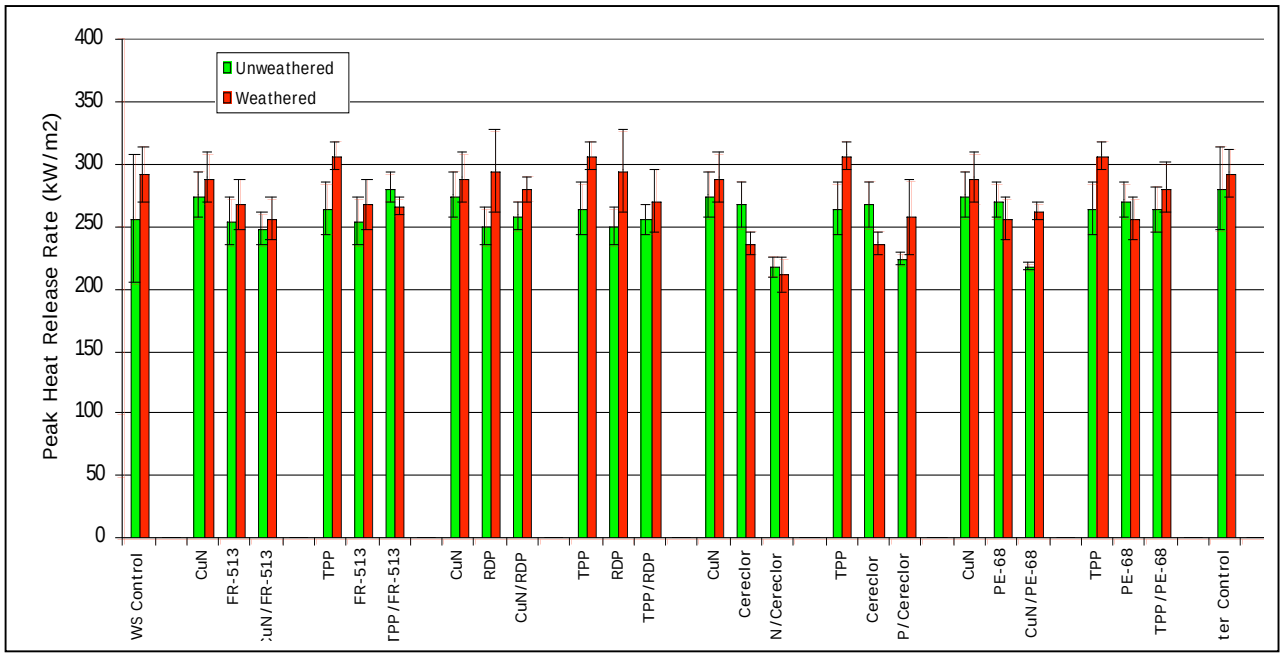


Figure 3: Comparison of peak heat release rate data of unweathered and QUV weathered *P. radiata* treated with preservative, fire retardant and a combination of both.

6.3 Termite and fungal performance of fire retardants and wood preservatives

The impact of the fire retardants upon the properties of the wood preservatives was evaluated using termite bioassays and soil block bioassays.

The termite bioassays were carried out on *P. radiata* in accordance with the minimum requirements specified for H3 conditions in the AWPC protocols[14], using two species of subterranean termite, *Mastotermes darwiniensis* (Froggatt) and *Coptotermes acinaciformis* (Froggatt). We were primarily interested in determining if the termite activity was altered by the addition of a fire retardant. A treatment was considered effective when the mean mass loss of specimens was 5% or less. A summary of the results is presented in Figure 4 with the 5% threshold indicated by the red line.

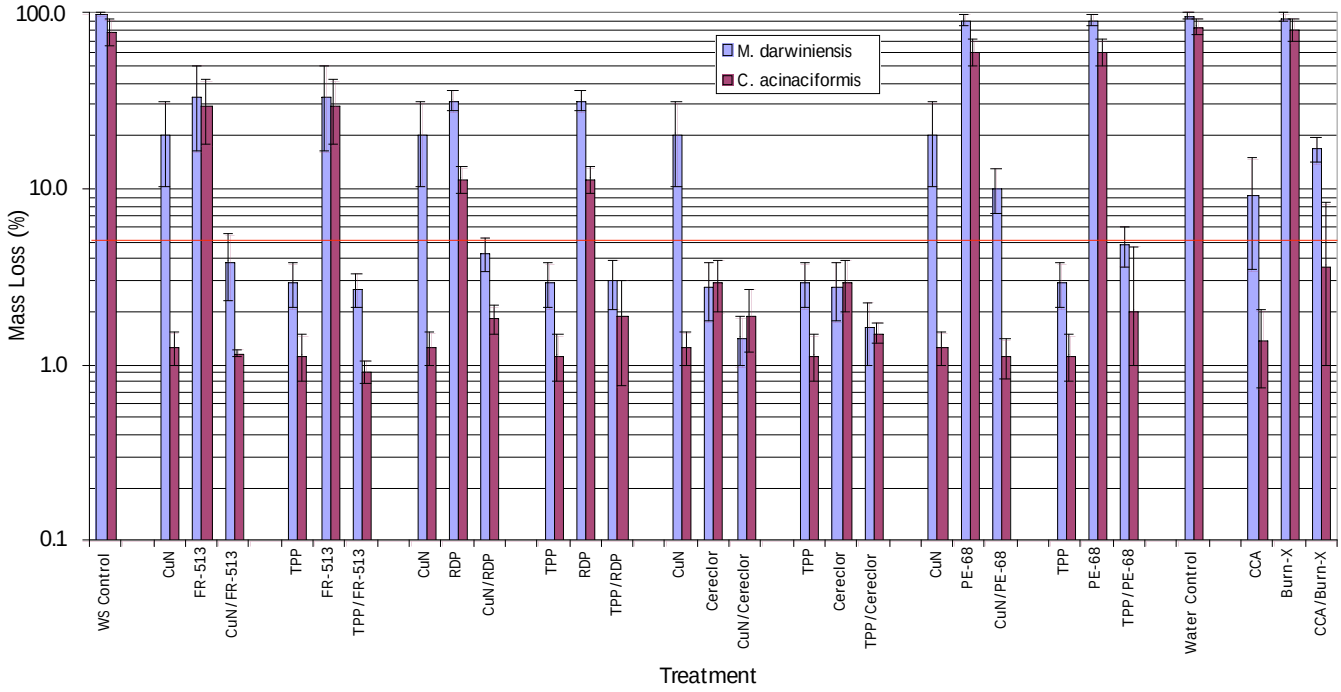


Figure 4: Termite Bioassay Data (the 5% threshold is indicated by a red line)

The soil block decay tests were carried out on *P. radiata* and *E. regnans* treated in accordance with the standard AWPC Protocols.[14] The fungi used for *P. radiata* were, brown rots *Coniophora olivacea* (isolate no. 1779), *Fomitopsis lilacino-gilva* (isolate no. 1109), *Gloeophyllum abietinum* (isolate no. 13851) and *Serpula lacrymans* (isolate no. 16508). For *E. regnans*, the white rot fungus *Perenniporia tephropora* (isolate no. 7904) was used

We were primarily interested in determining if the preservative anti-fungal activity was altered by the addition of a fire retardant. To do this we used the mass loss as the performance criterion. A treatment was considered effective when the mean mass loss of specimens was 3% or less.

The mean mass loss results of WP and FR treated *P. radiata* and *E. regnans* after exposure to test fungi are presented in Table 13. Data where mass loss was 3% or less is highlighted.

Table 13: Percent mean mass loss of specimens after exposure to five decay fungi for 12 weeks. Standard error of means is given in parentheses. Mean mass losses below 3% are highlighted.

Treatments	Mean Mass Loss* (%)				
	Brown Rot Fungi – *P.radiata*				White Rot Fungi *E.regnans*
	C.olivacea	***F.lilacino-gilva***	***G.abietinum***	***S.lacrymans***	***P.tephropora***
White spirit (control)	13.8 (4.0)	52.4 (0.9)	31.2 (1.1)	25.0 (1.9)	11.0 (0.4)
CuN (H3)	9.6 (5.1)	40.0 (2.4)	0.8 (0.3)	16.5 (1.6)	3.5 (0.4)
FR-513 (1%)	11.8 (1.5)	44.1 (2.2)	26.7 (0.8)	21.3 (0.9)	8.9 (0.9)
CuN/FR-513	5.2 (2.0)	22.7 (5.1)	2.4 (1.0)	6.4 (1.7)	0.7 (0.1)
TPP (H3)	-0.04 (0.1)	1.5 (0.2)	0.3 (0.1)	0.2 (0.1)	0.0 (0.1)
FR-513 (1%)	11.8 (1.5)	44.1 (2.2)	26.7 (0.8)	21.3 (0.9)	8.9 (0.9)
TPP/FR513	-0.1 (0.1)	-0.2 (0.1)	-0.1 (0.1)	-0.2 (0.1)	-0.1 (0.2)
CuN (H3)	9.6 (5.1)	40.0 (2.4)	0.8 (0.3)	16.5 (1.6)	3.5 (0.4)
Reofos RDP (5%)	5.4 (0.9)	31.2 (0.9)	33.5 (2.8)	22.1 (2.2)	12.8 (0.7)
CuN/Reofos	0.5 (0.3)	27.8 (1.4)	0.3 (0.2)	15.6 (2.3)	2.2 (0.6)
TPP (H3)	-0.04 (0.1)	1.5 (0.2)	0.3 (0.1)	0.2 (0.1)	0.0 (0.1)
Reofos RDP (5%)	5.4 (0.9)	31.2 (0.9)	33.5 (2.8)	22.1 (2.2)	12.8 (0.7)
TPP/Reofos	0.2 (0.2)	2.2 (0.6)	-0.2 (0.2)	0.1 (0.2)	0.6 (0.3)
CuN (H3)	9.6 (5.1)	40.0 (2.4)	0.8 (0.3)	16.5 (1.6)	3.5 (0.4)
Cereclor (20%)	6.6 (2.3)	38.2 (0.9)	22.1 (0.4)	13.6 (2.2)	8.3 (0.8)
CuN/Cereclor	0.2 (0.1)	23.8 (2.3)	0.6 (0.1)	7.0 (0.9)	1.1 (0.5)
TPP (H3)	-0.04 (0.1)	1.5(0.2)	0.3(0.1)	0.2(0.1)	0.0(0.1)
Cereclor (20%)	6.6 (2.3)	38.2(0.9)	22.1(0.4)	13.6(2.2)	8.3(0.8)
TPP/Cereclor	0.34 (0.2)	0.7(0.2)	0.4(0.04)	0.5(0.1)	-0.2(0.1)
CuN (H3)	9.6 (5.1)	40.0(2.4)	0.8(0.3)	16.5(1.6)	3.5(0.4)
PE-68 (2%)	10.0 (2.0)	47.7(1.0)	28.4(1.3)	23.5(0.9)	9.2(0.7)
CuN/PE-68	8.7 (4.1)	42.6(2.3)	1.3(0.8)	22.8(1.9)	2.8(0.6)
TPP (H3)	-0.04 (0.1)	1.5(0.2)	0.3(0.1)	0.2(0.1)	0.0(0.1)
PE-68 (2%)	10.0 (2.0)	47.7(1.0)	28.4(1.3)	23.5(0.9)	9.2(0.7)
TPP/PE-68	-0.2 (0.1)	1.0(0.03)	0.3(0.1)	0.21(0.1)	0.2(0.1)
Water (control)	12.3 (3.4)	47.7 (0.7)	37.4(1.3)	27.1(1.7)	8.2(0.6)
CCA (H3)	0.5 (0.1)	1.6(0.5)	0.7(0.1)	4.6(0.8)	0.3(0.1)
Burn-X (30%)	12.6 (3.0)	47.9(1.1)	37.5(1.8)	25.5(2.9)	10.3(2.0)
CCA/Burn-X	0.1 (0.1)	1.3(0.7)	0.23(0.1)	6.1(0.7)	0.5(0.2)

* Mean of six replicates per treatment

Summary of results after exposure to termites and fungi

- Incorporation of fire retardant with preservative generally had no negative effect on preservative performance.
- There were improvements in the termite performance of CuN when combined with LOS based fire retardants, FR-513, Cereclor AS 65, Reofos RDP and PE-68.
- Positive effects from combinations were much less apparent in the fungal bioassay. These effects, were apparent only with FR-513, Reofos RDP and Cereclor AS 65 and only in combination with CuN.
- The improved anti-fungal and anti-termite activity of CuN when used in combination with halogenated fire retardants, was likely to be due to the enhanced retention of copper in the presence of these halogenated FRs as indicated by electron microscopy analyses.
- No proof of any positive interaction between Burn-X and CCA could be found.

6.4 Evolved gas analysis

In this work evolved gases were collected from the combustion of the treated timbers and analysed for the following components: carbon monoxide (CO), carbon dioxide (CO_2), acrolein (C_3H_4O) and hydrocarbons (HCs) including methane (CH_4), acetylene (C_2H_2), ethylene (C_2H_4) and ethane (C_2H_6). This was done to ascertain the degree of combustion and the effectiveness of combustion. This data can be used as an index of the effectiveness and mode of action of the fire retardants. It can also give an indication of the volume and nature of any toxic evolved gases from the combustion process.

The combustion activity of the fire retardant-preservative treated timber was analysed by measuring the quantity and type of gases evolved. The total evolved HCs, CO and CO_2 gases from the combustion of unweathered treated *P. radiata* is detailed in Figure 5 and the amount of evolved acrolein gas is shown in Figure 6**.**

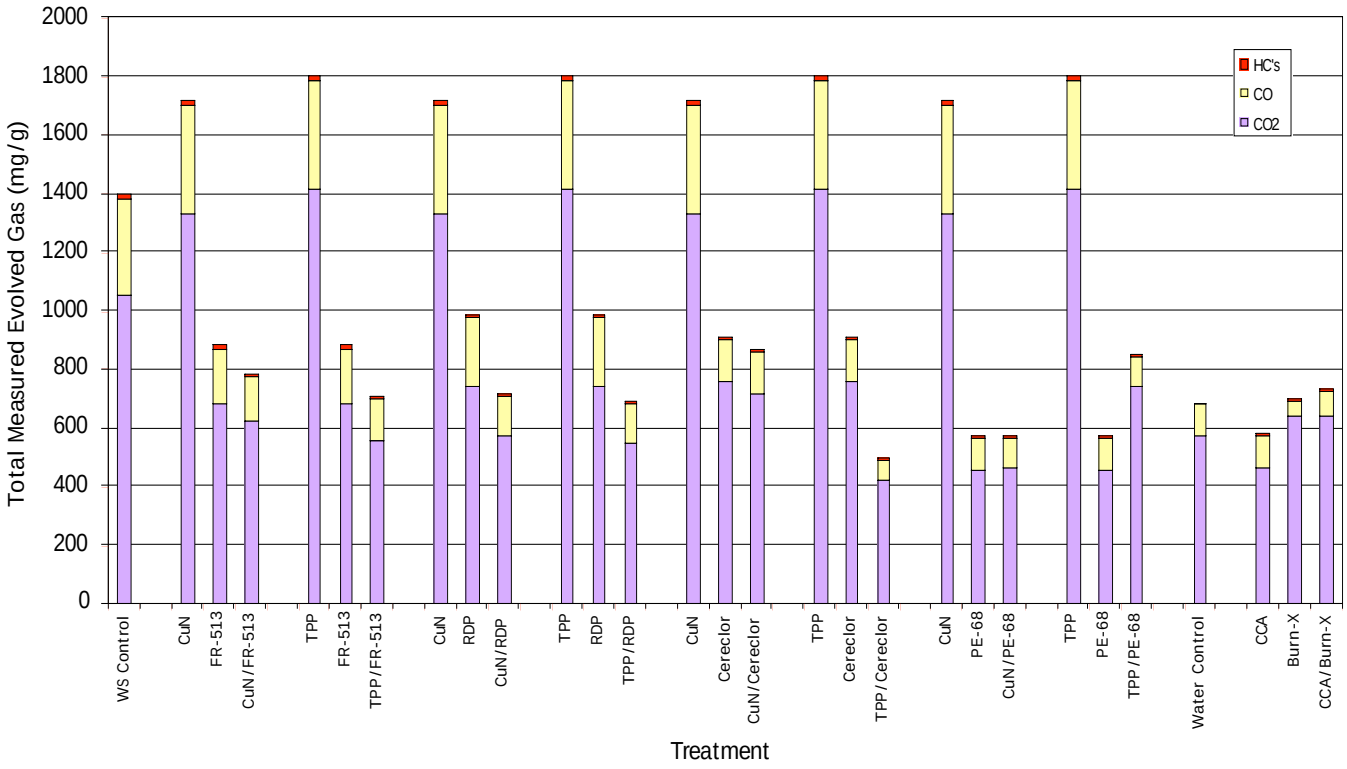


Figure 5: Total evolved gases from the combustion of unweathered *P. radiata* treated with preservative, fire retardant and a combination of both.

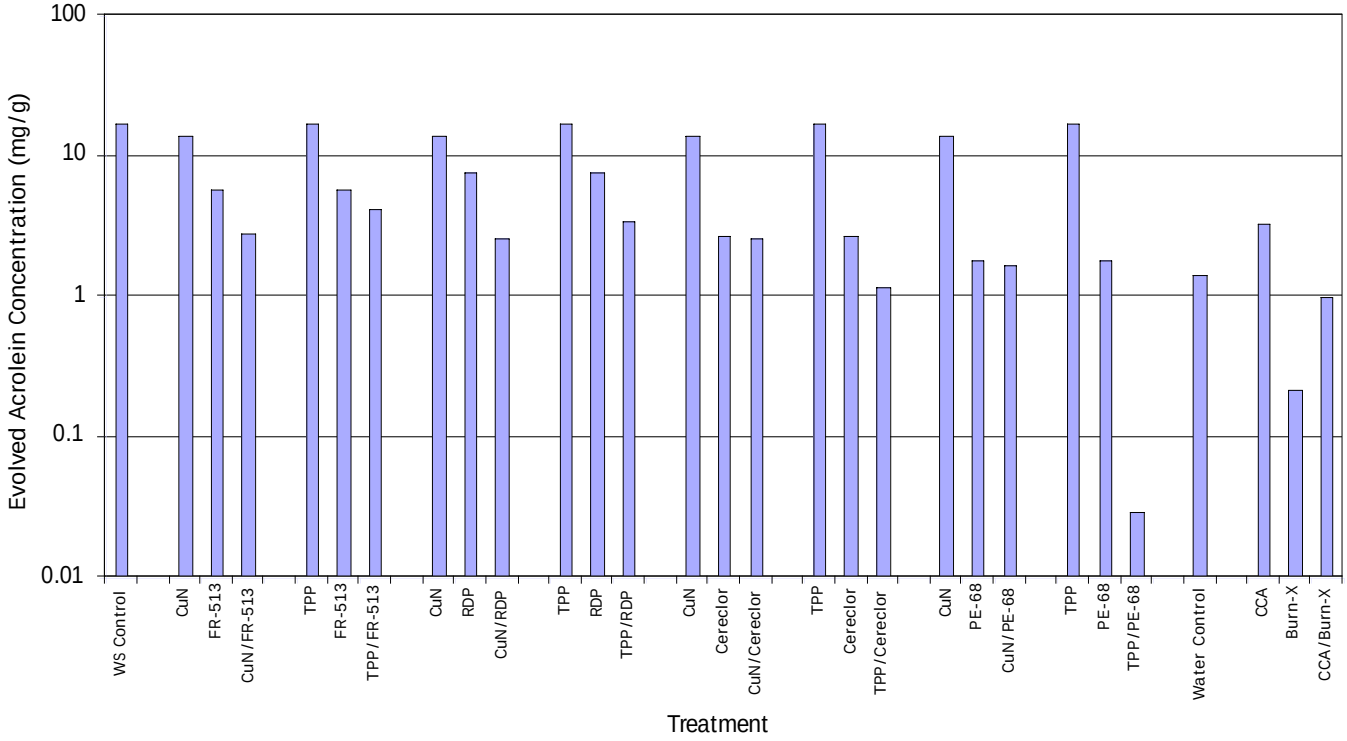


Figure 6: Measured acrolein gas concentration from the combustion of unweathered *P. radiata* treated with preservative, fire retardant and a combination of both.

Summary of results from evolved gas analyses

- The combination of LOSP based preservatives and FRs had a synergistic relationship in that the gas yield was lower than that for either additive on its own.
- The water based FR on the other hand had an antagonistic relationship with the corresponding preservative.
- The water based FR acted in the condensed phase to limit conversion of carbon from solid to gas.
- The LOSP based FR's appeared to act primarily in the gas phase to inhibit formation of gaseous species which promote fire spread (acetylene, acrolein, methane etc.). Instead it is suspected that they promoted the formation of soot.
- One of the positive effects of adding the various FRs to the WPs was that they acted to reduce the amount of the irritant acrolein formed during the normal combustion process of timber.

6.5 Activators

We explored the possibility that activators may improve the fire retardant performance. In this context we examined the potential for our target H3 preservatives to act as activators of fire retardants, and the capacity for boron in the form of boric acid equivalent to do the same. The use of boron was considered worth studying even though it was not a H3 preservative in its own right. This was due to the known ability of boron to act as a fire retardant adjunct[19].

The work carried out on the use of boron as an activator has not appeared in any of the previous milestones so a more detailed discussion of the results can be found in Apendix 2.

Summary of activators results

- The LOSP based preservative (TPP) activated the chosen FR (in this case Cereclor AS 65) by enhancing its combustion inhibition, *i.e.*, lower PHRR, as shown in Figure 7.
- The water based WP (CCA) on the other hand, did not impact upon the function of the Burn-X, when the FR was used at a dosage rate of 30%, as shown in Figure 8.
- TMB(II) in combination with other FRs generally enhanced the performance of the FRs; *i.e.*, it acted as an activator and reduced the PHRR, as shown in Figure 9.
- There appears to be some interaction occurring between TMB(II) and Cereclor AS 65 in that the PHRR for the combination is lower than for either additive.

[19] *Le Van, S.L. and Tran, H.C. (1990) The role of boron in flame-retardant treatments. 1st International Conference on Wood Protection with Diffusible. Nashville, T.E. ed. M. Hamel, Forest Products Research Society, pp. 39-41.*

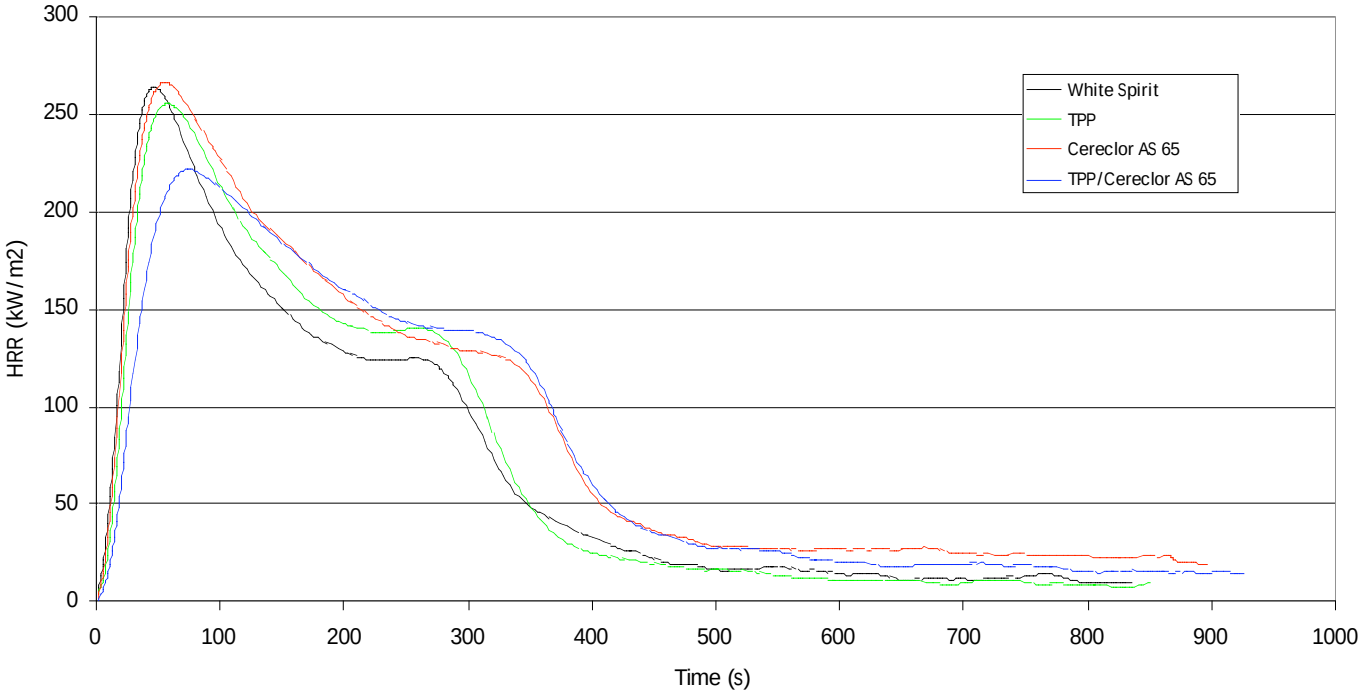


Figure 7: Peak heat release rate curves for unweathered *P. radiata* treated with TPP, Cereclor AS 65, a combination of both and white spirit (blank) at 25 kW/m^2 and corrected for ignition times.

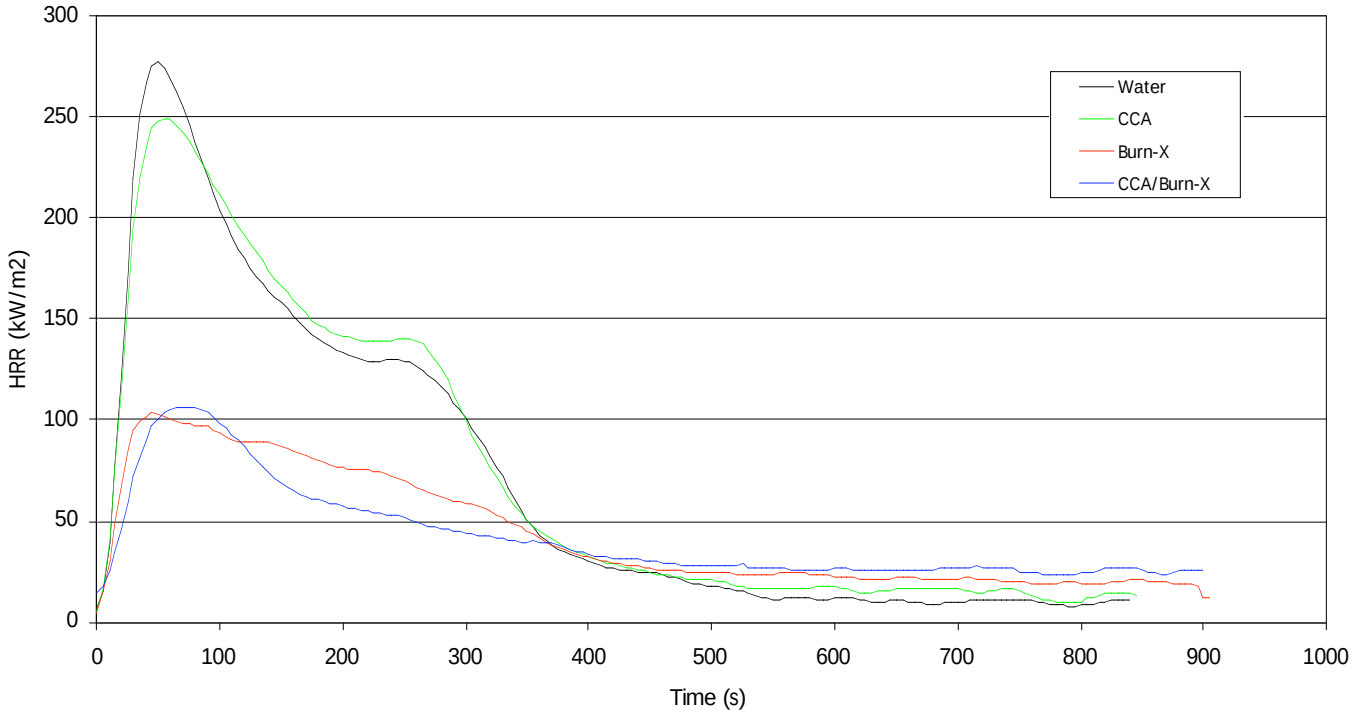


Figure 8: Peak heat release rate curves for unweathered *P. radiata* treated with CCA, 30 % Burn-X, a combination of both and water (blank) at 25 kW/m^2 and corrected for ignition times.

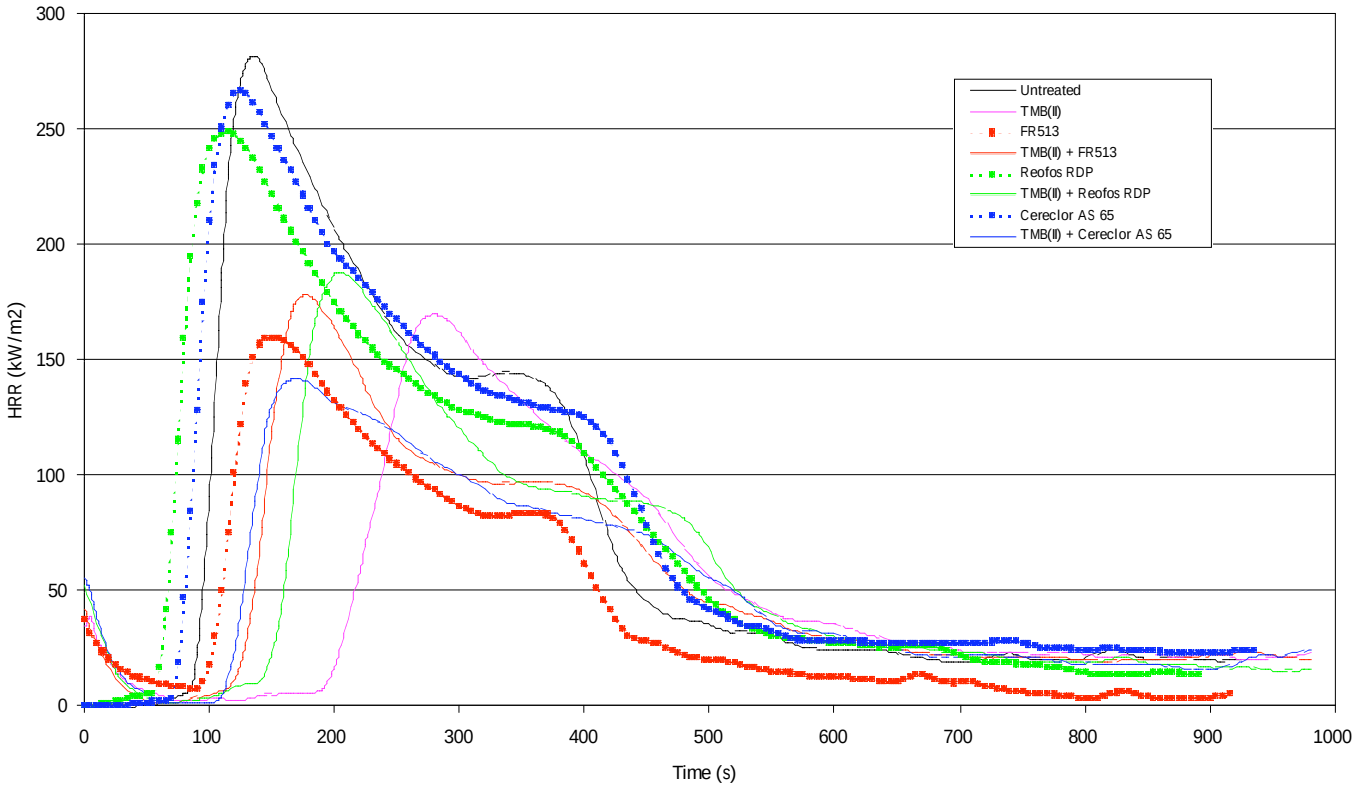


Figure 9: Heat release rate curves for *P. radiata* treated with TMB(II), FRs, a combination of both, as well as untreated timber at a radiation of 25 kWm^{-2}

7. ECONOMIC FEASIBILITY[20]

A preliminary costing of a selection of the most promising systems has been developed. The basis for the costing and any details can be found in the Milestone 7 Report.[20] A summary of the cost of the components of the selected systems is detailed in Table 14. A further breakdown of these costs on a per cubic metre basis are shown in Figure 10.

Summary of the costings of potential systems

- An indicative cost of implementing a single step FR/WP aqueous system (Burn-X/CCA) was approximately $\$600/m^3$ above current preservative system costs

- An indicative cost of implementing a single step FR/WP LOS based system (either Cereclor AS 65 or Reofos RDP with TPP) was around $\$424\text{-}454/m^3$ and $\$450\text{-}750$[21]$/m^3$ respectively, above the current preservative system cost.

Table 14: Summary of component costs

Chemical	**Price (FIS)**	**Source**
Cereclor AS 65	$2.80/kg (pallecons)	Orica chloralkali
Reofos RDP	$12.22/kg (drum)	Great Lakes (ISM)
White spirit	$1.01/L (bulk)	ICISLOR
Toluene	$1.28/L (bulk)	ICISLOR
Dichloromethane	$2.10/L (bulk)	ICISLOR
Burn-X	$2.50/kg (bulk)	Vega Chemicals

[20] Milestone 7 Report, CMIT-(C)-2006-193

[21] *$450 for a white spirit/toluene/dichloromethane solvent system, and $750 for a dichloromethane solvent system.*

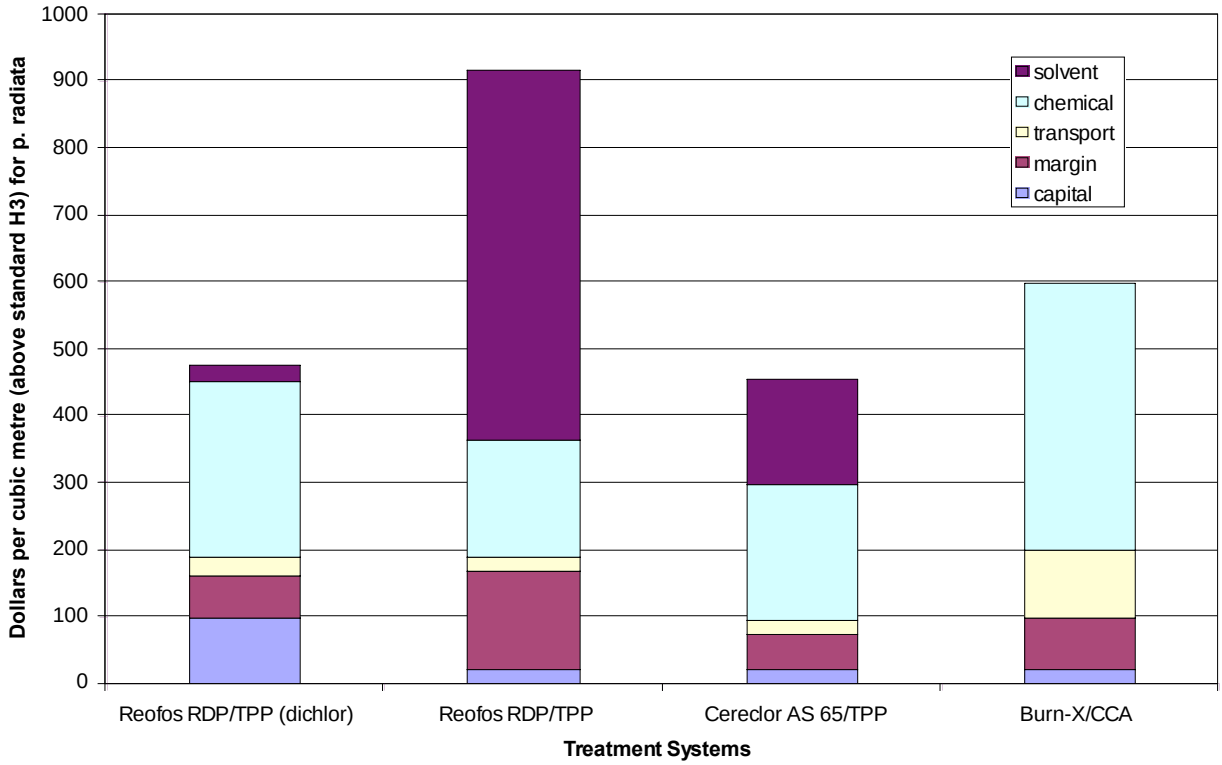


Figure 10: Costing of treatment systems

8. NEW OUTCOMES FROM THIS WORK

A combined fire retardant and preservative treatment for timber that provides resistance to both fire and biodegradation and can be applied using existing technology, is currently not available within Australia. One of the key challenges of this project was to understand how the fundamental properties of the two systems could be combined to produce a novel fire retardant/preservative system where the performance of each component was not compromised.

We identified and selected several fire retardants that appeared suitable in terms of their chemical and physical properties. There was however, no data available as to the loading of fire retardant required for satisfactory performance in timber, nor was there any data relating to the resistance of these fire retardants to weathering when impregnated into timber.

In this work we attempted to resolve some of these issues and the new outcomes that have emerged are:

1. We now have a suite of data on the fire performance of a range of aqueous and LOSP based H3 class wood preservatives, as well as data on the fungal and termite preservative performance of a range of fire retardants.

2. We have timber based fire performance data on fire retardants which are commonly used in polymers.

3. We have articulated the problems associated with co-formulating traditional polymer based fire retardants with H3 class timber preservatives.

4. The aqueous based fire retardant (Burn-X), showed remarkably good fire performance at the recommended dosage rate of 50%. However, at lower dosage rates its fire performance was not as good and was comparable to the other fire retardants considered in this study.

5. Burn-X was able to be co-formulated with the H3 preservative CCA, however, electron microscopic analysis showed that the FR restricted the depth of penetration of this WP into the timber.

6. The boron based compound – trimethylborate (TMB(II) in methanol / white spirit), when applied to a H2 retention level, showed better fire performance (*i.e.*, time to ignition and flame spread parameter) than all of the other organic based FR and WP systems. However, when applied in its proprietary solvent based formulation (TMB(I) in methanol / high flash kerosene / pale boiled linseed oil), it was in fact worse than the others. We know from the literature review that boron based systems will prove problematic on exposure to any weathering regime.

7. When used in combination with other FRs, TMB(II) generally enhanced the performance of the FRs, *i.e.,* it acted as an activator and reduced the PHRR. There appeared to be some interaction occurring between TMB(II) and Cereclor AS 65 in that the PHRR for the combination was lower than for either additive.

8. We have gained an understanding of some of the issues surrounding the penetration and dispersion of CCA and CuN in timber when used in combination with a fire retardant.

9. We have developed an understanding of the problems associated with retaining fire performance in timber after exposure to weathering.

10. We have established that after weathering (according to AS 3959), the retention of copper in timber treated with CuN is low. However when combined with the three halogenated FRs (FR-513, Cereclor AS 65 and PE-68), the uptake of CuN in the timber is enhanced. The FRs seem to 'fix' the copper in the timber thus making the preservative more stable to the weathering regime called for in the standard.

11. Burn-X was leached from the timber during weathering to the point where its fire performance was ineffectual. Electron microscopy analysis showed that the levels of copper in timber treated with CCA were reduced after weathering suggesting that the preservative was also leached from the timber. This raises questions regarding its efficacy after weathering.

12. We have indicated how the anti-fungal / anti-termite activity of CuN was improved when in combination with halogenated fire retardants.

13. We have established that an indicative cost of implementing a single step FR/WP aqueous system (*i.e.*, Burn-X/CCA) was approximately \$600/m^3 above current preservative system costs and the costs for LOSP based systems (*i.e.*, either Cereclor AS 65 or Reofos RDP with TPP) were relatively similar.

APPENDIX 1

A.1.1 Fire retardant and preservative distribution

- *Determination of macro- and micro-distribution of fire retardant and preservative, i.e. quantification of the distribution of chemicals in specimens. This will be undertaken using various organic and inorganic analytical techniques to quantify the amount and location of chemical present in the timber after treatment. Methods will have to be developed for analysis of fire retardants in wood. The systems that have been identified have, to the best of our knowledge, not previously been examined in wood. These studies will also serve to establish if the presence of the fire retardant impacts upon the preservative distribution.* (Refer Detailed Project Proposal, Methodology #4, dot point #1)

- *The analytical methodologies developed above will also serve to quantify the retention of fire retardant remaining in specimens after accelerated weathering (both ultraviolet irradiation and leaching), using a scaled down version of ASTM D2898 Method B. The treated specimens will be exposed to cyclic heating/wetting conditions in a modified QUV cabinet.* (Refer Detailed Project Proposal, Methodology #4, dot point #2)

The depth of penetration and distribution of the additives within the timber after impregnation was evaluated using a Field Emission Scanning Electron Microscope (FE-SEM). We also evaluated whether a fire retardant impacted upon the distribution of the preservative within the timber, along with the effects of weathering upon additive concentration.

We conducted SEM imaging at three locations along the edge (left hand side-LHS, middle and right hand side-RHS) and in the centre (left hand side-LHS, middle and right hand side-RHS) of each sectioned specimen (six locations in all – as shown in Figure 1). The elements of interest appear as bright areas on the back-scattered electron images and the distribution of each element throughout the specimen is shown in Figure 2 to Figure 19. The elements that were monitored from the treatments were bromine in BE-51, FR-513 and PE-68; chlorine in Cereclor AS 65, calcium in Burn-X, phosphorus in Reofos 35 and Reofos RDP, and copper in CCA and CuN. The percentage area coverage of each element at each of the six locations on the specimen is presented in Table 1, Table 2, and Table 3.

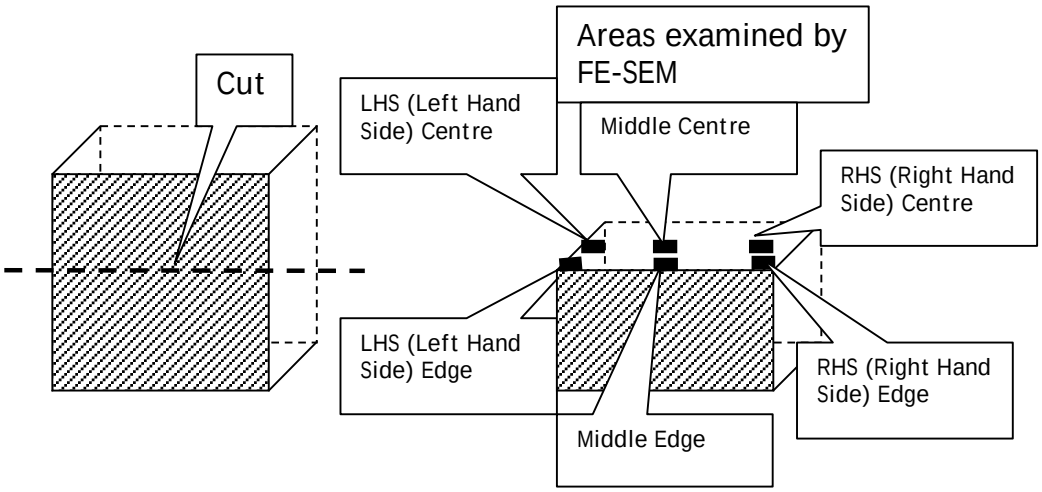


Figure 1: Profile of FE-SEM examination points

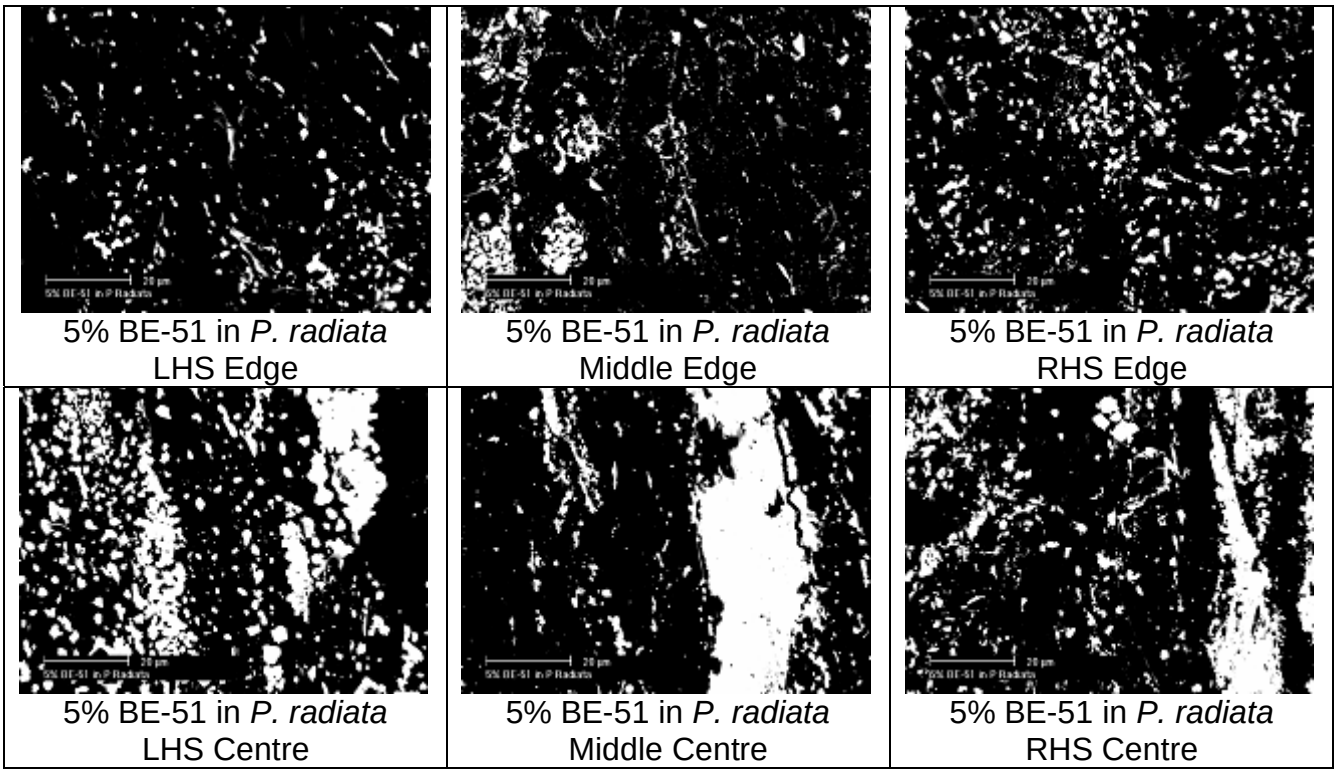


Figure 2: Back scattered electron image showing distribution of bromine in *P. radiata* treated with BE-51 (unweathered)

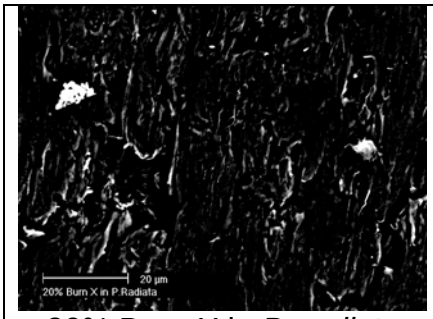
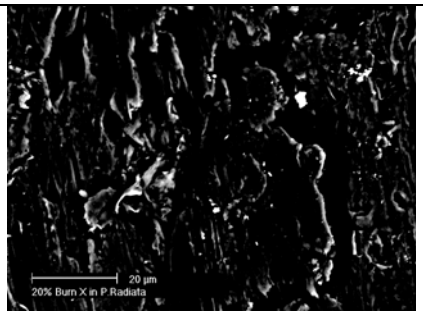
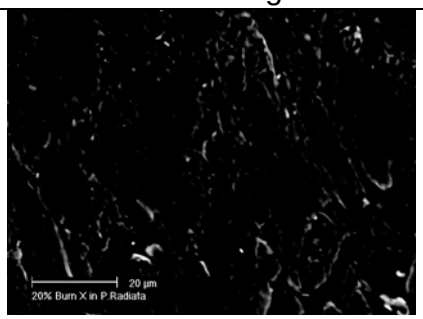
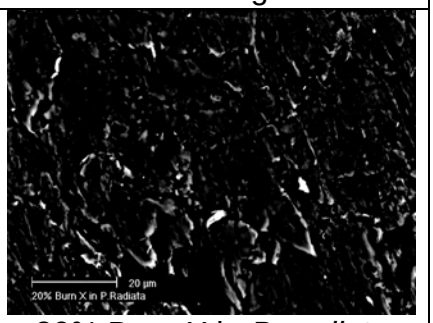
 20% Burn-X in *P. radiata* LHS Edge	 20% Burn-X in *P. radiata* Middle Edge	 20% Burn-X in *P. radiata* RHS Edge
No image available	 20% Burn-X in *P. radiata* Middle Centre	 20% Burn-X in *P. radiata* RHS Centre

Figure 3: Back scattered electron image showing distribution of calcium in *P. radiata* treated with Burn-X (unweathered)

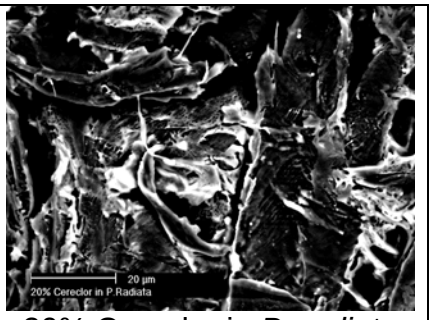
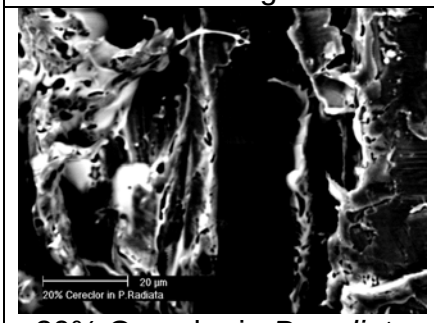
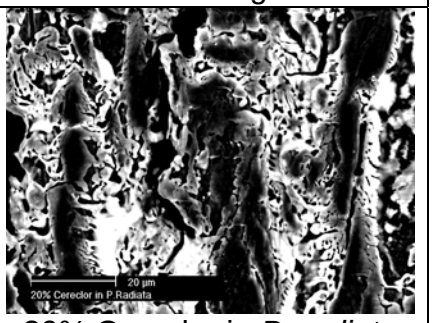
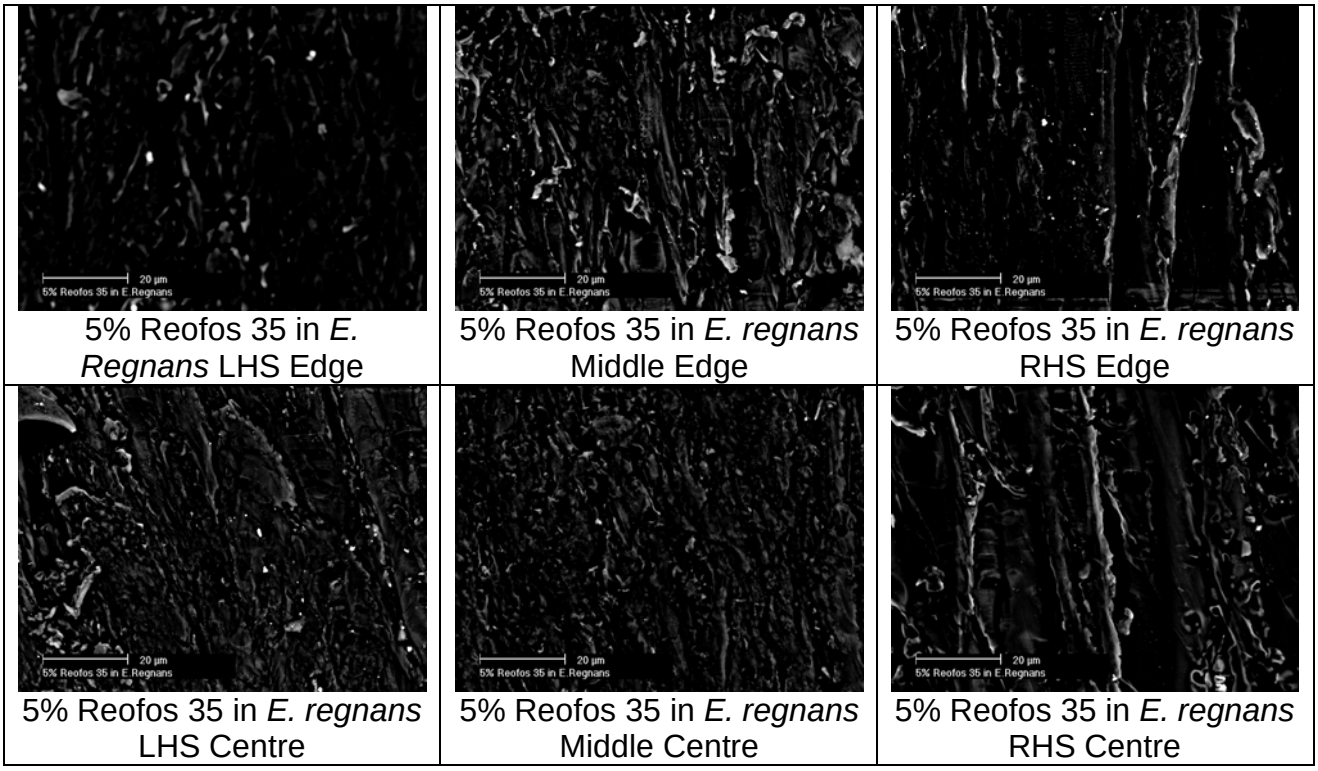
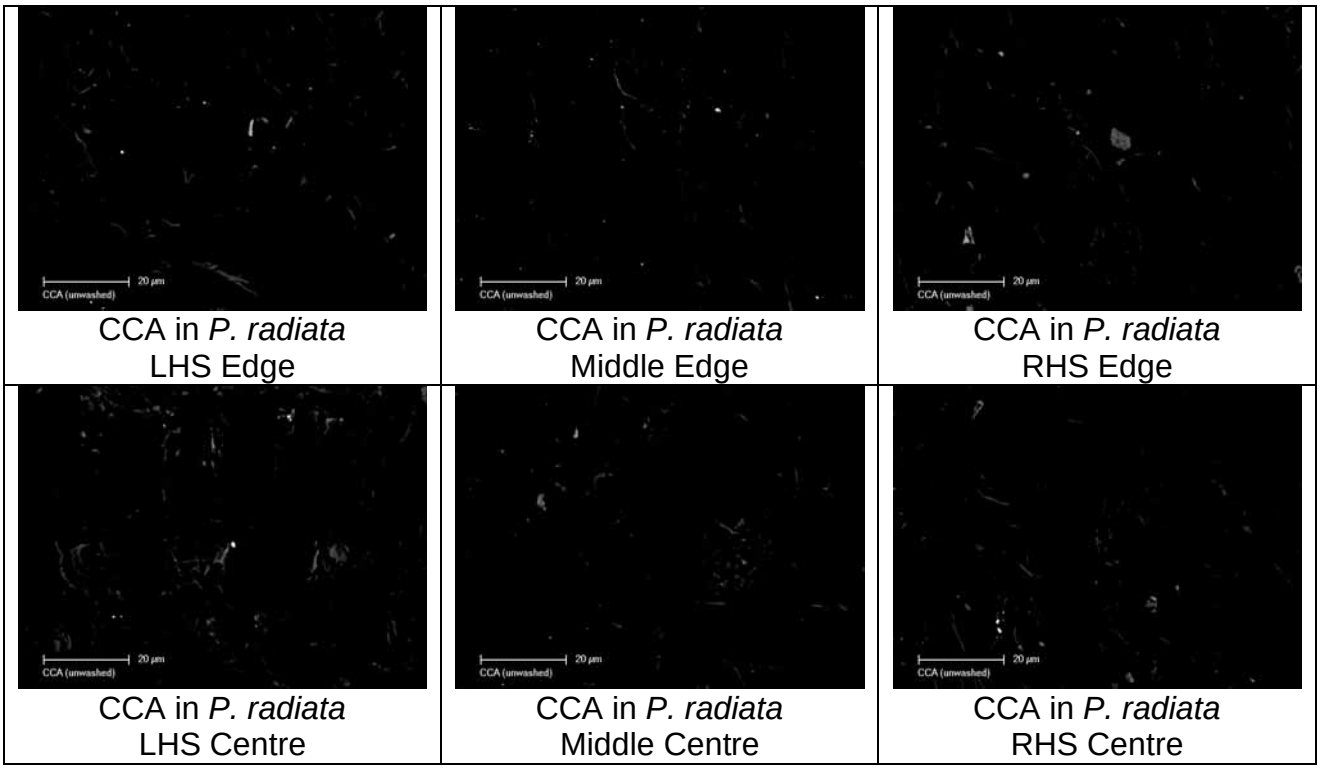
 20% Cereclor in *P. radiata* LHS Edge	 20% Cereclor in *P. radiata* Middle Edge	 20% Cereclor in *P. radiata* RHS Edge
 20% Cereclor in *P. radiata* LHS Centre	 20% Cereclor in *P. radiata* Middle Centre	 20% Cereclor in *P. radiata* RHS Centre

Figure 4: Back scattered electron image showing distribution of chlorine in *P. radiata* treated with Cereclor AS 65 (unweathered)



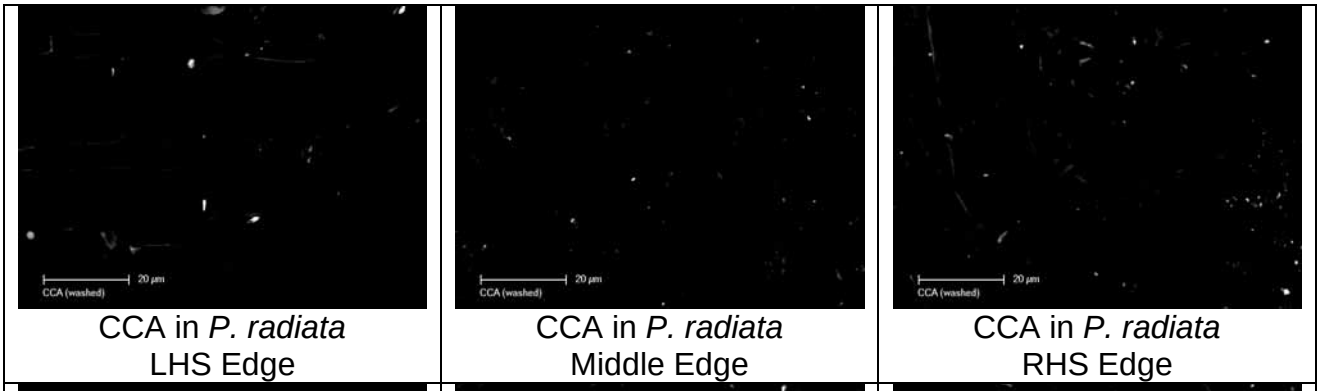
5% Reofos 35 in *E. Regnans* LHS Edge	5% Reofos 35 in *E. regnans* Middle Edge	5% Reofos 35 in *E. regnans* RHS Edge
5% Reofos 35 in *E. regnans* LHS Centre	5% Reofos 35 in *E. regnans* Middle Centre	5% Reofos 35 in *E. regnans* RHS Centre

Figure 5: Back scattered electron image showing distribution of phosphorus in *E. regnans* treated with Reofos 35 (unweathered)



CCA in *P. radiata* LHS Edge	CCA in *P. radiata* Middle Edge	CCA in *P. radiata* RHS Edge
CCA in *P. radiata* LHS Centre	CCA in *P. radiata* Middle Centre	CCA in *P. radiata* RHS Centre

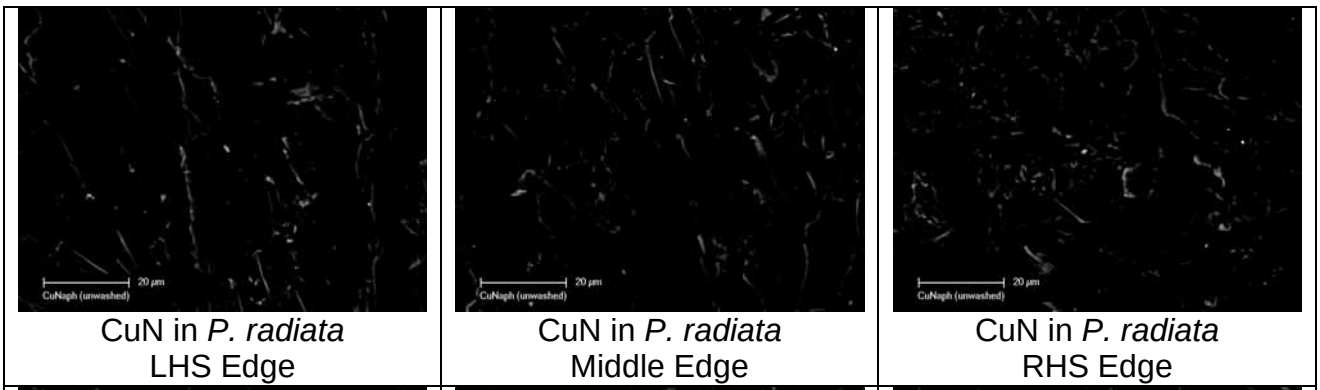
Figure 6: Back scattered electron image showing distribution of copper in *P. radiata* treated with CCA to H3 level (unweathered)



CCA in *P. radiata* LHS Edge | CCA in *P. radiata* Middle Edge | CCA in *P. radiata* RHS Edge

CCA in *P. radiata* LHS Centre | CCA in *P. radiata* Middle Centre | CCA in *P. radiata* RHS Centre

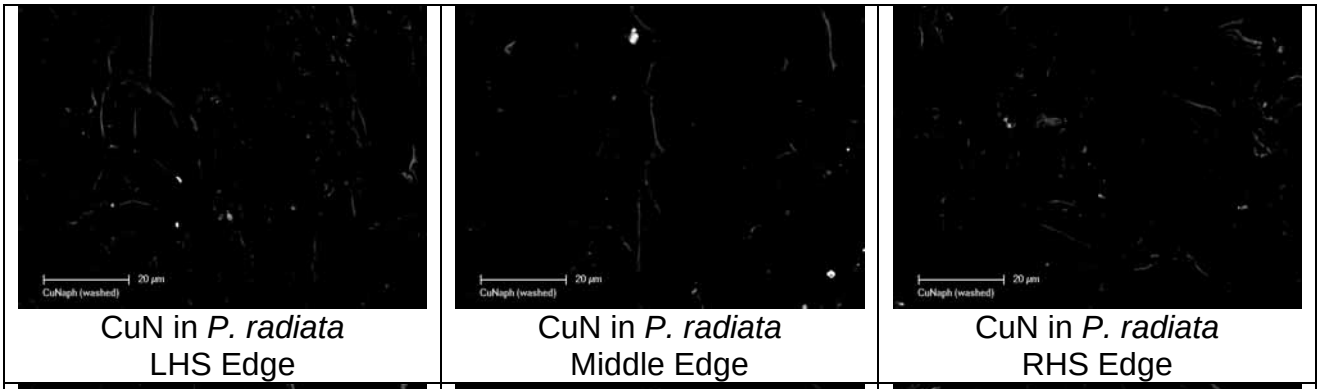
Figure 7: Back scattered electron image showing distribution of copper in *P. radiata* treated with CCA to H3 level (weathered to AS 3959)



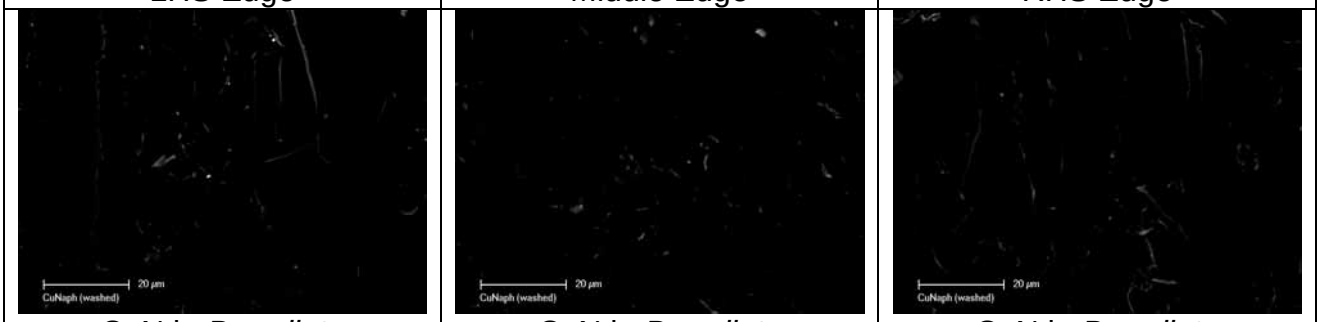
CuN in *P. radiata* LHS Edge | CuN in *P. radiata* Middle Edge | CuN in *P. radiata* RHS Edge

CuN in *P. radiata* LHS Centre | CuN in *P. radiata* Middle Centre | CuN in *P. radiata* RHS Centre

Figure 8: Back scattered electron image showing distribution of copper in *P. radiata* treated with CuN to H3 level (unweathered)



CuN in *P. radiata* LHS Edge | CuN in *P. radiata* Middle Edge | CuN in *P. radiata* RHS Edge



CuN in *P. radiata* LHS Centre | CuN in *P. radiata* Middle Centre | CuN in *P. radiata* RHS Centre

Figure 9: Back scattered electron image showing distribution of copper in *P. radiata* treated with CuN to H3 level (weathered to AS 3959)

Table 1: Distribution of fire retardants and wood preservatives after separate treatment (and weathering)

Treatment	Element of Interest	% Area Coverage & Location of Analysis Area					
		LHS Edge	Middle Edge	RHS Edge	LHS Centre	Middle Centre	RHS Centre
BE-51 in *P. radiata*	Bromine	8.5%	14.3%	14.5%	30.5%	28.3%	24.1%
Burn-X in *P. radiata*	Calcium	10.3%	12.0%	19.4%	N/A	3.4%	9.5%
Cereclor AS 65 in *P. radiata*	Chlorine	76.1%	50.2%	39.6%	36.4%	50.2%	57.0%
Reofos 35 in *E. regnans*	Phosphorus	3.0%	13.2%	6.3%	10.3%	6.3%	8.7%
CCA in *P. radiata*	Copper	0.47 (0.48)	0.21 (0.17)	0.63 (0.50)	0.93 (0.19)	0.31 (0.90)	0.45 (0.74)
CuN in *P. radiata*	Copper	0.15 (0.06)	0.07 (0.14)	0.13 (0.04)	0.09 (0.03)	0.21 (0.02)	0.33 (0.01)

Findings

The findings from Figure 2 to Figure 9 and Table 1, regarding the distribution of fire retardants and wood preservatives after separate treatments and subsequent weathering were as follows:

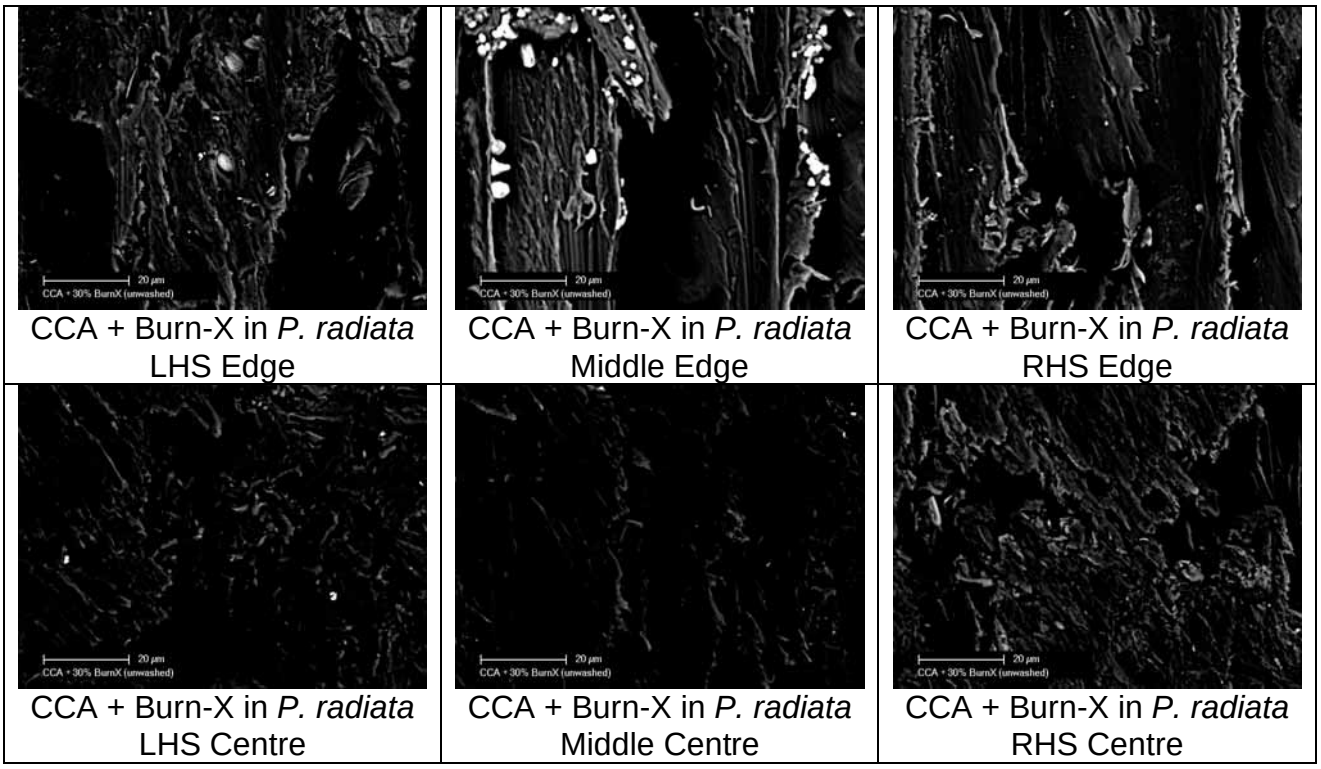
1. The white spirit based fire retardants, BE-51 and Cereclor AS 65 were able to penetrate through to the centre of the timber specimen with around 30% bromine and around 50% of chlorine detected at this depth.

2. The white spirit based Reofos 35 and water based Burn-X did not penetrate the timber as well as BE-51 and Cereclor AS-65 with only around 10% of the element of interest detected in the centre of the treated timber specimens.

3. The distribution of chlorine, shown in Table 1, indicated that there was good dispersion of Cereclor AS 65 throughout the timber specimen.

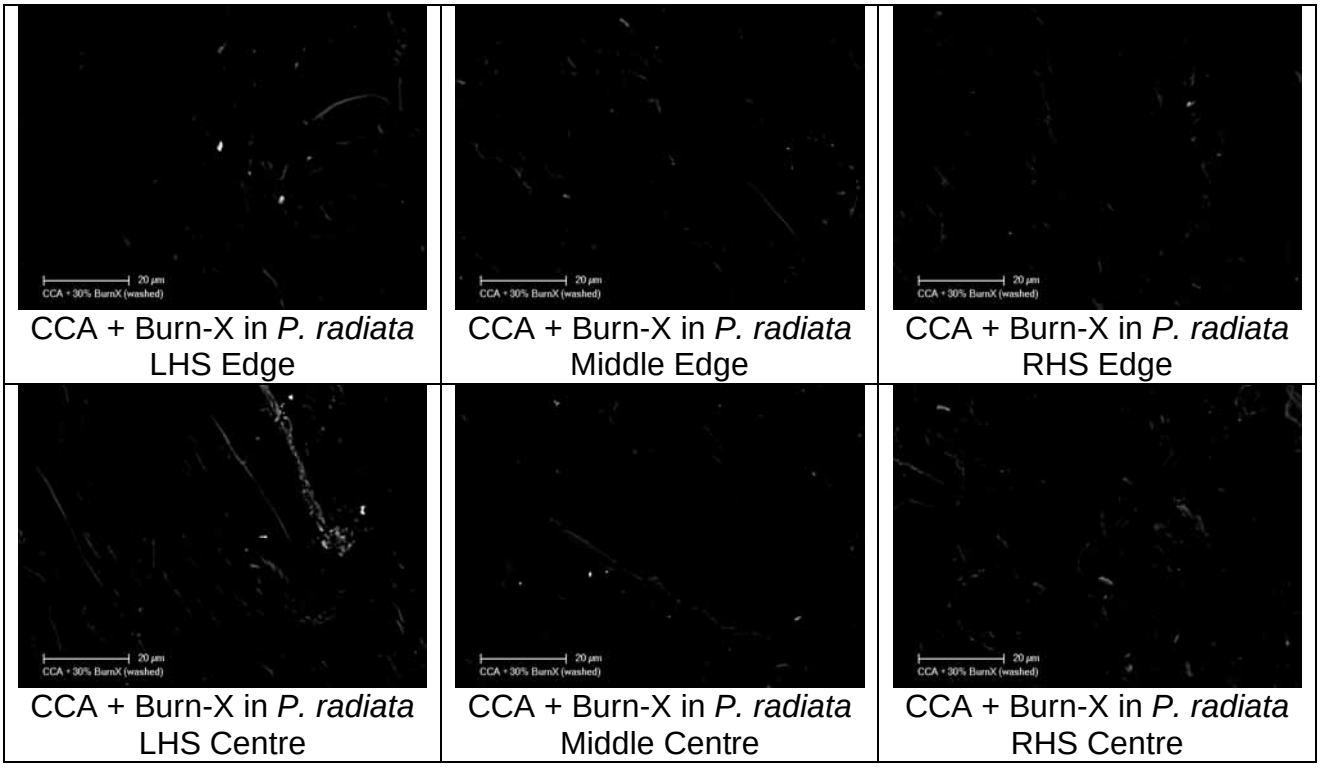
4. The distribution of bromine, calcium and phosphorus, shown in Figure 2**,** Figure 3 and Figure 5 respectively, was very uneven indicating that the dispersion of BE-51, Burn X and Reofos 35 was not uniform.

5. The CCA treatment resulted in a slightly higher retention of copper within the timber compared to CuN. The weathering regime employed in AS 3959, had relatively little impact upon the retention of copper in CCA. Whilst there was relatively little copper retained in CuN treated sample after weathering.



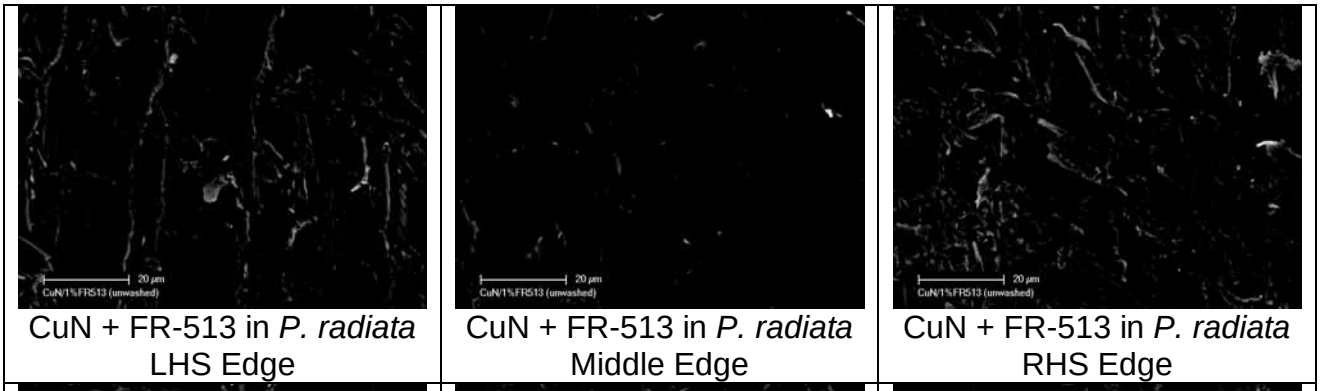
CCA + Burn-X in *P. radiata* LHS Edge	CCA + Burn-X in *P. radiata* Middle Edge	CCA + Burn-X in *P. radiata* RHS Edge
CCA + Burn-X in *P. radiata* LHS Centre	CCA + Burn-X in *P. radiata* Middle Centre	CCA + Burn-X in *P. radiata* RHS Centre

Figure 10: Back scattered electron image showing distribution of copper in *P. radiata* treated with CCA + Burn-X (unweathered)



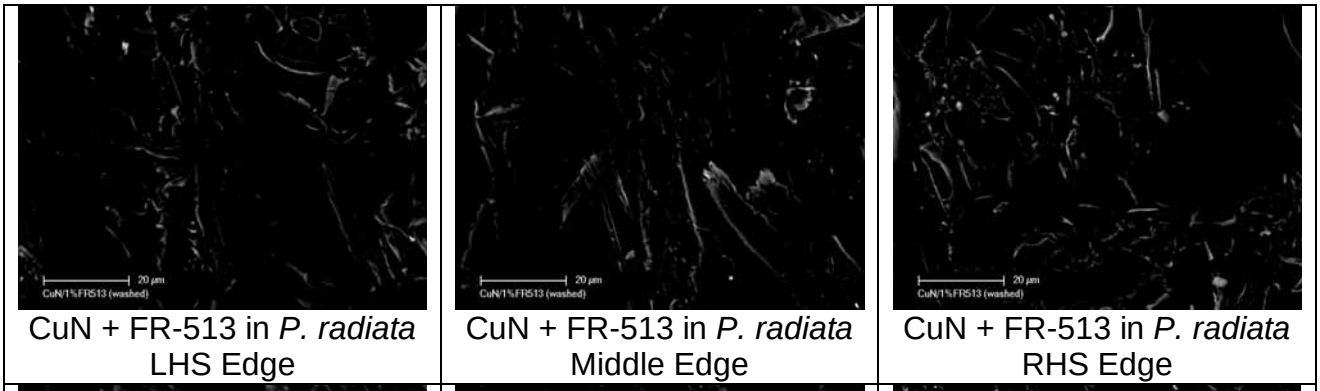
CCA + Burn-X in *P. radiata* LHS Edge	CCA + Burn-X in *P. radiata* Middle Edge	CCA + Burn-X in *P. radiata* RHS Edge
CCA + Burn-X in *P. radiata* LHS Centre	CCA + Burn-X in *P. radiata* Middle Centre	CCA + Burn-X in *P. radiata* RHS Centre

Figure 11: Back scattered electron image showing distribution of copper in *P. radiata* treated with CCA + Burn-X (weathered to AS 3959)



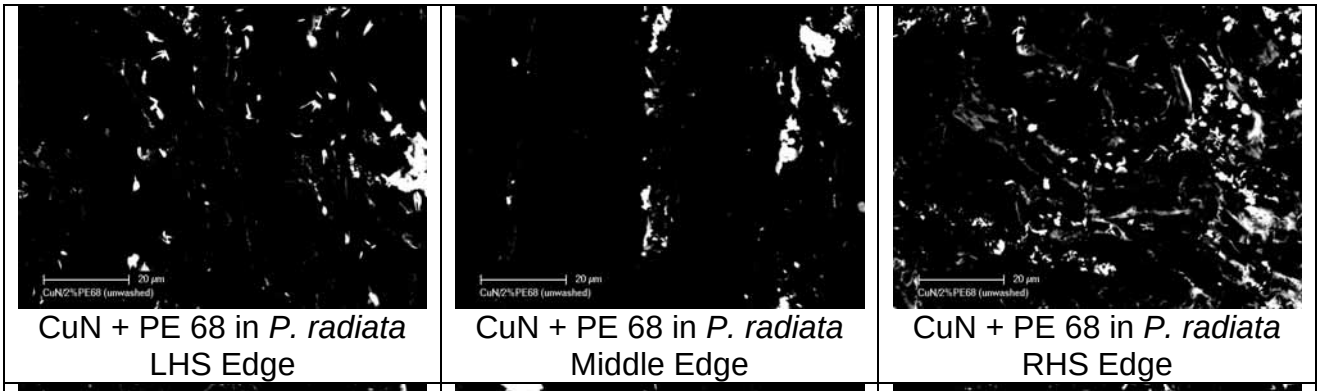
CuN + FR-513 in *P. radiata* LHS Edge	CuN + FR-513 in *P. radiata* Middle Edge	CuN + FR-513 in *P. radiata* RHS Edge
CuN + FR-513 in *P. radiata* LHS Centre	CuN + FR-513 in *P. radiata* Middle Centre	CuN + FR-513 in *P. radiata* RHS Centre

Figure 12: Back scattered electron image showing distribution of copper in *P. radiata* treated with CuN + FR-513 (unweathered)



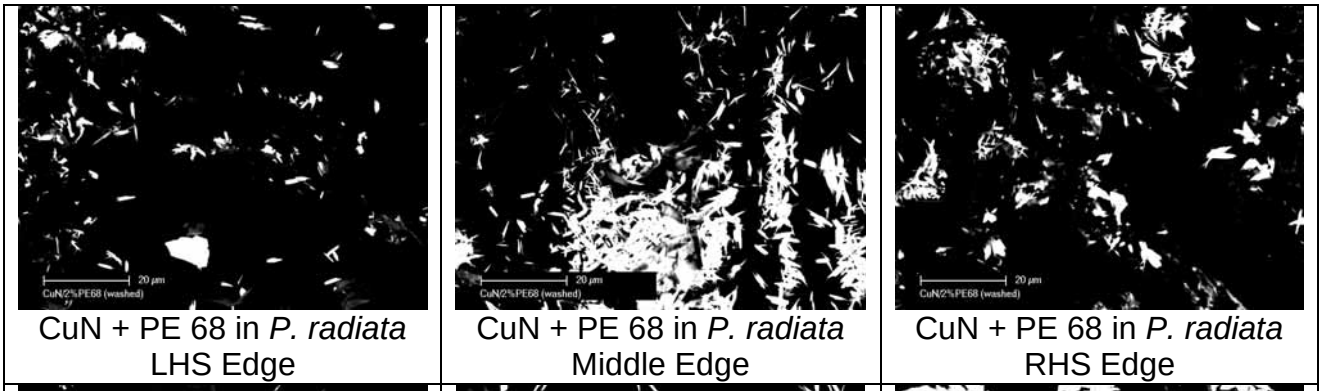
CuN + FR-513 in *P. radiata* LHS Edge	CuN + FR-513 in *P. radiata* Middle Edge	CuN + FR-513 in *P. radiata* RHS Edge
CuN + FR-513 in *P. radiata* LHS Centre	CuN + FR-513 in *P. radiata* Middle Centre	CuN + FR-513 in *P. radiata* RHS Centre

Figure 13: Back scattered electron image showing distribution of copper in *P. radiata* treated with CuN + FR-513 (weathered to AS 3959)



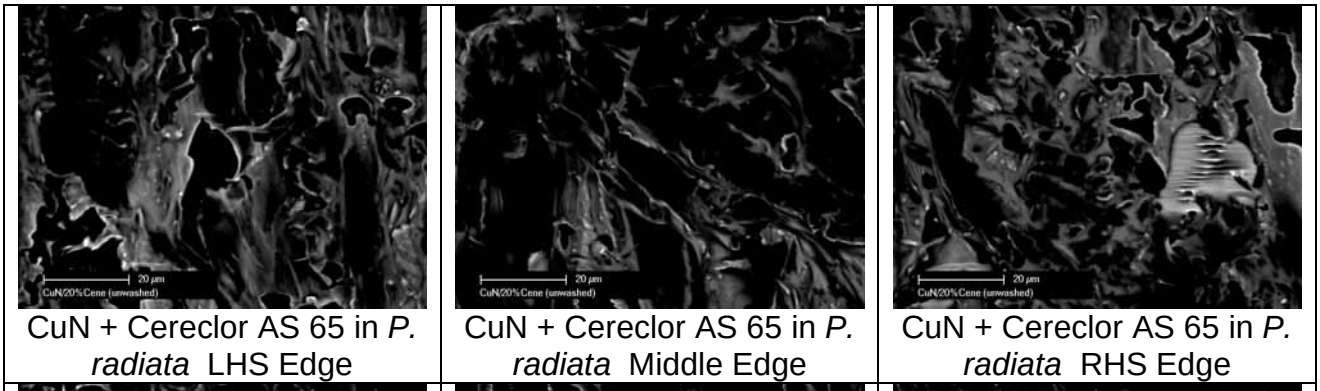
CuN + PE 68 in *P. radiata* LHS Edge	CuN + PE 68 in *P. radiata* Middle Edge	CuN + PE 68 in *P. radiata* RHS Edge
CuN + PE 68 in *P. radiata* LHS Centre	CuN + PE 68 in *P. radiata* Middle Centre	CuN + PE 68 in *P. radiata* RHS Centre

Figure 14: Back scattered electron image showing distribution of copper in *P. radiata* treated with CuN + PE 68 (unweathered)



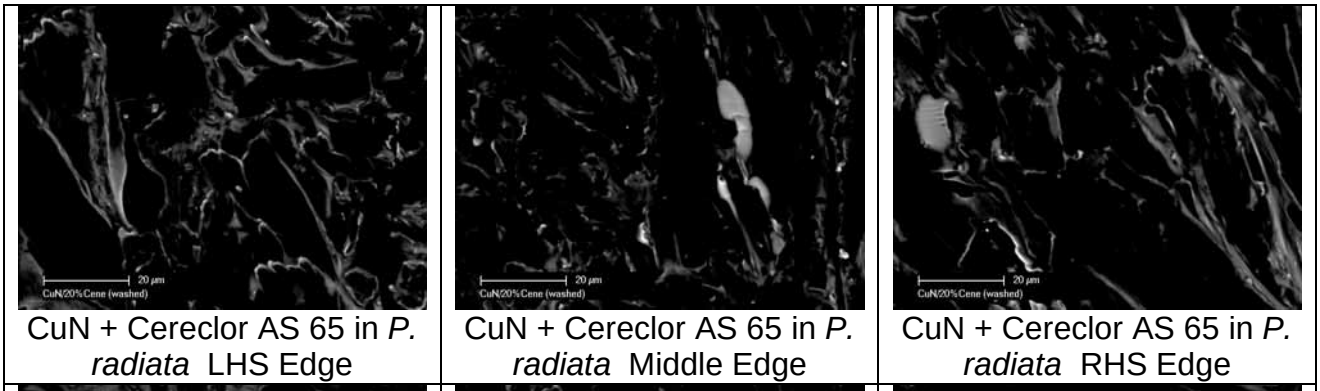
CuN + PE 68 in *P. radiata* LHS Edge	CuN + PE 68 in *P. radiata* Middle Edge	CuN + PE 68 in *P. radiata* RHS Edge
CuN + PE 68 in *P. radiata* LHS Centre	CuN + PE 68 in *P. radiata* Middle Centre	CuN + PE 68 in *P. radiata* RHS Centre

Figure 15: Back scattered electron image showing distribution of copper in *P. radiata* treated with CuN + PE 68 (weathered to AS 3959)



CuN + Cereclor AS 65 in *P. radiata* LHS Edge	CuN + Cereclor AS 65 in *P. radiata* Middle Edge	CuN + Cereclor AS 65 in *P. radiata* RHS Edge
CuN + Cereclor AS 65 in *P. radiata* LHS Centre	CuN + Cereclor AS 65 in *P. radiata* Middle Centre	CuN + Cereclor AS 65 in *P. radiata* RHS Centre

Figure 16: Back scattered electron image showing distribution of copper in *P. radiata* treated with CuN + Cereclor AS 65 (unweathered)



CuN + Cereclor AS 65 in *P. radiata* LHS Edge	CuN + Cereclor AS 65 in *P. radiata* Middle Edge	CuN + Cereclor AS 65 in *P. radiata* RHS Edge
CuN + Cereclor AS 65 in *P. radiata* LHS Centre	CuN + Cereclor AS 65 in *P. radiata* Middle Centre	CuN + Cereclor AS 65 in *P. radiata* RHS Centre

Figure 17: Back scattered electron image showing distribution of copper in *P. radiata* treated with CuN + Cereclor AS 65 (weathered to AS 3959)

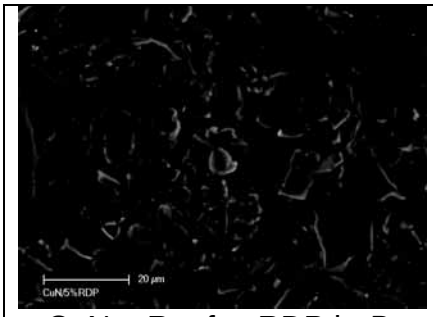
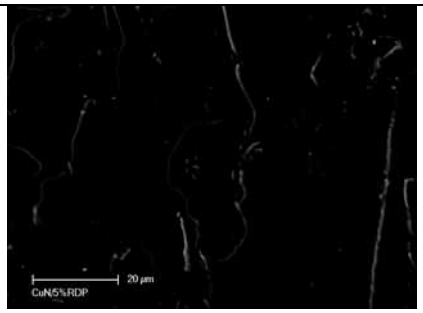
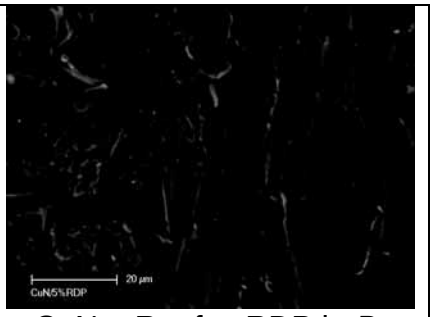
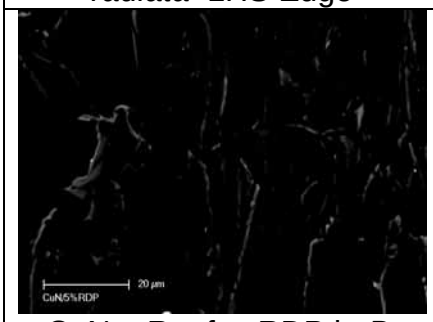
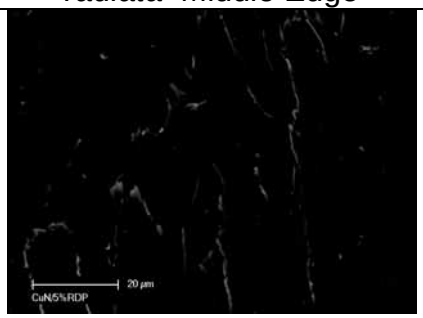
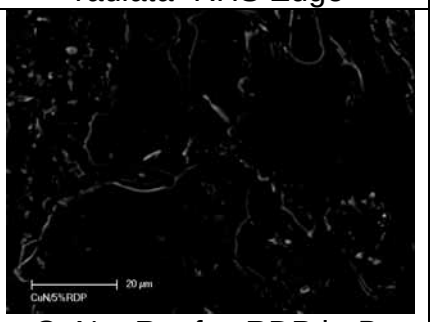
		
CuN + Reofos RDP in *P. radiata* LHS Edge	CuN + Reofos RDP in *P. radiata* Middle Edge	CuN + Reofos RDP in *P. radiata* RHS Edge
		
CuN + Reofos RDP in *P. radiata* LHS Centre	CuN + Reofos RDP in *P. radiata* Middle Centre	CuN + Reofos RDP in *P. radiata* RHS Centre

Figure 18: Back scattered electron image showing distribution of copper in *P. radiata* treated with CuN + Reofos RDP (unweathered)

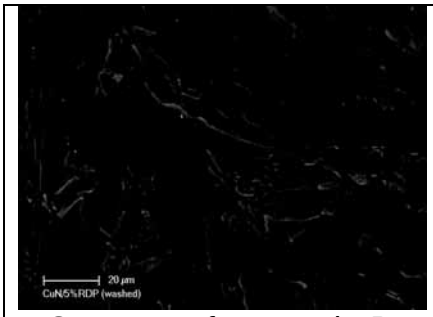
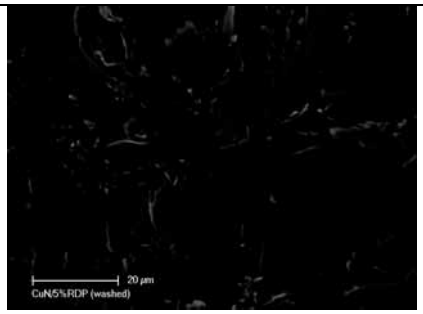
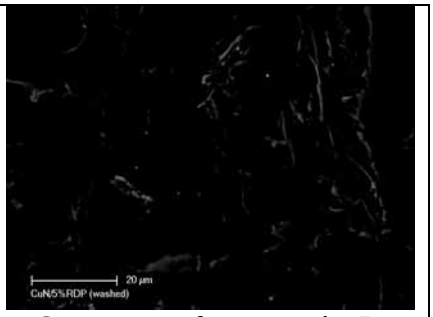
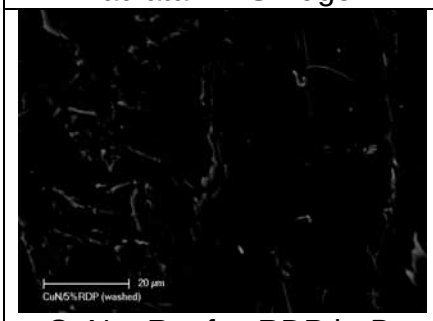
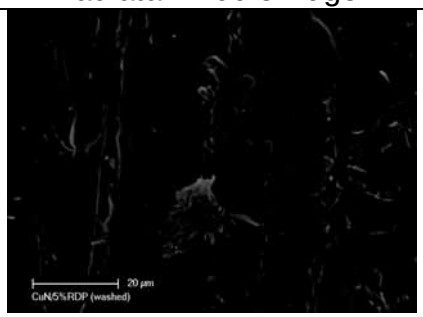
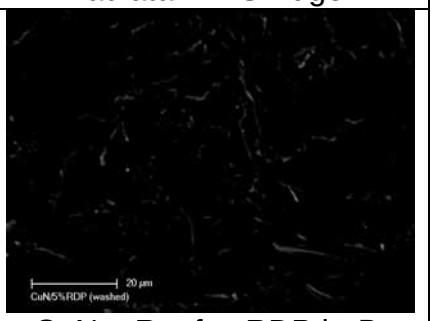
		
CuN + Reofos RDP in *P. radiata* LHS Edge	CuN + Reofos RDP in *P. radiata* Middle Edge	CuN + Reofos RDP in *P. radiata* RHS Edge
		
CuN + Reofos RDP in *P. radiata* LHS Centre	CuN + Reofos RDP in *P. radiata* Middle Centre	CuN + Reofos RDP in *P. radiata* RHS Centre

Figure 19: Back scattered electron image showing distribution of copper in *P. radiata* treated with CuN + Reofos RDP (weathered to AS 3959)

Table 2: Distribution of wood preservatives after co-treatment with fire retardants (and weathering)

Treatment	Element of Interest	% Area Coverage & Location of Analysis Area					
		LHS Edge	Middle Edge	RHS Edge	LHS Centre	Middle Centre	RHS Centre
CCA + Burn-X in *P. radiata*	Copper	1.9 (0.06)	2.6 (0.04)	1.6 (0.02)	0.29 (0.15)	0.15 (0.05)	0.19 (0.10)
CuN + FR513 in *P. radiata*	Copper	0.34 (0.24)	0.19 (0.35)	0.55 (0.49)	0.51 (0.27)	0.17 (0.11)	0.53 (0.34)
CuN + Reofos RDP in *P. radiata*	Copper	0.20 (0.02)	0.06 (0.01)	0.05 (0.02)	0.10 (0.02)	0.06 (0.01)	0.15 (0.01)
CuN + Cereclor AS 65 in *P. radiata*	Copper	5.2 (1.7)	1.8 (2.1)	6.7 (2.7)	3.0 (3.7)	2.8 (0.86)	2.2 (3.8)
CuN + PE 68 in *P. radiata*	Copper	3.0 (4.5)	2.5 (20.5)	5.3 (7.5)	1.7 (1.0)	1.5 (7.7)	6.5 (10.7)

Findings

The findings from Figure 10 to Figure 19 and Table 2, regarding the penetration and distribution of wood preservatives after co-treatments with fire retardants and subsequent weathering were as follows:

1. The treatment of timber with a combined Burn-X and CCA system resulted in elevated levels of CCA on the edge of the specimen, and a reduction in CCA in the centre of the specimen.
2. Weathering of the co-treated sample resulted in the loss of most of the CCA from the specimen; *i.e.*, the presence of Burn-X reduced the fixability of the CCA. Burn-X prevented the deeper penetration of CCA causing a build up of CCA on the edges (near the surface) allowing this to be easily removed by weathering.
3. The brominated fire retardant, FR-513, enhanced the penetration and distribution of CuN. In addition the fire retardant acted to fix the retention of CuN in the timber thus making it more stable to weathering.
4. The data for phosphorus based fire retardant, Reofos RDP, is somewhat scattered, and it was difficult to draw a clear set of conclusions, apart from the statement that the FR did not enhance the uptake of CuN, nor did it assist in fixing CuN into the timber.
5. The fire retardant additive, Cereclor AS 65, acted to enhance the uptake of CuN into the timber; as well it fixed a relatively high level of the preservative into the timber making it stable to the weathering regime employed in AS 3959.
6. The fire retardant PE-68, like Cereclor AS 65, also improved the uptake of CuN in the timber. This FR fixed a much larger proportion of CuN in the timber, allowing a relatively high amount of CuN to be retained in the timber after weathering.
7. Of the two brominated fire retardants (FR-513 and PE-68), the latter enhanced the uptake of CuN within the timber.

Table 3: Distribution of fire retardants after co-treatment with wood preservatives (and weathering)

Treatment	Element of Interest	% Area Coverage & Location of Analysis Area					
		LHS Edge	Middle Edge	RHS Edge	LHS Centre	Middle Centre	RHS Centre
Burn-X in *P. radiata*	Calcium	10.3%	12.0%	19.4%	21.6%	3.4%	9.5%
CCA + Burn-X *P. radiata*	Calcium	11.8 (0.29)	26.1 (0.18)	16.6 (0.11)	3.9 (1.1)	1.6 (0.10)	13.1 (0.44)
Cereclor AS 65 in *P. radiata*	Chlorine	76.1%	50.2%	39.6%	36.4%	50.2%	57.0%
CuN + Cereclor AS 65 in *P. radiata*	Chlorine	39.3 (18.8)	24.0 (12.9)	47.8 (17.8)	19.8 (36.3)	15.2 (8.8)	22.0 (17.6)
CuN + FR513 *P. radiata*	Bromine	4.9 (4.2)	4.3 (4.6)	6.5 (5.3)	5.1 (5.4)	3.6 (3.4)	6.0 (5.2)
CuN + Reofos RDP in *P. radiata*	Phosphorus	4.9 (2.0)	2.1 (2.0)	2.5 (2.6)	3.6 (2.6)	1.9 (2.4)	4.0 (1.9)
CuN + PE 68 in *P. radiata*	Bromine	5.3 (7.8)	4.0 (27.2)	14.6 (12.9)	5.7 (3.2)	2.4 (9.2)	8.8 (12.8)

Findings

The findings from Figure 2 to Figure 19 and Table 3, regarding the penetration and distribution of fire retardants after co-treatments with wood preservatives and subsequent weathering were as follows:

1. The distribution of Burn-X as indicated by the calcium distribution, was relatively unaffected by the presence of CCA, however, as expected, the level was strongly influenced by the weathering; *i.e.*, the level was reduced to less than 5% of what was originally present.

2. The presence of CuN, acted to reduce the amount of the FR, Cereclor AS 65, taken up by the timber. In addition its presence acted as some sort of barrier limiting the penetration of chlorine. Upon weathering, the amount of FR retained in the timber was further reduced, and was more equally distributed between the centre and the outer edges.

3. The weathering regime had relatively little effect upon the amount of the fire retardant FR-513 retained within the timber, when it was co-added with CuN.

4. Weathering seemed to reduce the amount of Reofos RDP retained in timber in the presence of CuN.

5. The amount of the brominated FR, PE-68 was relatively unaffected by the weathering regime in the presence of CuN.

A summary of the findings were:

- The two halogenated fire retardants, BE-51 and Cereclor AS 65, were well distributed within the timber. In addition a relatively high amount of FR penetrated to the centre of the timber specimen.
- The CCA treatment delivered more additive to the timber than CuN.
- All of the LOS based fire retardants studied except for the Reofos RDP, enhanced the uptake of CuN in the timber. Burn-X acted to limit penetration of the CCA into the timber.
- The halogenated FRs seemed to fix the copper in the timber thus making the preservative stable to weathering.
- The presence of Burn-X resulted in a substantial loss of CCA during weathering according to AS 3959. The questions arising from this are:
    - Is the preservative still active after this weathering regime?
    - Is AS 3959 relevant for preservatives?

APPENDIX 2

A.2.1 Activators – the use of boron

- *Exploring the effects of activators. Improvement in performance may be obtained by the addition of an activator. This approach can be utilised to optimise the fire retardant performance so that smoke emission is reduced, flame spread is limited, heat of combustion is reduced and the rate of combustion is limited.* (Refer Detailed Project Proposal, Methodology #4, dot point #4)

In the milestone 6 report we explored the possibility that activators may improve the fire retardant performance. In this context we examined the potential for our target H3 preservatives to act as activators of fire retardants. However we did not investigate the capacity of boron in the form of boric acid equivalent to do the same. This additional portion of work presents these results.

The use of boron was considered worth studying even though it was not a H3 preservative in its own right. This was due to the known ability of boron to act as a fire retardant adjunct[1].

A.2.1.1 Treatment of timber

P. radiata was the only timber species used for this work. Trimethyl borate (TMB(II)) in methanol), was diluted with white spirit before being co-formulated with three LOS based fire retardants for subsequent impregnation of the timber. The treatment level of TMB(II) was designed to give a boron equivalent in the timber of 8%.

The timber specimens, measuring 50 mm x 50 mm x 10 mm (length x width x height), were treated using a full-cell process. The specimens were weighted down in a vacuum desiccator and a vacuum of -90 kPa was applied for 30 minutes. The treatment solution was admitted to the desiccator under vacuum, after which the vacuum was released and the specimens left to adsorb solution at atmospheric pressure for 60 minutes. Each specimen was weighed before and after treatment to determine the uptake. After treatment, the specimens were wrapped in plastic bags and left for one week, then slowly air-dried. The specimens were then vacuum oven dried at -90 kPa and 40 °C for five days, after which they were reconditioned to an e.m.c. of approximately 10%.

[1] *Le Van, S.L. and Tran, H.C. (1990) The role of boron in flame-retardant treatments. 1st International Conference on Wood Protection with Diffusible. Nashville, T.E. ed. M. Hamel, Forest Products Research Society, pp. 39-41.*

Table 4: Treatments included in this work

Treatment	Carrier	Treatment Level	Wt % of Additive
P. radiata			
Untreated			
TMB(II)	LOS		8% B
FR-513	LOS	1%	1%
Reofos RDP	LOS	5%	4.25%
Cereclor AS 65	LOS	20%	20.6%
TMB(II)/FR-513	LOS		8% B and 1% FR-513
TMB(II)/Reofos RDP	LOS		8% B and 5% Reofos RDP
TMB(II)/Cereclor AS 65	LOS		8% B and 20.6% Cereclor AS 65

A.2.1.2 Results and discussion

Figure 20 indicates that timber treated with TMB(II) by itself, has significant fire performance in its own right (*i.e.*, long time to ignition and relatively low heat release rate[2]). This figure also shows that when combined with FR-513, the TMB(II) improves the TTI of the FR but burns with a slightly greater intensity, *i.e.*, higher PHRR. However when combined with either Reofos RDP or Cereclor AS 65, it significantly improves both the TTI and PHRR of each of the FRs, *i.e.,* higher TTI and lower PHRR.

Interestingly, when TMB(II) is combined with Cereclor AS 65, the PHRR is less than that of both the additives indicating that there is some synergistic interaction occurring between the two additives.

Findings

- TMB(II) in combination with other FRs generally enhances the performance of the FRs; *i.e.*, it acts as an activator, as shown in Figure 20.
- There appears to be some interaction occurring between TMB(II) and Cereclor AS 65 in that the PHRR for the combination is lower than for either additive.

[2] *Heat release rate is a measure of a material's ability to spread a flame or fire.*

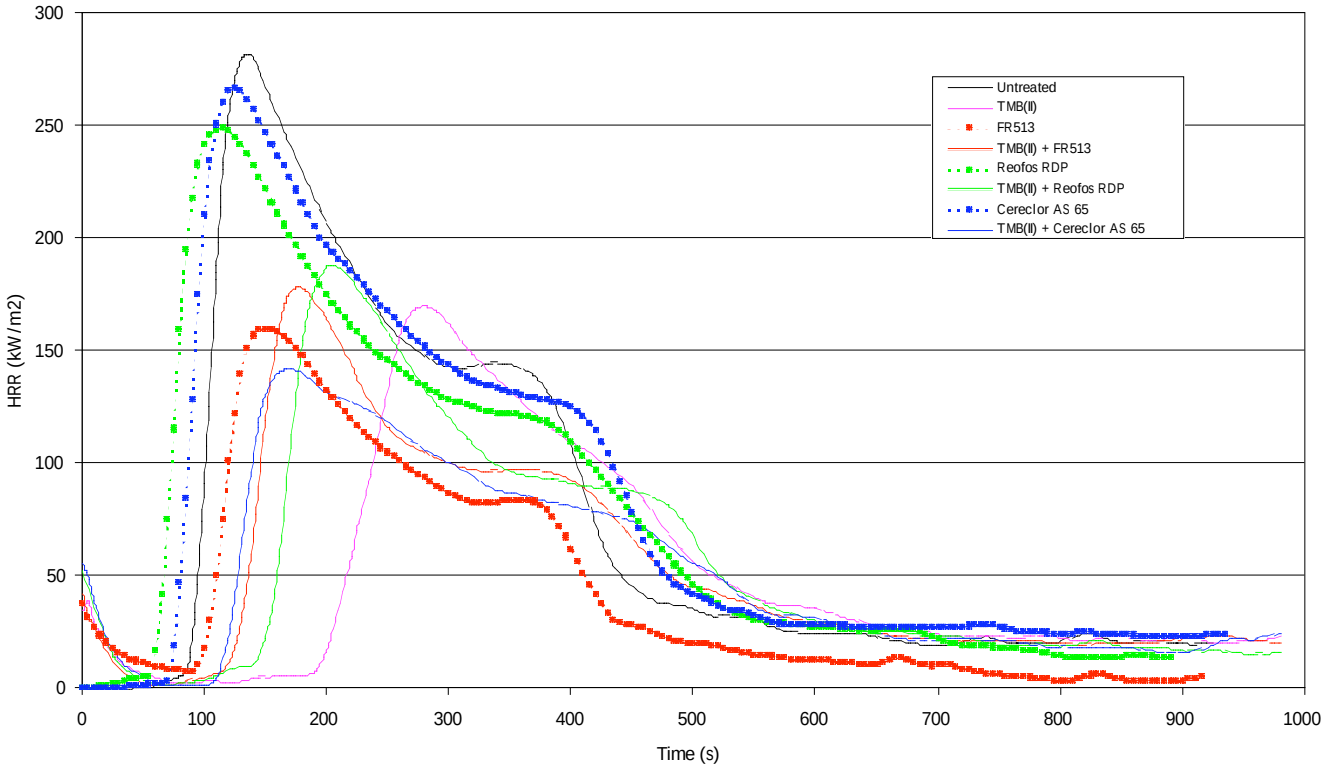


Figure 20: Heat release rate curves for *P. radiata* treated with TMB(II), FRs, a combination of both, as well as untreated timber at a radiation of 25 kWm^{-2}