

Factors affecting distribution of borate to protect building envelope components from biodegradation

by

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Abstract

Borate can be a potential candidate to protect building envelope components from biodegradation as it has low toxicity and can penetrate wood without pressure treatment, even in the refractory species commonly used in construction industries as structural components. In this research, wood moisture content, grain direction, formulation and species that affect the diffusion of borate in refractory species were investigated. Two highly concentrated formulations were applied and a novel approach (borate bandage) was used to keep the preservative on the surface and enhance the diffusion by reducing surface drying. From ANOVA test for different diffusion periods and depths of penetration, it was found that grain directions and moisture content are significant factors. A mould test was performed, the diffusion co-efficients were calculated and some recommendations were made about the quantity required to protect a specific volume of wood considering the distance moved by diffusion and volume treated in different directions.

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List of Abbreviations

ACQ	alkaline copper quat
ANOVA	analysis of variance
ASTM	American society for testing and materials
AWPA	American wood protection association
BAE	boric acid equivalent
CCA	chromated copper arsenate
CLT	cross laminated timber
Cu bor	copper borate
Cu Mea	copper monoethanolamine
DDAC	didecyldimethyl ammonium chloride
D-fir	Douglas-fir
DOT	disodium octaborate tetrahydrate
EPA	environment protection agency
Gly bor	glycerol borate
Glulam	Glue laminated timber
ICP	inductive coupled plasma
MC	moisture content
NEWBuildS	network for engineered wood-based building systems
NSERC	natural sciences and engineering research council of Canada
PVAC	polyvinyl acetate
R.H.	relative humidity
SciDAVis	scientific data analysis and visualization

CHAPTER 1 INTRODUCTION AND RESEARCH CONTEXT

1.1 Introduction

The energy crisis of the 1970s brought forth the rapid evolution of building materials and construction practices that improved the energy efficiency of the building envelope system by making walls tighter. However, they tend to stay wet longer due to the lack of water drainage and lack of air circulation to facilitate the evaporation process. As a result, often there accumulates enough moisture to cause severe problems like decay and mould growth (e.g. 'leaky condo' crisis in Vancouver).

Wood structures have the ability to absorb, distribute and dissipate small amounts of water. However, problems arise when design or construction errors allow water into wall cavities at a rate higher than the structure can absorb and eliminate it. Eventually, the problem becomes acute for the areas difficult to access after construction or for the components costly to repair. Whenever the building design practices alone cannot eliminate the problem, fungal attack is inevitable and a safe and low toxicity wood preservative like borate ought to be a suitable choice for the vulnerable areas in the building envelope. Borates have several advantages including low cost, low mammalian and environmental toxicity, simplicity of application, effectiveness against insects and decay fungi (basidiomycetes) and high solubility in water (US Borax and Chemical Corp. 1986; Dickinson et al. 1989; Barnes et al. 1989; Murphy 1990; Greaves 1990; Williams 1990). Moreover, its 'less resistant to leaching' property can complement its effectiveness as it can diffuse into vulnerable areas when they are wet and penetrate refractory wood such as Douglas-fir heartwood (Fowlie et al. 1988; Rhatigan et al. 2002) which is not possible by pressure treatment.

The objective of this research was to investigate variables to understand the mobility and distribution of borate depending on wood species, direction of movement, borate formulation and wood moisture content (MC). The concentrations at different depths were tested by ANOVA to understand the significant differences between the variables at 5% level. Diffusion co-efficients were calculated to evaluate comparison and provide a quantitative measurement to understand the diffusion of borate in refractory species. Practical situations were also evaluated

by keeping the samples under wet exposure conditions; a mould test was performed to determine the efficacy of the formulations and necessity of co-biocides. Finally, recommendations are made regarding the volume of wood that can be effectively protected considering the distance moved by boron diffusion and volume treated in different directions.

1.2 Research context

1.2.1 Moisture and other issues related to mid-rise buildings

At present, there is a growing interest in using more timber products in the construction industry, especially for mid-rise buildings (4 to 10 storey) which is supported by the change of the building code in BC in 2009 (permission of maximum 6 storey timber building instead of previous 4 storey). To use more renewable and sustainable resources like wood and to meet the required structural performance for these structures, new products like cross-laminated timber (CLT) are being introduced in the building envelope. However, use of wood in tall buildings also raises concerns about several unknown issues such as fire protection, structural performance, appropriate joints and connections and potential for water ingress and accumulation problems resulting in decay and deterioration of structural assemblies.

To advance scientific knowledge in construction technologies and to face the aforementioned challenges a multi-disciplinary NSERC strategic research Network for Engineered Wood-Based Building Systems (NEWBuildS) has been established in collaboration with FPInnovations, the wood industry and design community. They are working on four themes (NEWBuildS website) regarding:

- 1) Material characterization and evaluation of structural performance
- 2) Development and evaluation of hybrid system for mid-rise construction
- 3) Analysis of fire performance, acoustic and vibration services in timber buildings
- 4) Improvement of durability and sustainability with the potential for enhanced products and design

As a part of the last theme, this research addresses the durability and environmental challenges through evaluation of the factors affecting borate distribution and the compatibility of borate with CLT structure. The long term goal of the research is to develop specifications for the pre-

treatment (factory treatment) or in-service treatment of CLT and other timber components of mid-rise structures with low toxicity, diffusible borate based preservatives.

1.2.2 Buildings with cross-laminated timber (CLT)

CLT is the one of the newest products introduced in the construction sector of Canada. It is also commonly known as solid wood panel (X-lam). It was invented in Austria in the mid 1990s and now it has gained popularity in Europe; there are several major CLT-based structures like tall buildings and bridges in the UK, Austria, Germany and Scandinavian countries.

CLT structures, like any other wood products, have the advantages of carbon storage, smaller carbon footprint and less green-house gas emission factors established by life cycle analysis. They also facilitate construction industries with faster completion, increased safety, less noise and less waste. CLT also provides its specialty in flexible long spans with better strength-to-weight ratio, higher degree of off-site prefabrication ability and capability to fit with any hybrid system, such as with light-wood frame, heavy timbers, steel or concrete. These factors make it a potential choice for the designers and engineers. This emerging successful system has been identified as a potential new product for commercial buildings by the forest products industry and the research and wood design communities in Canada.

1.2.3 Potential use of preservative in timber building components

Wood is considered to be a good building material because of its proven physical and mechanical properties, high strength-to-weight ratio, good insulating properties against heat, sound and electricity. It has the additional ability to absorb and dissipate a certain amount of moisture present in a building. The building environment is the main reason behind fungal attack. Whether it is condensation within wall systems and layers or leakage from rain or plumbing failures or transportation of moisture by end grain, wood can reach the moisture content needed for fungi to establish a viable mycelial mat to grow. However, Viitanen and Ritchkoff (1991) noted that water leakage caused about 90% of decay observed within buildings.

In most cases, the use of preservative by pressure treatment (e.g., CCA or ACQ) is not cost effective due to the low risk of building components getting wet and decayed. Borate diffusion through the building components can protect them by preventing or stopping infection by mould

and fungal decay. It also justifies the cost of protection to large dimension members such as glulam (glue laminated timber) or CLT which dry out slower than the other components. This can be effectively applied to critical components or locations in two ways: factory treatment, by which borate is used in the manufacturing process before installation to provide latent protection if the component becomes wet and ‘in-situ’ treatment where it is applied after observing moisture penetration rendering the component in service wet or vulnerable to moisture transfer from nearby components by diffusion. In this way, borate can play a significant role in protecting the parts vulnerable to moisture entrapment and movement after condensation or leaks occur. Figure 1 asserts that with proper maintenance (use of preservatives) progress of decay can be retarded for a significant number of years.

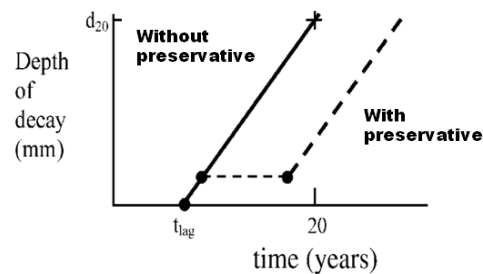


Fig. 1: Idealized concept of relationship between maintenance and progress of decay (modified from Leicester 2001).

1.3 Advantages of using borates

Borate preservatives have been used as an active ingredient in wood preservative systems for over 70 years (Cockcroft and Levy 1973; Barnes et al. 1989) and still continue to draw much interest and promote research (e.g. special session in IRG Conf. 2011). The advantages of borate preservatives over the other preservatives are:

- It is inexpensive and therefore cost effective.
- It is colorless and odorless; wood’s natural and aesthetic color can be visible even after the wood treatment.
- It is a proven fungicide and insecticide and has been used for a long time against termites, carpenter ants and beetles.
- It has corrosion inhibition property which is important for consideration in the use of nails and other metallic connectors.

- It has fire resistance and flame retardant properties when used at high loading; no other wood preservative possesses this quality.
- It is safe for human health and not more toxic than table salt.
- It can diffuse into vulnerable areas as soon as they are wet enough to initiate decay and it can even penetrate refractory wood like Douglas-fir heartwood.
- Only borate can be used either as a primary treatment or ‘in-situ’ treatment as per requirement.

1.4 Research gaps

After reviewing the literature, the following challenges or research gaps were identified where further investigation is necessary.

- 1) There is limited specific information on lower moisture content limits, borate distribution rate and extent and how these interact with formulation type and wood species (Blow and Summers 1985; Dirol 1988; Fowlie et al. 1988; Morrell et al. 1990; Rhatigan et al. 2002)
- 2) Inadequate quantitative information is available about the factors affecting distribution of borate in wood. The only extensive work was done by Ra et al. (2001) who evaluated southern pine which is highly permeable. So it is necessary to examine refractory wood species for the same purpose.
- 3) There is lack of information on the relationship between amount of chemical applied in different ways and the volume of wood effectively protected. This information is important to know to improve the efficacy as well as the cost-effectiveness of the preservative

1.5 Objectives

The following are the main objectives for this research -

- 1) To investigate variables affecting borate movement
- 2) To evaluate and develop suitable borate treatment procedures for timber components, joints and other vulnerable areas
- 3) To investigate the suitability of borate preservatives for cross-laminated timber (CLT)

CHAPTER 2 LITERATURE REVIEW

2.1 Factors affecting distribution of borate

Diffusion of boron in wood depends on many factors and some of them are critical for preservative treatment. For example, timbers with both sapwood and heartwood exposed complicate the problem of obtaining uniform penetrations and distributions (Vinden 1988). The concentration of boron inside the timber, obtained by diffusion, mainly depends on the following factors:

1. Thickness and density of timber and timber species
2. Grain direction of the treated surface
3. The moisture content of the timber
4. The concentration of the solution
5. The diffusion storage time and temperature
6. The thickness of film and solution retained on the wood surfaces
7. The diffusion coefficient

2.1.1 Moisture effects on borate diffusion

The effect of moisture content is very significant in diffusion process. The presence of a continuous medium and saturation condition of cells greatly affect the diffusion rate and extent. The rate of boron diffusion depends on the uniform distribution of water medium and the conformity that no air bubbles are present to restrict boron movement (Smith and Williams 1969).

The minimum moisture content (MC) required for a diffusion process depends on the type of salt used, but is generally near the fiber saturation of the cell wall, i.e. about 30% moisture content (Becker 1976). But, under practical conditions there is much doubt about the minimum moisture contents which should be investigated properly. The rate of boron diffusion and movement in wood treated with borate preservatives is also largely dependent on wood MC (Smith and Williams 1969; Morrell et al. 1990), since water is the medium for boron movement into wood. Ra et al (2001) asserted that the range of MC to occur diffusion is rather broad and the diffusion rates increase with the increase of MC; however, it is also notable that diffusion is a complex process and zones of gas or vapor break up the water phase and act as localized

barriers to diffusion in unsaturated wood. Hence, there occurs a decrease in the number of pathways available for diffusion per unit area. Diffusion is only uncomplicated in wood saturated with water i.e., water is present throughout the pore structure of the wood in all directions (Ra et al. 2001). At moisture contents exceeding 100 percent MC does not significantly influence the diffusion of boron observed in Scots pine and Sitka spruce (Smith and Williams 1969). Theoretically, diffusion will continue as long as there are differences in concentration between the solution at the surface of the wood and the chemical concentration in the free water within the wood.

2.1.2 Effects of grain direction on boron diffusion

Ra et al. (2001) observed in southern pine that the fastest rates of diffusion occur in the longitudinal direction followed by radial and tangential directions. The diffusion rate increased in the longitudinal direction rapidly with moisture content, while a slow increase was found in the radial direction at moisture content above 90% and not much in the tangential direction (within 70-110%). They also found that a repeated dip-treatment increased the rate of diffusion in the tangential direction, but only small differences were found in longitudinal and radial directions.

2.1.3 Effects of species

Dirol (1988) described that diffusion is easier in species with good impregnability (pine sapwood, poplar). Spruce being a refractory wood was always at high moisture content and diffusion of borate was possible, because it can 'keep' water easier than other species after a short drying period. So treatment of moist spruce with fused rod can be efficient.

In another study, Morris et al.(1996) asserted that pre-steaming is highly effective in some wood (western hemlock), but less effective in spruce-pine-fir or white pine and 30 minutes vacuum was better than 60 minutes or no vacuum. The addition of DDAC (didecyldimethyl ammonium chloride), a surfactant with pesticidal property to the borate solution provided improvement only for more refractory species (e.g. western hemlock, white spruce and Douglas-fir) and overall, most Canadian species can be borate treated to meet AWWA C31-95 standard if the right moisture content, treatment process and diffusion period were selected.

2.1.4 Effects of formulations

Glycerol is hygroscopic and affects both the shrinkage and surface hygroscopic properties of wood by holding water. Llic et al. (2003) found that surface checking can be reduced by 10% if glycerol is used in the early stages of drying. This property can affect both timber processing and behavior in service by reducing losses of timber through over-cutting green wood in finishing and making wood components less vulnerable to surface checking. With borate treatment, it can facilitate boron diffusion by holding water when the surface is drying out. Copper and borate are also used effectively in different formulations for many years. Studies (Amburgey 1990; Gray and Dickinson 1982) suggest that if copper is present in borate formulation it may increase the resistance against soft-rot fungi effectively.

2.2 Efficacy of borate against deterioration

The effectiveness of borates against termites has a long history of contradictions as contradictory results occur frequently (Nicholas et al. 1990). However, recent studies show promising results. For example, borates proved to be effective against termites by the 10 years sill plate test conducted by FPInnovations at different locations such as Japan, Hawaii and Ontario (Wang et al. 2007). All of the borate treated samples proved to give long term protection and a 3% BAE through treatment can be compared to CCA treated hem fir, although the addition of DDAC showed different levels of efficacy in field test and laboratory tests.

The threshold limit value of boron concentration varies widely between laboratory testing and field testing. Studies also show variable results for different kinds of pests. For example, BAE as high as 0.5% -1.2% was recommended for termites in some studies (Drysdale 1994; Lloyd 1995) whereas it is also recommended that low concentration (0.02% BAE) is sufficient for certain types of fungus (Manning et al. 1996). These widely varying results can occur from the lack of real standards in the field and wide variability of pests being tested (Peters 2006). However, 0.2% BAE is generally accepted as sufficient to combat against fungal decay, termites and wood boring insects (Drysdale 1994).

2.3 Application features of borates

Different techniques have been developed to accelerate the treatment by borate preservatives (Vinden et al. 1985; Barnes et al. 1990; Greaves 1990), but they have not gained general acceptance except in Australia because of the availability of a low cost and simple treatment such as dipping and diffusion (Ra et al. 2001). Surface treatments by borate preservatives are very effective in treating a variety of refractory species, because boron can diffuse into them whenever moisture content is above the fiber saturation point (Fowle et al. 1988; Lebow and Morrell 1989; Williams 1990; Williams and Amburgey 1987) and this cannot be achieved by other preservatives with pressure treatment. In another study, Highley and Ferge (1995) stated that spacing of boron rods (12×76 mm) in pine timbers of every 51mm across the grain and 305 mm along the grain would appear to be sufficient to protect the wood. On the other hand, red oak needs 25 mm across the grain and 152 mm along the grain while white oak requires 25 mm across the grain and 76mm along the grain.

According to Edlund et al. (1983) borate preservative can go through glue lines of carbamide as well as PVAC type. So engineered wood products can be treated by borate preservatives.

2.4 Leaching issue of borate

Borates are subjected to leaching where there is ground contact or frequent exposure to water and rain and hence they are not suitable for outdoor uses. Leaching is one of the major reasons why borate preservative does not gain much success in North America. However, studies (Harrow 1951, William and Michoff 1990, Williams 1991, Murphy et al. 1996, Manning 2004, Manning et al. 1996) show that even after extreme exposures, borate retentions do not drop below efficacious level. In all cases, the boron retention level reaches more than 0.1-0.2% BAE which is sufficient to protect from most kinds of biodegradation.

On the other hand, wood must remain totally wet across the cross section and there must be an external sink for borate to be leached completely (Manning 1996; Harrow 1959). However, this situation rarely occurs. In a review, Lloyd (1995) explains how the problems associated with leaching were initially overstated. He went on to say that the mechanism of leaching actually can help borate to penetrate deeper in the wood and if the initial loading is sufficiently high, there would remain enough boron to give adequate protection after leaching. Finally, Williams

(1996) has described leaching as a positive attribute by which borate (e.g., boron rod) can migrate to untreated wood if sufficient moisture from condensation is present.

CHAPTER 3 MATERIALS & METHODS

3.1 Sample preparation and analysis

Nominal 2 by 6 lumber of spruce (*Picea glauca or mariana*) and Douglas-fir (*Pseudotsuga menziesii*), also known as D-fir were bought from Home depot and Oliver Timber Co. respectively. Species were confirmed by microscopic analysis. Samples 36mm × 36mm × 36mm were cut from knot-free areas of the lumber. Samples were selected for uniform density by using those with an oven-dry weight basis of 18±3 gm per sample for spruce and 30±3 gm for D-fir. At first, samples were dipped into water and when they reached 30%, 50% and 70% overall moisture content (checked by weight), they were autoclaved at 120⁰ C for 40 minutes to result in a more uniform distribution of moisture. The samples were kept in re-sealable plastic bags and then inside airtight containers to get an even moisture distribution inside them and prevent them from surface drying. To minimize the moisture content gradient, they were kept for 4 weeks and representative samples were sliced and checked for moisture distribution through the thickness. When the moisture distribution was even, they were coated with water-proof silicone except on the side that would be treated. The sides where the formulations were to be applied were sealed with Tuck[®] tape, an impermeable sheathing tape (commonly used in the construction industry) to ensure no surface drying occurred. Then they were kept for 2-3 days for the silicone coating to be dried.

For the borate treatment, the samples were treated with glycerol borate and copper borate formulations (described in section 3.2) by brush-on application, the treated sides were again sealed with Tuck[®] tape to prevent moisture loss from the surface and kept for 7, 14 and 21 days to evaluate the change in boron distribution with time. After these periods, silicone seals and tapes were removed and samples were sliced to 6 mm consecutively by band-saw. The determination of boron concentration in these slices provided the distribution gradient inside the samples. Hence, the slices were ground to pass a 2mm mesh, boron extraction was done by hot water at 92⁰ C for 4 hours (Winters, ca. 1965) and boron content was determined by ICP analysis (AWPA A21). All data were generated by three replicates.

To check the statistical significance of the factors affecting borate diffusion, the data set of different concentrations for different depths acquired by ICP analysis were divided into 18 groups based on their depths and diffusion periods. Then, they were analyzed by ANOVA to see

the significance of different variables such as moisture content, grain direction, species and formulations at 5% level and the factors within the group were also checked by post-hoc (Tukey HSD and Scheffe) analysis. The ANOVA results given in Appendix B were generated by SPSS software.

3.2 Formulations:

Two types of high concentrated borate formulations were developed in the laboratory. They are glycerol borate (Gly bor) which is 40% Timbor[®] (DOT - Na₂B₈O₁₃.4H₂O, BORAX Inc.) in glycerol (glycerine) and copper borate (Cu bor) which is 40% Timbor[®] (DOT) and 10% copper monoethanolamine (NW 100C, OSMOSE Inc.) in aqueous solution. The use of glycerol can help to increase the hygroscopicity and reduce checking of wood. Using high concentration of borate may result in the deposition of some borate on the surface which could effectively protect the wood from mould and sapstain. For copper borate formulation, it was thought that the copper remaining on the surface might give protection to the surface as borate always tends to penetrate deeper in the wood. The borate bandage (Tuck[®] tape) can help to confine the high concentration of the borate in a specific area that needs protection. It can also help the borate to get proper surface sorption by preventing surface drying.

3.2.1 Glycerol Borate (Gly bor):

Gly bor contains 40% Timbor[®] (DOT) in 60% glycerine. At first, 120g glycerol (Calden Inc.) was heated to 60⁰ C. 80g Timbor was added slowly with continuous stirring. When all the Timbor was added and mixed properly around 70⁰ C, the heat source was removed, but stirring was continued. When the mixture was cooled down to room temperature the viscosity became too high and the formulation solidified. However, if the solution was heated to around 60⁰ C again, the viscosity reduced significantly (500 cps) and the fluid could be applied to the wood surface. The pH of the solution was 5.5 at 60⁰ C.

3.2.2 Copper Borate (Cu bor):

This formulation consists of 40% Timbor[®] (DOT), 10% Copper monoethanolamine (Cu Mea) and 50% water. At first, 20g Cu Mea was mixed with 100g of water at room temperature. The solution was heated to 60⁰ C and 80g Timbor[®] added in the solution slowly. Vigorous stirring was maintained throughout the process. When the temperature reached around 70⁰ C, all of the

Timbor[®] had been added to the solution and it was stirred well. The solution is immiscible under 40⁰ C. So when using this formulation, it has to be heated over 60⁰ C and stirred well. The viscosity is 20cps and the pH is 7 at 60⁰ C.

CHAPTER 4 RESULTS & DISCUSSIONS

4.1 Boron concentrations (% wt/wt BAE) along the diffusion depths:

The following tables (1,2,3 & 4) show the boron concentrations (% wt/wt boric acid equivalent, BAE) at different thickness within the treated samples determined by the ICP analysis to measure the diffusion gradients. The assay values in % BAE were calculated taking the basis of 0.5 g chemical/ 12.96 cm² surface area, i.e. 0.39 kg/m² basis. Concentration at different depths and diffusion periods were measured to understand the concentration profiles inside the samples. It was found that the surface layer concentrations dropped steadily with time as boron diffused deeper inside the samples. It can be noted here that the values were achieved only up to 21 days and there were still high concentrations of borate present near the treated part of the samples which could possibly diffuse deeper. So, further investigation is needed for longer diffusion times which can allow prediction of the time when the concentrations reach the threshold value throughout the samples.

Table 1: Boron concentration (% BAE) for spruce, glycerol borate treatment

		30% Moisture Content						50% Moisture Content						70% Moisture Content					
		Distance from treated surface, cm						Distance from treated surface, cm						Distance from treated surface, cm					
DIRECTIONS	Days	0.3	0.9	1.5	2.1	2.7	3.3	0.3	0.9	1.5	2.1	2.7	3.3	0.3	0.9	1.5	2.1	2.7	3.3
LONGITUDINAL	7	5.79	0.34	0.11	0.09	0.08	0.07	5.34	1.12	0.47	0.22	0.08	0.05	4.96	1.23	0.57	0.28	0.13	0.11
	14	3.21	0.60	0.11	0.09	0.08	0.07	2.44	1.21	0.55	0.40	0.25	0.17	1.84	1.20	0.76	0.50	0.27	0.18
	21	1.40	0.89	0.12	0.09	0.08	0.08	1.35	1.04	0.74	0.54	0.30	0.28	1.28	1.08	0.86	0.65	0.51	0.47
TANGENTIAL	7	2.97	0.07	0.05	0.03	0.01	0.01	2.86	0.16	0.06	0.05	0.05	0.05	2.82	0.27	0.16	0.09	0.09	0.06
	14	1.89	0.09	0.05	0.05	0.05	0.05	1.70	0.19	0.09	0.08	0.09	0.08	1.63	0.49	0.21	0.17	0.11	0.10
	21	1.29	0.27	0.08	0.06	0.06	0.05	1.18	0.38	0.11	0.10	0.09	0.09	1.08	0.59	0.30	0.17	0.15	0.12
RADIAL	7	3.55	0.21	0.14	0.08	0.07	0.06	3.32	0.32	0.19	0.14	0.10	0.10	3.13	0.50	0.20	0.13	0.13	0.07
	14	2.10	0.40	0.15	0.09	0.08	0.08	1.81	0.45	0.20	0.15	0.12	0.10	1.71	0.65	0.32	0.21	0.17	0.15
	21	1.31	0.54	0.16	0.08	0.08	0.08	1.23	0.70	0.20	0.17	0.13	0.11	1.14	0.74	0.5	0.35	0.27	0.22

Table 2: Boron concentration (% BAE) for spruce, copper borate treatment

		30% Moisture Content						50% Moisture Content						70% Moisture Content					
		Distance from treated surface, cm						Distance from treated surface, cm						Distance from treated surface, cm					
DIRECTIONS	Days	0.3	0.9	1.5	2.1	2.7	3.3	0.3	0.9	1.5	2.1	2.7	3.3	0.3	0.9	1.5	2.1	2.7	3.3
LONGITUDINAL	7	5.83	0.33	0.10	0.09	0.09	0.08	5.41	1.49	0.59	0.11	0.09	0.08	4.60	1.50	0.69	0.25	0.16	0.15
	14	3.04	0.63	0.19	0.10	0.09	0.09	2.53	1.54	0.62	0.25	0.14	0.12	2.51	1.17	0.74	0.40	0.23	0.14
	21	1.65	0.84	0.21	0.15	0.13	0.12	1.61	1.12	0.84	0.50	0.31	0.26	1.61	1.12	0.90	0.67	0.55	0.45
TANGENTIAL	7	3.38	0.19	0.08	0.03	0.02	0.01	3.22	0.19	0.09	0.08	0.08	0.08	2.46	0.42	0.16	0.08	0.07	0.07
	14	2.02	0.20	0.13	0.09	0.08	0.07	2.00	0.39	0.12	0.10	0.10	0.08	1.38	0.53	0.19	0.08	0.08	0.08
	21	1.28	0.25	0.18	0.12	0.13	0.12	1.19	0.54	0.14	0.12	0.10	0.1	1.04	0.57	0.28	0.18	0.15	0.15
RADIAL	7	3.65	0.23	0.15	0.09	0.07	0.05	3.57	0.32	0.14	0.08	0.08	0.08	2.96	0.44	0.23	0.14	0.08	0.08
	14	2.26	0.28	0.16	0.09	0.08	0.08	2.06	0.54	0.18	0.12	0.12	0.1	1.47	0.66	0.28	0.14	0.10	0.08
	21	1.51	0.29	0.19	0.13	0.13	0.13	1.12	0.62	0.25	0.15	0.13	0.11	1.08	0.73	0.54	0.43	0.36	0.21

Table 3: Boron concentration (% BAE) for D-fir, glycerol borate treatment

		30% Moisture Content						50% Moisture Content						70% Moisture Content					
		Distance from treated surface, cm						Distance from treated surface, cm						Distance from treated surface, cm					
DIRECTIONS	Days	0.3	0.9	1.5	2.1	2.7	3.3	0.3	0.9	1.5	2.1	2.7	3.3	0.3	0.9	1.5	2.1	2.7	3.3
LONGITUDINAL	7	4.80	0.65	0.08	0.05	0.04	0.05	3.96	1.19	0.5	0.17	0.11	0.09	3.47	1.15	0.57	0.34	0.19	0.13
	14	2.29	0.70	0.11	0.08	0.06	0.06	2.14	1.28	0.69	0.32	0.13	0.1	1.43	1.05	0.66	0.53	0.43	0.38
	21	1.54	0.77	0.23	0.13	0.12	0.11	1.51	1.24	0.87	0.45	0.20	0.18	1.05	0.91	0.73	0.61	0.53	0.50
TANGENTIAL	7	2.52	0.06	0.05	0.05	0.03	0.02	1.95	0.11	0.05	0.04	0.04	0.04	1.93	0.24	0.1	0.05	0.04	0.03
	14	1.89	0.10	0.08	0.07	0.06	0.06	1.15	0.21	0.09	0.09	0.09	0.08	1.10	0.34	0.10	0.08	0.08	0.07
	21	1.60	0.20	0.09	0.09	0.09	0.08	0.94	0.31	0.13	0.11	0.08	0.08	0.75	0.45	0.25	0.14	0.12	0.10
RADIAL	7	3.54	0.07	0.07	0.06	0.04	0.03	3.16	0.34	0.08	0.04	0.03	0.03	2.60	0.51	0.13	0.06	0.04	0.04
	14	2.18	0.13	0.08	0.08	0.06	0.06	1.92	0.53	0.17	0.08	0.07	0.06	1.18	0.58	0.30	0.21	0.14	0.10
	21	1.62	0.26	0.11	0.10	0.11	0.08	1.50	0.57	0.32	0.15	0.10	0.08	0.94	0.59	0.45	0.30	0.24	0.20

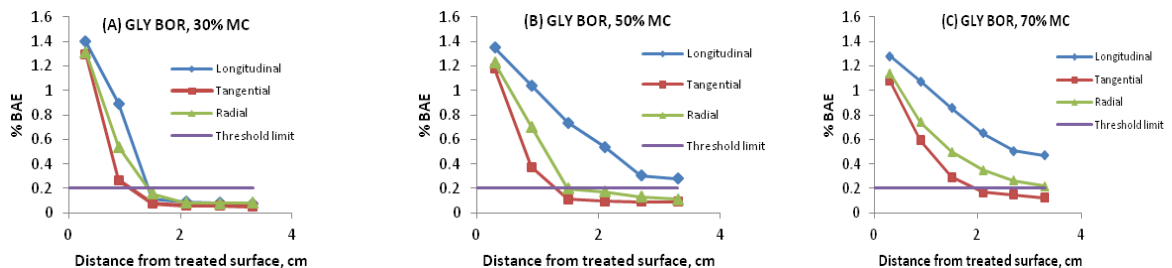
Table 4: Boron concentration (% BAE) for D-fir, copper borate treatment

DIRECTIONS	Days	30% Moisture Content						50% Moisture Content						70% Moisture Content					
		Distance from treated surface, cm						Distance from treated surface, cm						Distance from treated surface, cm					
		0.3	0.9	1.5	2.1	2.7	3.3	0.3	0.9	1.5	2.1	2.7	3.3	0.3	0.9	1.5	2.1	2.7	3.3
LONGITUDINAL	7	5.10	0.66	0.12	0.09	0.07	0.06	4.45	1.43	0.36	0.10	0.06	0.05	3.60	1.19	0.69	0.49	0.34	0.23
	14	2.83	0.99	0.14	0.09	0.08	0.07	2.48	1.55	0.70	0.28	0.14	0.11	1.69	1.24	0.86	0.70	0.57	0.55
	21	1.73	1.04	0.42	0.17	0.10	0.10	1.39	1.15	0.81	0.50	0.22	0.19	1.11	1.00	0.92	0.73	0.59	0.53
TANGENTIAL	7	3.30	0.08	0.06	0.05	0.03	0.03	2.78	0.11	0.07	0.06	0.06	0.06	2.48	0.28	0.12	0.07	0.05	0.05
	14	2.45	0.16	0.07	0.06	0.05	0.04	2.04	0.26	0.12	0.12	0.11	0.10	1.42	0.31	0.14	0.12	0.11	0.10
	21	1.43	0.22	0.08	0.08	0.08	0.07	1.26	0.33	0.13	0.11	0.12	0.11	1.00	0.38	0.24	0.17	0.11	0.10
RADIAL	7	4.39	0.17	0.09	0.07	0.06	0.05	3.88	0.34	0.10	0.06	0.05	0.05	3.16	0.34	0.20	0.13	0.12	0.12
	14	2.54	0.23	0.10	0.08	0.08	0.07	2.21	0.54	0.20	0.13	0.12	0.11	1.59	0.50	0.27	0.14	0.13	0.13
	21	1.47	0.39	0.14	0.12	0.10	0.10	1.31	0.62	0.29	0.16	0.14	0.11	1.28	0.66	0.43	0.28	0.19	0.17

4.2 Diffusion in different grain directions:

Figures 2 & 3 are the comparisons for the effect of grain directions on boron distribution after 21 days. The **threshold limit is considered as 0.2% BAE** which is shown in all plots. Only data for 21 days are given in the figures to show the maximum effect of diffusion for this research and the rest of the plots are given in Appendix A.

SPRUCE:



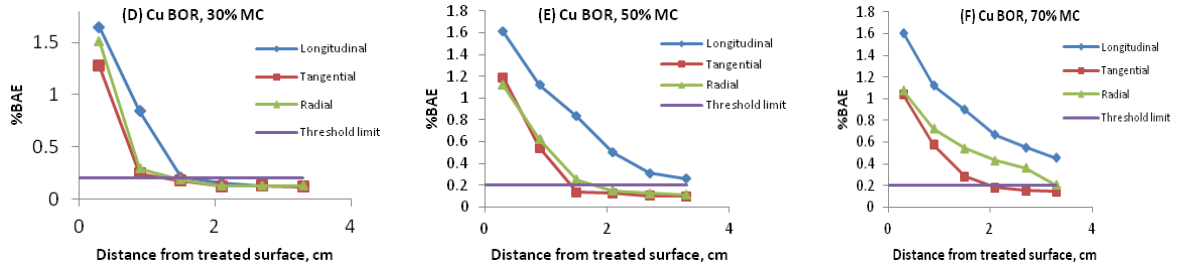


Fig. 2: Comparison of the effect of grain direction on boron diffusion in spruce for different moisture content and formulations (21 days).

D-FIR:

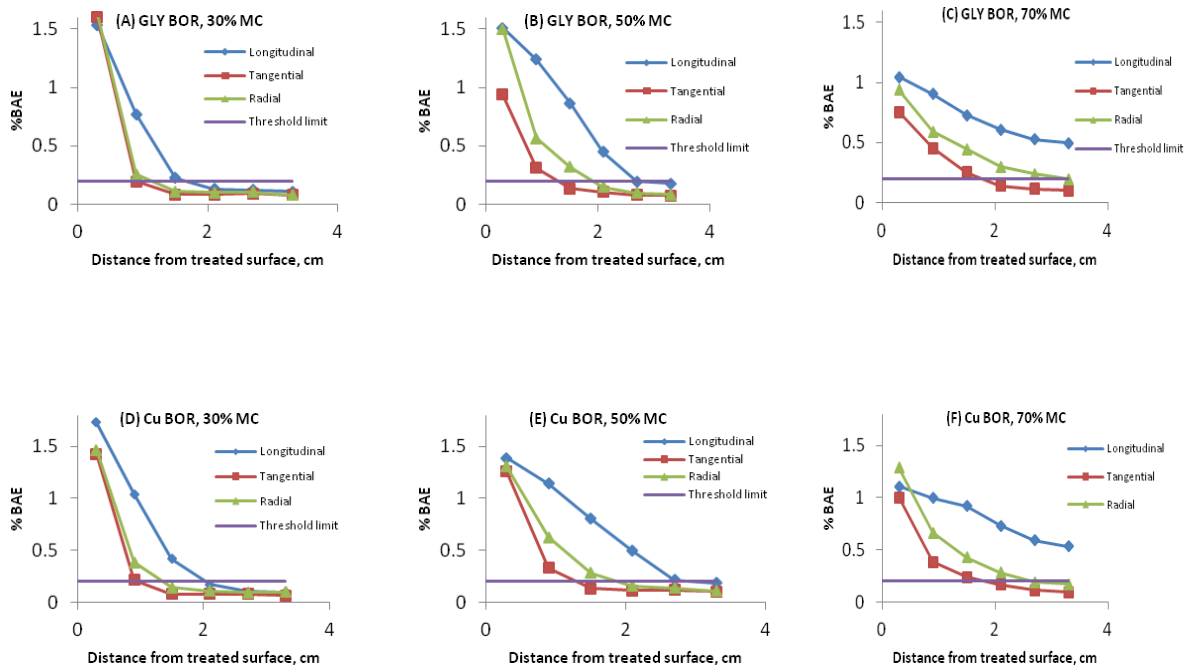


Fig. 3: Comparison of the effect of grain direction on boron diffusion in D-fir for different moisture content and formulations (21 days).

It was found that for both spruce and Douglas-fir at 30% MC the diffusion extent was low and not very different in the three directions (longitudinal, radial and tangential) (Figs. 2 & 3). Overall, longitudinal diffusion was higher than radial and tangential directions and radial diffusion was higher than tangential diffusion, but it was more prominent at higher moisture contents. At 30% MC, the threshold limit of 0.2% BAE could be achieved up to about 1.0 cm by both formulations in case of spruce after 21 days in longitudinal and radial directions (Fig. 2-A) whereas for D-fir it was 1.5 cm in the longitudinal direction by glycerol borate (Fig. 3-A)

and 2.0 cm for the copper borate formulation (Fig. 3-D). At 50% MC, the threshold limit could be achieved up to a greater depth by both formulations. Quantitatively, for spruce, it was about 1 cm in the tangential direction, 1.5 cm in the radial direction and 3 cm in the longitudinal direction (Figs. 2-B & E). For D-fir, in the same situation the depth was about 1 cm in the tangential direction, 2 cm in the radial direction and 2.5 cm in the longitudinal direction by both formulations (Figs. 3-B & E). At 70% MC, the trends remained the same as at 50% MC and more depth of threshold value was achievable in spruce than D-fir, especially in the radial direction (Figs. 2-C,F & 3- C,F). For both formulations, it was about 1.8 cm in the tangential direction, 3 cm in the radial direction and 4 cm in the longitudinal direction for spruce (Figs. 2- C & F) and about 1.5 cm in the tangential direction, 2.5 cm in the radial direction and 4 cm in the longitudinal direction for D-fir (Figs. 3- C & F). From the ANOVA test results (Table 5) it was observed that difference between borate concentration due to grain direction was statistically significant for all depths and diffusion periods.

Table 5: ANOVA test result for grain direction (√ = statistically significant difference at 5% level)

DIFFUSION DEPTH, cm	DIFFUSION PERIODS		
	7 DAYS	14 DAYS	21 DAYS
0.3	√	√	√
0.9	√	√	√
1.5	√	√	√
2.1	√	√	√
2.7	√	√	√
3.3	√	√	√

Greater boron penetration in the longitudinal direction was possible due to availability of free water in the cell lumens and the fact that material passes through fewer pits and cell walls. The primary means of longitudinal flow in softwood is through tracheids, lumens and bordered pit pairs; wood rays are not involved primarily (Bailey 1969; Cote & Krahmer 1962; Wardrop & Davies 1961). In the longitudinal direction, the area to transport water is double compared to that in radial and tangential directions which helps the higher diffusion (Becker 1976). However, for radial and tangential directions, the cell wall, bound water and rays play significant roles in boron diffusion. Theoretically, both the radial and tangential direction flows are restricted by both the thin cell walls in the earlywood cells and the thick parts in the

latewood cells (Ra et al 2001; Becker 1976). The radial direction flow gets advantage of the high permeability of the rays which results in higher penetration than the tangential flow (Becker 1976).

4.3 Effects of moisture content on diffusion:

The effects of three moisture contents (30%, 50% and 70%) on boron diffusion in 21 days are compared in Figures 4 & 5.

SPRUCE:

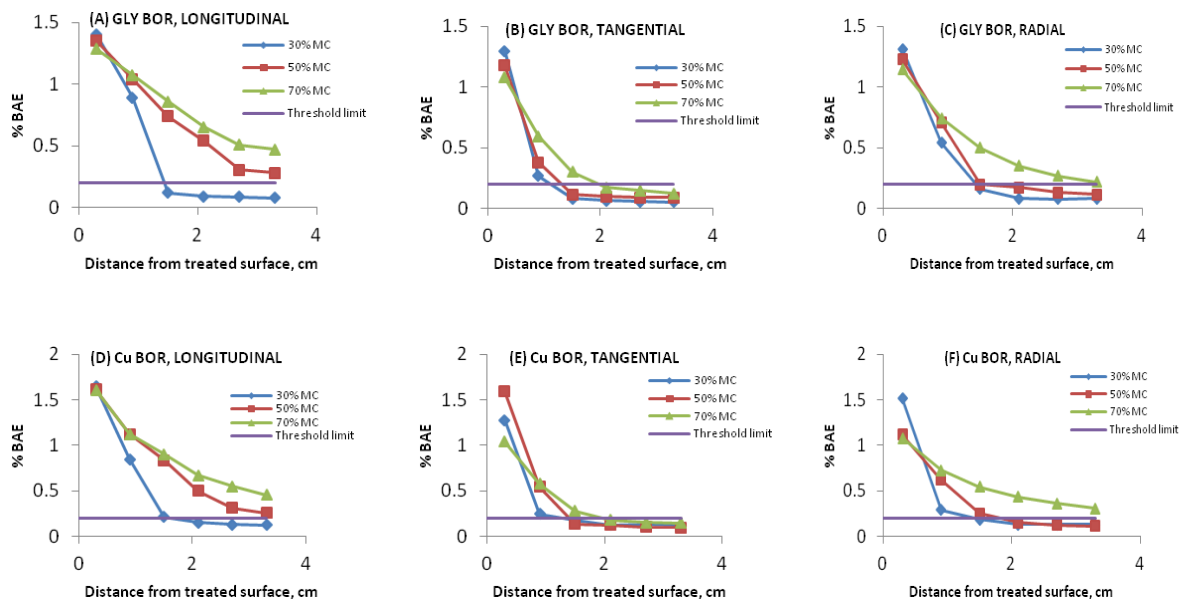
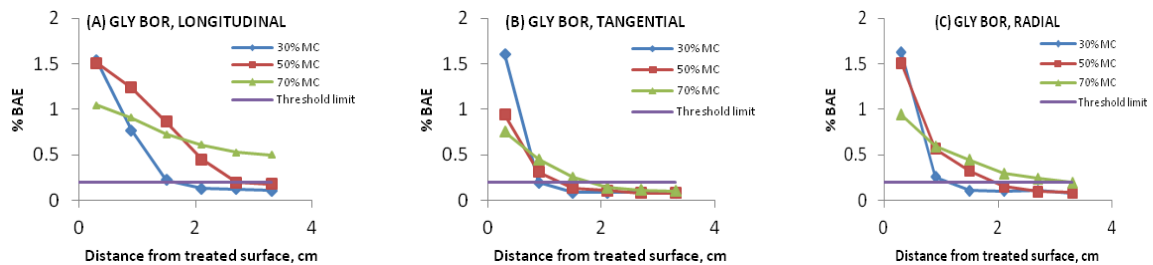


Fig. 4: Comparison of the effect of moisture content on boron diffusion in spruce for different grain directions and formulations (21 days).

D-FIR:



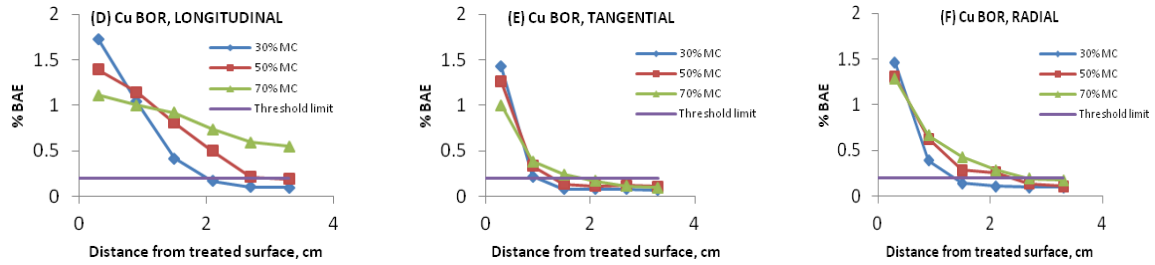


Fig. 5: Comparison of the effect of moisture content on boron diffusion in D-fir for different grain directions and formulations (21 days).

In this research, the use of Tuck[®] tape prevented the moisture on the surface from evaporation which facilitated the diffusion process. Also, there was a high concentration of borate present on the surface initially and as the concentration difference decreased, the boron diffusion rate decreased. So, there was good diffusion rate at the beginning due to greater concentration difference. The diffusion extent also depends on availability of continuous medium and hygroscopicity of borates (Becker 1976). The samples were maintained to a set moisture content and the highest moisture content ensured that the cell walls were saturated and free water was present, allowing greater extent of diffusion. It was observed that the borate penetration increased with higher moisture content regardless of species and formulations. Ra et al (2001) describes that at high moisture content, the limiting factor depends on bound water, not in free water which explains the reason of less moisture content effects on boron diffusion rate in radial and tangential directions. They went on to say that the moisture content of the sample surface plays a vital role to affect the initial uptake and the total amount of borate available for diffusion. From the Figures 4 & 5, in the longitudinal direction the diffusion was significant at 50% and 70% moisture content and their extent was close and much higher than that at 30% moisture content. For the tangential direction, the moisture content effect was not as prominent as for longitudinal and radial directions. Considering different species, the diffusion extent due to moisture content effect was more prominent in longitudinal and radial directions for spruce whereas for D-fir it was obvious only in the longitudinal direction. Overall, the difference between the extents was less obvious in D-fir than spruce, especially in radial and tangential directions. For spruce, boron penetrations at 50% and 70% MC were similar in the longitudinal direction; boron extent at 30% and 50% MC were not very different for tangential and radial directions for both formulations (Fig. 4). For D-fir, the differences of concentration among

different moisture content situations were only prominent in the longitudinal direction for both formulations (Fig. 5).

After 21 days, spruce showed slightly better depth of boron penetration at or above the threshold limit than D-fir. For example, for spruce and at 50% and 70% MCs both formulations showed boron concentration equal or above threshold limit up to ca. 3.0 and 4.0¹ cm respectively in the longitudinal direction (Figs. 4-A & D); for D-fir it showed up to about 2.5 cm and 4.0 cm for 50% and 70% respectively (Figs. 5-A & D). With both formulations, other notable depths were 3.5 cm and 2.5 cm at 70% MC in the radial direction for spruce and D-fir respectively. From the ANOVA test results (Table 6), it was observed that the effect of moisture content was statistically significant at all depths and diffusion periods.

Table 6: ANOVA test result for moisture content (√ = statistically significant difference at 5% level)

DIFFUSION DEPTH, cm	DIFFUSION PERIODS		
	7 DAYS	14 DAYS	21 DAYS
0.3	√	√	√
0.9	√	√	√
1.5	√	√	√
2.1	√	√	√
2.7	√	√	√
3.3	√	√	√

4.4 Effects of species:

The comparisons of the effects of two species (spruce and D-fir) on boron diffusion in 21 days are shown in Figures 6 & 7.

¹ In a few cases, the concentration values were significantly higher than the threshold limit value and effective penetration can be higher. For simplicity, they were considered as 4.0 cm in this study.

GLY BOR:

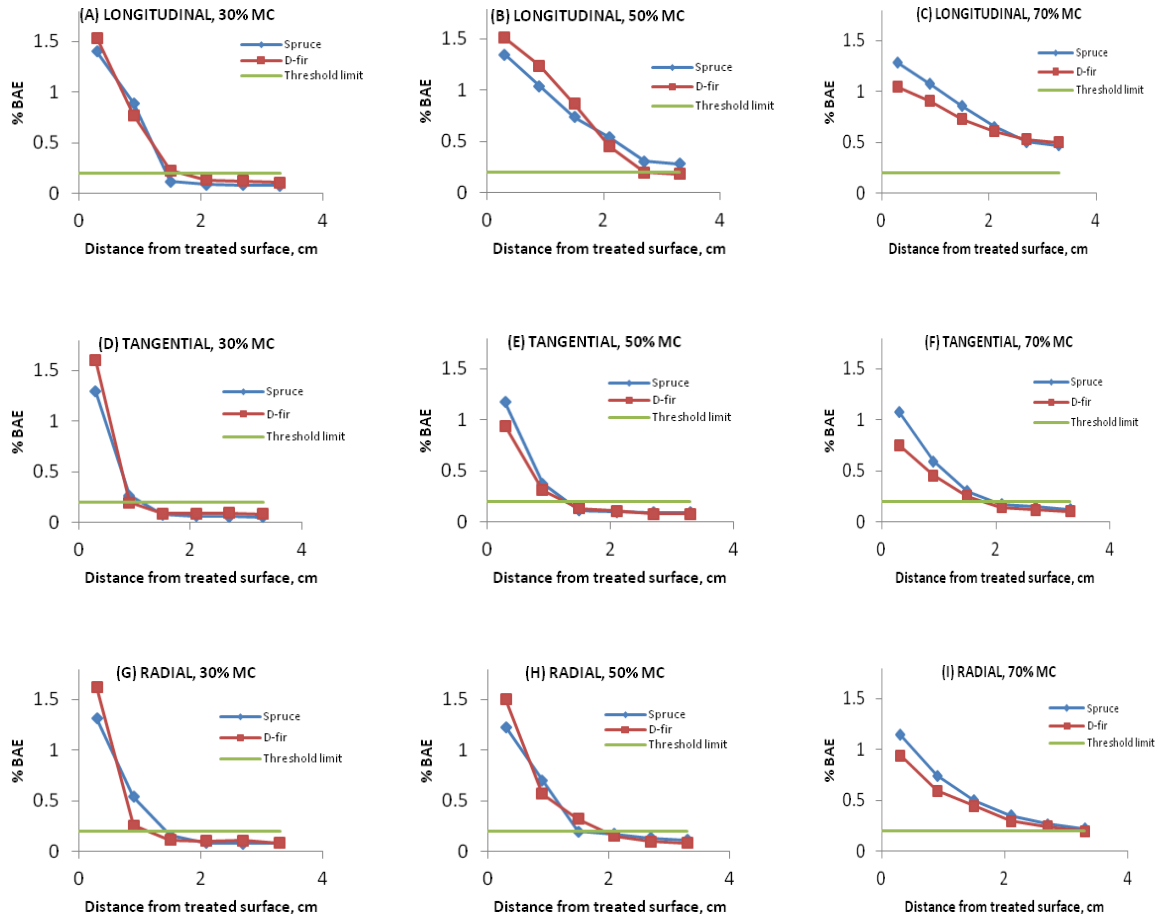
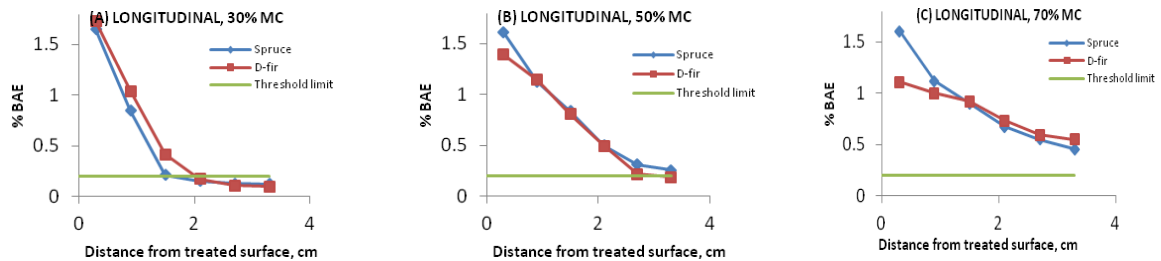


Fig. 6: Comparison of the effect of glycerol borate treated species on boron diffusion for different moisture content and grain directions (21 days).

Cu BOR:



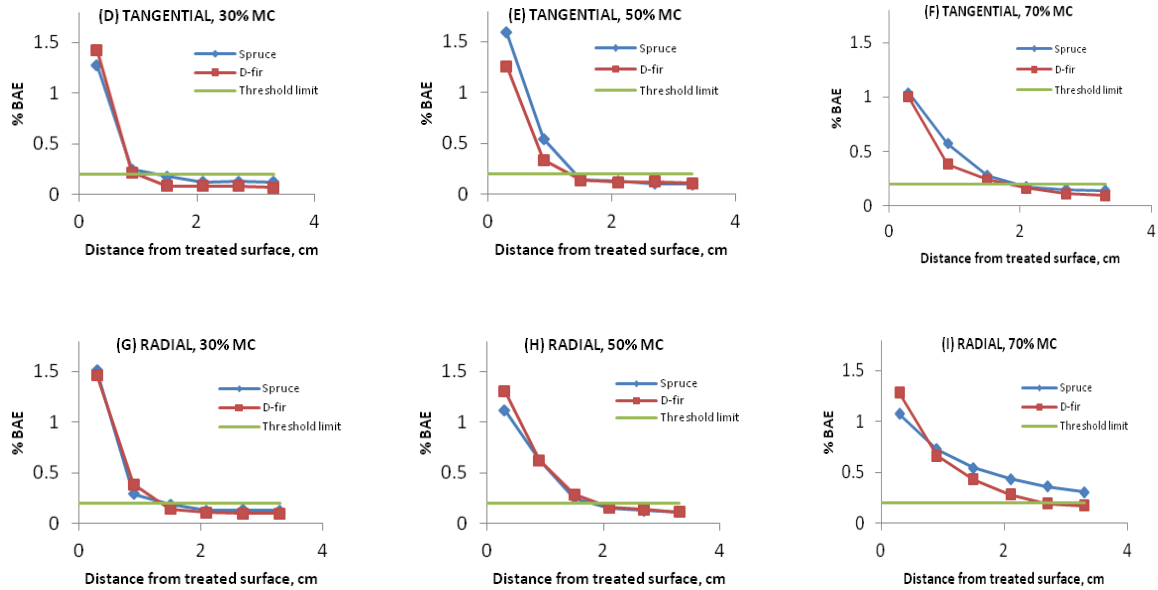


Fig. 7: Comparison of the effect of copper borate treated species on boron diffusion for different moisture content and grain directions (21 days).

It was observed that the difference in diffusion extent due to species was only prominent at high moisture content for both formulations (Figs. 6 & 7). For glycerol borate, spruce showed higher borate concentration than D-fir, especially near the treated surface and it was more obvious at 70% MC (Figs. 6-C,F & I); for copper borate it was similar, but the intensity was less than glycerol borate formulation (Figs. 7-C,F & I). For glycerol borate, notable protection (equal or above threshold value) could be achieved at 70% MC for both species up to ca. 4 cm and 3 cm in longitudinal and radial directions respectively (Figs. 6-C & I); for copper borate penetration up to about 4 cm was achieved only in the longitudinal direction (Fig. 7-C). However, for copper borate, the maximum achievable effective depths were up to about 2.5 cm for D-fir and 3.5 cm for spruce in the radial direction (Fig. 7-I). The other notable results were, at 50% MC and for both formulations, 2.5 cm for D-fir and 3.5 cm for spruce in longitudinal direction for both formulations (Figs. 6-B & 7-B). However, Table 7 indicates that the difference between two species is statistically significant only for 7 and 14 days. Hence, with a longer diffusion period, species became a less important factor for boron diffusion.

Table 7: ANOVA test result for species (√ = statistically significant difference at 5% level)

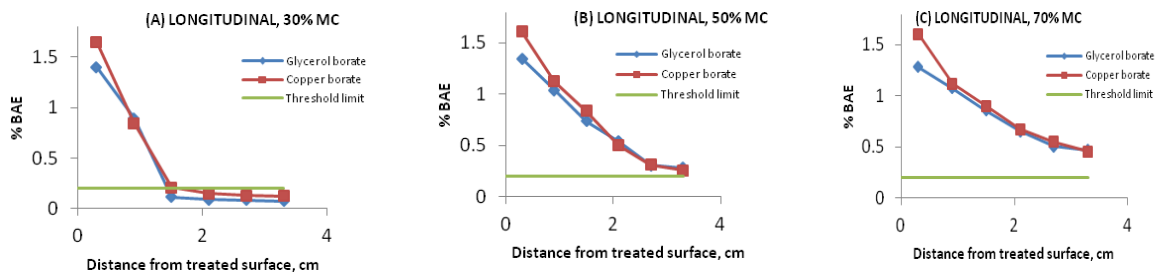
DIFFUSION DEPTH, cm	DIFFUSION PERIODS		
	7 DAYS	14 DAYS	21 DAYS
0.3	√	√	-
0.9	-	-	-
1.5	-	-	-
2.1	-	-	-
2.7	√	-	-
3.3	√	√	-

Overall, boron showed slightly better diffusion extent in spruce than D-fir. The density difference might be the main reason. The density of spruce is 450-460 kg/m³ which is lower than the density of D-fir (530 kg/m³). The lower density facilitates the diffusion as it exerts less restriction to the flow of the diffusing material. Williams (1991) asserted that there is an inverse relationship between the density of wood and the rate of diffusion, but inadequate literature is available. It can be an important issue while treating a mixture of species with variable densities. In general, spruce represents the CLT as it is the most used species in CLT production in Canada whereas D-fir can be related to glulam, another massive timber component used for beams and columns in the construction industry.

4.5 Effects of formulations:

The effects of different formulations (glycerol borate and copper borate) on boron diffusion in 21 days are shown in Figures 8 & 9.

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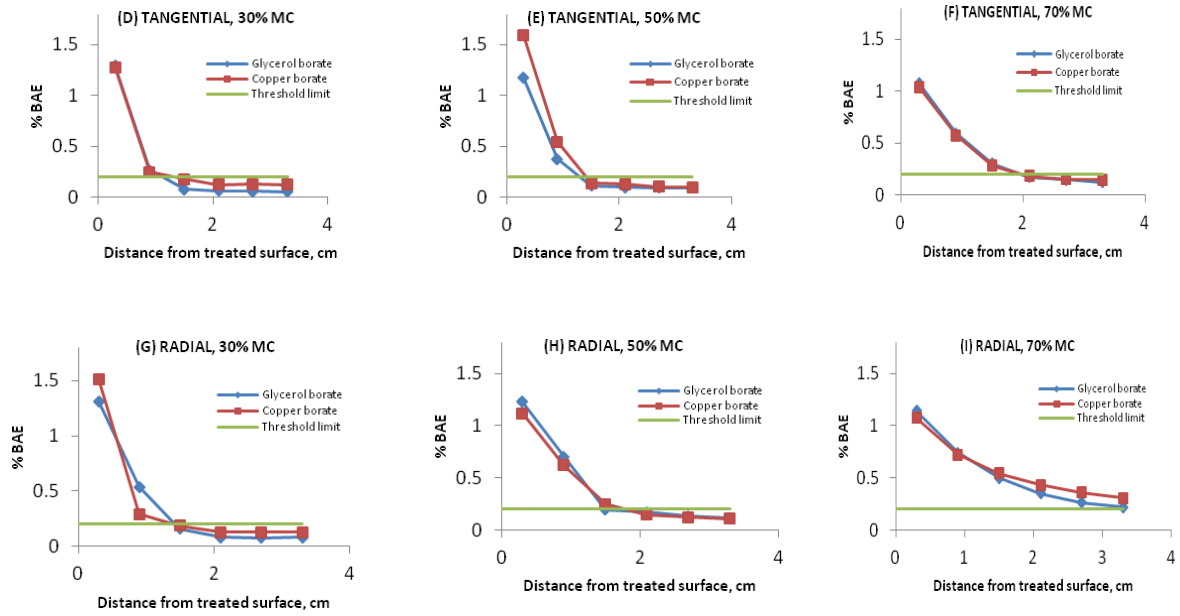
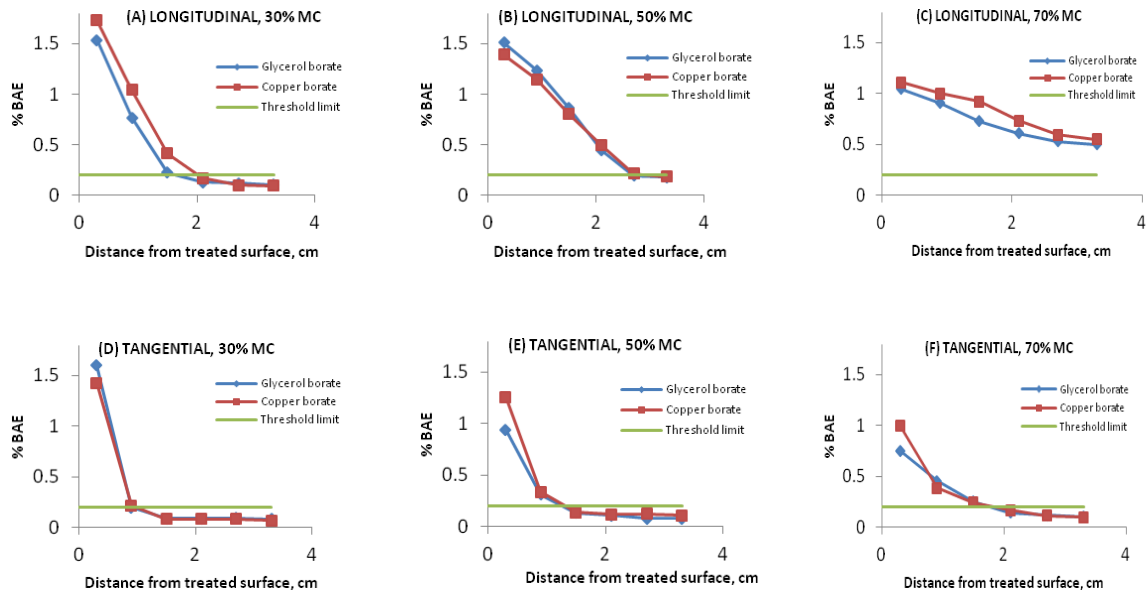


Fig. 8: Comparison of the effect of formulations on boron diffusion in spruce for different moisture content and grain directions (21 days).

D-FIR:



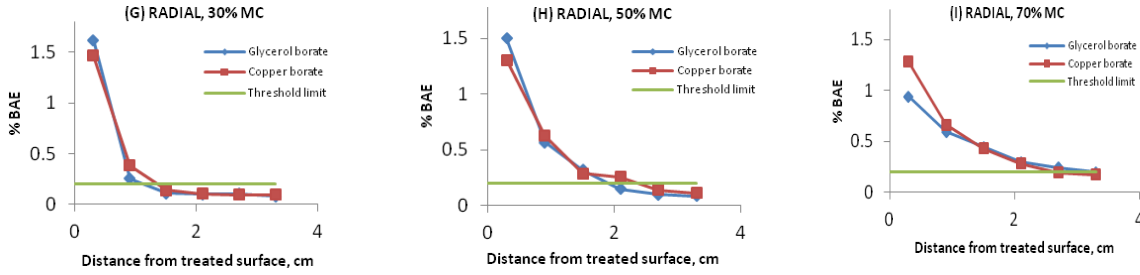


Fig. 9: Comparison of the effect of formulations on boron diffusion in D-fir for different moisture content and grain directions (21 days).

It was observed that copper borate showed slightly higher borate concentration compared to glycerol borate, especially near the treated surface (Figs. 8 & 9). This was more obvious for D-fir than spruce. For both species, it was more obvious in the longitudinal direction. The threshold value of 0.2% BAE could be achieved up to about 3.5 cm for spruce and 2.5 cm for D-fir both in longitudinal direction at 50% MC (Figs. 8-B & 9-B) and in radial direction at 70% MC (Figs. 8-I & 9-I). For both species and formulations, at 70% MC and in the longitudinal direction, the boron concentration of 0.2% BAE or more could be achieved up to about 4.0 cm (Figs. 8-C & 9-C). However, ANOVA results (Table 8) showed that differences in boron concentrations due to formulations were statistically significant mainly near the treated surface and at shorter diffusion period. Hence, it is not as prominent a factor as grain direction and moisture content in the boron diffusion process.

Table 8: ANOVA test result for formulation (\checkmark = statistically significant difference at 5% level)

DIFFUSION DEPTH, cm	DIFFUSIO PERIODS		
	7 DAYS	14 DAYS	21 DAYS
0.3	\checkmark	\checkmark	\checkmark
0.9	-	\checkmark	-
1.5	-	\checkmark	-
2.1	\checkmark	-	-
2.7	-	-	-
3.3	-	-	-

Overall, copper borate showed slightly higher boron concentrations than glycerol borate. This may be because copper borate contained 50% water which could help boron in the surface absorption and diffusion through the samples. Christensen (1951) found that the rate of diffusion depends on the nature of the solute and the rate is restricted if pathways are partly

blocked by chemical deposits. This can be possible for solutes that react with wood. However, borates do not react significantly with wood. So, non disassociated solutes such as borates diffuse rapidly as they move through the wood as electrically neutral molecules (Williams 1991). For the two formulations used in the research, glycerol is hygroscopic and more viscous than aqueous copper Mea solution which may result in keeping more boron on the surface.

CHAPTER 5 MEASUREMENT OF DIFFUSION COEFFICIENTS

5.1 Diffusion coefficients by Egner's solution

Quantitative measurements of the rate at which a diffusion process occurs can be expressed in terms of diffusion coefficients. The diffusion coefficient can be defined as the rate of transfer of the diffusing substance across a unit area of a section divided by the concentration gradient of that section with respect to diffusion depth (Ra et al. 2001). The mathematical theory of diffusion in isotropic substances is based on the hypothesis that the rate of transfer of diffusing substance through unit area of a section is proportional to the concentration gradient measured normal to the section, i.e.

$$F = -D (\partial C / \partial x) \dots \dots \dots (1)$$

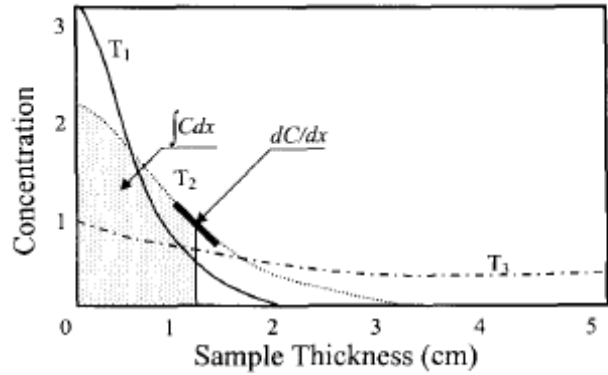
Where, F is the rate of transfer per unit area of section, C is the concentration of diffusing substance, x the space coordinate measured normal to the section and D is the constant diffusion coefficient. This is known as Fick's first law of diffusion. But as the concentration is changed with time (t), diffusion of salt can be best described by Fick's second law of one-dimensional diffusion,

$$\frac{\partial C}{\partial t} = D \frac{\partial^2 C}{\partial x^2} \dots \dots \dots (2)$$

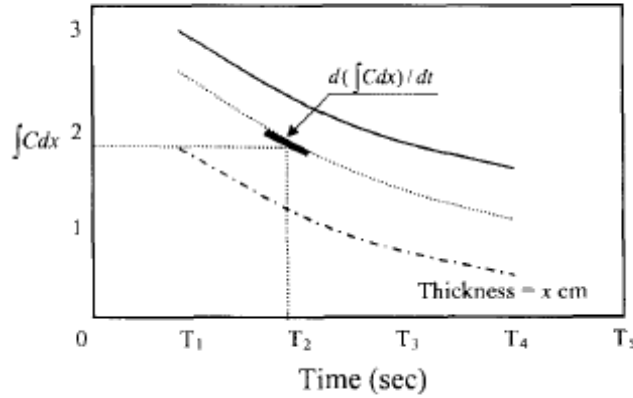
Fick's first and second laws assume a constant diffusion coefficient independent of concentration change, but in the case of wood, if it is heated, impregnated with liquids, or dried, steady-state flow changes and the diffusion coefficient will vary. Then the solution to Fick's second law is possible by Egner's method (Skaar 1954, 1958) where diffusion coefficients are obtained graphically, numerically and analytically. However, the most important criteria in this method is that accurate information is needed about the boron distribution in a wood specimen at various times which is hard to achieve and the equation itself is very sensitive to experimental errors (Ra et al. 2001). The Egner's equation can be written as

$$D = \frac{\frac{\partial(\int_0^x C dx)}{\partial t}}{\frac{\partial C}{\partial x}} \dots \dots \dots (3)$$

A graphical illustration of the derivative and integration terms of the above mentioned equation is shown in Fig. 10. The derivative terms ($\partial C/\partial x$) and integration terms ($\int C dx$) are to be calculated at various sample thicknesses (Fig. 10-A) and the calculated integration terms at various distances are graphed against time to calculate $\frac{\partial(\int_0^x C dx)}{\partial t}$ (Fig. 10-B).



(A) Denominator in Egner's solution



(B) Numerator in Egner's solution

Fig. 10: Graphical description of the terms in Egner's solution - (A) Concentration (% BAE) vs. diffusion depths (cm) curves, (B) The calculated integration terms at various distances are graphed against time (second) (Source: Ra et al. 2001)

At first, the datasets for 7, 14 and 21 days were plotted and integrated to 0.5, 1.0 and 1.5 cm consecutively. Then the integrals were plotted against time (seconds) and differentiated for 7, 14 and 21 days. These values make the numerators of the equation. From the concentration vs. distance graph, derivatives for 0.5, 1.0 and 1.5 cm were acquired and they are the denominators in the equation. The ratio of the numerator and denominator gave the diffusion co-efficient from

the equation (5). To analyze all the data SciDAViS (Scientific Data Analysis and Visualization), a computer program for interactive scientific graphing and data analysis, was used and $r^2 \geq 0.96$ was achieved for all the curves in both cases. Table 9 shows the diffusion coefficients measured at different depths and diffusion periods by Egner's solution.

TABLE 9: DIFFUSION COEFFICIENTS BY EGNER'S SOLUTION

DIRECTION	MOISTURE CONTENT %	DAYS	GLY BOR, SPRUCE			Cu BOR, SPRUCE			GLY BOR, D-FIR			Cu BOR, D-FIR		
			DISTANCE, cm			DISTANCE, cm			DISTANCE, cm			DISTANCE, cm		
			0.5	1	1.5	0.5	1	1.5	0.5	1	1.5	0.5	1	1.5
			$\times 10^{-7} \text{ cm}^2/\text{s}$			$\times 10^{-7} \text{ cm}^2/\text{s}$			$\times 10^{-7} \text{ cm}^2/\text{s}$			$\times 10^{-7} \text{ cm}^2/\text{s}$		
LONGITUDINAL	30	7	4	45	-	4	57	-	4	27	-	3	22	-
		14	5	24	91	5	25	80	6	18	49	4	13	37
		21	1	19	31	3	7	11	9	20	36	3	6	7
	50	7	5	21	76	5	17	46	4	14	46	4	12	26
		14	9	22	42	8	17	26	7	13	24	7	14	20
		21	7	7	12	3	4	5	18	21	31	10	20	27
	70	7	5	20	76	5	16	66	6	22	62	5	18	44
		14	12	21	39	7	18	25	15	28	39	15	29	39
		21	25	32	42	2	9	12	30	45	54	48	63	21

TANGENTIAL	30	7	3	-	-	3	-	-	3	-	-	3	-	-
		14	3	67	-	3	20	-	2	56	-	3	23	-
		21	3	7	-	2	9	-	1	2	-	2	10	-
	50	7	3	35	-	4	38	-	1	17	-	2	-	-
		14	3	18	-	3	13	-	1	5	38	2	18	-
		21	2	2	9	4	10	26	1	2	4	4	16	-

		7	3	32	-	4	19	-	3	23	-	3	27	-
	70	14	4	13	37	4	11	22	4	12	34	3	16	34
		21	1	4	3	1	1	2	1	1	6	1	1	3

RADIAL	30	7	3	-	-	3	-	-	4	-	-	3	-	-
		14	4	17	-	3	25	-	3	61	-	4	34	-
		21	2	6	6	2	9	13	1	2	4	3	11	13
	50	7	4	40	-	3	30	-	3	17	-	3	22	-
		14	4	16	-	4	13	-	3	9	-	4	14	-
		21	3	3	6	5	13	21	4	7	12	3	8	8
	70	7	4	20	-	4	22	-	5	22	80	4	43	-
		14	5	10	18	6	13	24	8	16	16	5	14	34
		21	1	2	6	9	16	22	10	13	22	7	12	12

OBSERVATIONS:

1. It was found that diffusion rates were usually higher for the early periods of diffusion. This may be because there were higher concentration differences and the diffusion co-efficient is dependent on concentration gradient (difference).
2. It was observed that diffusion rate increased with depth up to 1.5 cm, but after 1.5 cm from the treated surface significant diffusion coefficient could not be determined due to the limitation of the method. This was because after 1.5 cm the curves became nearly flat (the concentration difference becomes very low) and as the diffusion coefficient, D was to be determined by the slopes of the curves, they gave insignificant values.
3. Maximum erroneous results occurred in the tangential direction. This may occur because of the variable earlywood and latewood portions with diffusion rates, constant cell wall thickness and more variance of internal conditions in that direction.

4. As there was great variance of boron concentration at the surface with time due to the presence of borate bandage and the concentrations within the samples differ substantially with time and location, the erroneous results are not unexpected. The equation itself by nature is too sensitive for experimental accuracy (Ra et al. 2001). There can be future research about more suitable methods to determine diffusion coefficients in an anisotropic material like wood. In this dissertation, to provide a better comparison between different factors, the average diffusion coefficients were calculated below. These considered the total diffused substance deposited initially on the treated surface. However, it shows the overall constant diffusion coefficients and does not consider the variance for different diffusion points inside the sample.

5.2 Average diffusion coefficients:

Fick's second law can be solved as eq. 4 which is applicable to a plane source in an infinite volume when all the diffusing substance is concentrated initially on the plane. The main assumption is the constant diffusion coefficient. The equation for the diffused substance initially deposited at time $t = 0$ and diffuses throughout the infinite medium ($-\infty < x < \infty$), from a point $x = 0$ (Ra et al. 2001)

$$C = \frac{M}{2\sqrt{\pi Dt}} e^{-x^2/4Dt} \dots \dots \dots (4)$$

where, M = total mass of the diffusing substance; D = diffusion coefficient; C = the concentration of diffusion substance (W/W %); and x = the distance penetrated in the direction of diffusion. In that case, half the diffusing substance moves in the direction of positive x and the other half in the opposite direction (Crank 1975).

For the study described here, borate was applied at one surface only and could only diffuse in one direction. This situation is analogous to diffusion through a membrane with an impermeable boundary. The solution for diffusion through the semi-infinite medium with an impermeable boundary at $x = 0$ can be obtained by considering the solution for negative x to be reflected in the plane $x = 0$, superposed on the original distribution in the region $x > 0$ and all the diffusion occurs in the direction of positive x [Eq. (5)].

$$C = \frac{M}{\sqrt{\pi Dt}} e^{-x^2/4Dt} \dots \dots \dots (5)$$

Since, the original solution was symmetrical, reflection at $x = 0$ simply means the adding of two solutions of the diffusion equation. As the equation (4) is linear, the sum of the two solutions in opposite directions gives a solution as the equation (5) and it also shows that the total amount of diffusing substance remains constant at M and equal to the amount originally deposited in the plane $x = 0$. Also, here the condition for an impermeable boundary, $dC/dx = 0$, $x = 0$ is satisfied (Crank 1975; Ra et al. 2001, 2002). Overall, the procedure of reflection and superposition is mathematically sound for semi-infinite medium, because the solution is the mathematical condition for zero flow across a boundary.

Average diffusion coefficients can be calculated by using equation (5). At $x = 0$ the variation of boron concentration with time can be expressed as

$$D = \frac{M^2}{C^2 \pi t} \dots\dots\dots(6)$$

This can give the simple way to measure the average diffusion coefficient where $M = \int_0^x C dx$ and C is calculated at $x = 0$ from the concentration vs. distance graphs plotted by SciDAVis software. The main limitation of this process is that it gives a constant diffusion coefficient which cannot explain the internal diffusion condition of the wood properly.

TABLE 10 : AVERAGE DIFFUSION COEFFICIENTS

DIRECTION	MOISTURE CONTENT %	DAYS	GLY BOR, SPRUCE	CU BOR, SPRUCE	GLY BOR, D-FIR	CU BOR, D-FIR
			Average Diffusion Coefficients			
			$\times 10^{-7} \text{ cm}^2/\text{s}$	$\times 10^{-7} \text{ cm}^2/\text{s}$	$\times 10^{-7} \text{ cm}^2/\text{s}$	$\times 10^{-7} \text{ cm}^2/\text{s}$
LONGITUDINAL	30	7	0.15	0.14	0.39	0.38
		14	0.37	0.38	0.58	0.71
		21	1.15	1.27	1.01	1.35
	50	7	0.81	1.34	1.47	1.32
		14	2.05	2.47	2.30	2.44

		21	3.36	4.54	3.09	4.97
	70	7	1.10	1.85	1.83	2.25
		14	3.79	2.34	4.40	9.44
		21	6.97	7.50	10.50	9.55

TANGENTIAL	30	7	0.09	0.17	0.02	0.05
		14	0.05	0.16	0.03	0.08
		21	0.25	0.30	0.10	0.15
	50	7	0.13	0.13	0.13	0.06
		14	0.13	0.35	0.30	0.19
		21	0.52	0.64	0.62	0.41
	70	7	0.24	0.61	0.40	0.36
		14	0.77	1.08	0.76	0.44
		21	1.73	2.04	2.50	1.08

RADIAL	30	7	0.18	0.21	0.03	0.09
		14	0.35	0.21	0.05	0.12
		21	0.82	0.27	0.16	0.41
	50	7	0.23	0.26	0.30	0.23
		14	0.55	0.59	0.58	0.51
		21	1.32	1.68	0.89	1.33
	70	7	0.51	0.57	0.66	0.33
		14	1.28	1.53	2.30	0.93
		21	3.67	6.12	4.40	2.07

OBSERVATIONS:

1. This method can be a good way to present and compare the diffusion rates in the wood samples, because in this research initially there was high concentration at the surface and due to borate bandage system the concentration at the surface played an important role in the diffusion process by acting as a continuous source of boron.
2. The diffusion coefficient increased with moisture content. On average, the diffusion coefficients at 50% moisture content were 3 times higher than those at 30% moisture content and diffusion coefficients at 70% moisture content were 8 times higher than those at 30% moisture content.
3. On average for 21 days' data, the longitudinal diffusion coefficients were 7 times higher than tangential diffusion coefficients and 3 times higher than radial diffusion coefficients.
4. For the same species, the copper borate formulation showed higher diffusion coefficients than the glycerol borate formulation.
5. For the same formulations, spruce showed higher diffusion coefficients than Douglas-fir in tangential and radial directions, but in the longitudinal direction, higher diffusion coefficients were observed in D-fir samples than in spruce samples in most cases. The diffusion coefficients can show different effects than those observed from the diffusion concentration values, because diffusion coefficient is determined by rate of diffusion which is dependent on the concentration gradient (difference).

5.3 Potential volumes protected by borate preservatives

Table 11 contains the approximate volumes that can be protected by applying 0.39 kg/m² of preservative by topical application. These values were calculated by considering the maximum distances along the diffusion path that contains boron equal to or more than the threshold value of 0.2% wt/wt BAE for 3.6 × 3.6 × 3.6 cm samples. It demands further investigation to acquire predicted values for larger samples by determining penetration up to threshold value for longer diffusion times.

TABLE 11: APPROXIMATE VOLUMES OF WOOD PROTECTED BY DIFFERENT BORATE FORMULATIONS (FOR 3.6 × 3.6× 3.6 cm SAMPLES)

DIRECTIONS	MOISTURE CONTENT, %	DAYS	GLY BOR, SPRUCE	Cu BOR, SPRUCE	GLY BOR, D-FIR	Cu BOR, D-FIR
			APPROX. VOLUME (cm ³)PROTECTED BY APPLYING 0.39 Kg/m ² OF CHEMICALS			
LONGITUDINAL	30	7	15	15	17	18
		14	17	19	17	19
		21	19	20	21	26
	50	7	27	26	25	23
		14	38	30	31	32
		21	47	47	35	36
	70	7	30	30	32	43
		14	40	38	49	49
		21	52	52	52	52

TANGENTIAL	30	7	11	12	10	11
		14	12	12	11	11
		21	14	16	12	12
	50	7	12	12	10	11
		14	12	17	12	14
		21	17	18	15	17
	70	7	13	18	13	14
		14	19	19	15	15
		21	25	25	22	22

RADIAL	30	7	13	14	11	12
		14	15	15	11	12
		21	18	18	14	17
	50	7	17	16	15	15
		14	19	19	19	19
		21	19	23	24	24
	70	7	19	19	17	19
		14	27	23	17	23
		21	43	45	41	34

It was found that maximum ca. 52 cm³ could be protected at 70% moisture content in the longitudinal direction by all formulations and species. Maximum ca. 45 cm³ in the radial direction and 25 cm³ in the tangential directions could be protected by treating spruce with copper borate formulation. Copper borate treated spruce samples showed maximum protection in most cases, especially in radial and tangential directions, but in the longitudinal direction copper borate treated D-fir samples showed better results in many cases. It can be noted here that these values were achieved considering the boron diffusion only after 21 days. However, there was still a high concentration of borate present near the treated part of the samples which could possibly diffuse deeper if the samples were kept in the same moisture content condition for longer periods of time.

CHAPTER 6 ANCILLARY PROPERTIES OF BORATE TREATMENT

6.1 Protection from mould growth

6.1.1 Introduction

Moulds can grow anywhere especially on organic substances if suitable temperature, oxygen, source of nutrients (e.g., sugar, starch and protein) and water or moisture are available. All these elements can possibly be found in the building environment. Hence, mould growth on the building envelope components is probable if there is excessive relative humidity inside the house or moisture accumulated due to leaks or condensation and it is undiscovered or unaddressed for reasonable time. Moulds continue their reproduction by creating and spreading spores which can be seen only under microscope. When these spores land on damp indoor spots they start to grow and digest nutrients. This results in unsightly colors from the surface spore deposits, mildew odor and the potential for health problems from airborne spores of mould metabolites.

Since mould requires water to survive, the presence of mould is an indication of a moisture problem in the building envelope. This moisture problem can occur due to many reasons including excessive humidity. The changes in building practices after the 1970s' energy crisis have resulted in tightly sealed wall systems with inadequate ventilation which can be a major cause of potential moisture problem (EPA report 2008). In addition to internal leakage and condensation, bad construction practice or design and delayed maintenance can also initiate moisture problems suitable for mould growth.

The presence of mould in the building can affect the occupants in two major ways:

1. **Health issues:** Most of the moulds cause adverse effects on occupants' health. They can produce allergens in the spores which can cause allergic reactions and even asthma attacks (EPA report 2008). Moreover, some of them create metabolic VOC (volatile organic compounds) by-products such as alcohols, ketones, esters and hydrocarbons which are harmful to human health. These are called mycotoxins.

2. **Aesthetic issues:** Although moulds do not affect the strength of structural components as decay fungi do, the unaesthetic look of the wood components can cause substantial devaluation of the property and the health risks involved can deter buyers. The presence of mould can also increase the cost of maintenance from the frequent painting necessary to cover up the mouldy parts. Moulds can also sustain high moisture content which leads to decay of wood.

In this research, preliminary tests were done to understand the mould development criteria in exposure situations, the effectiveness of borate preservatives against mould. Also, a standard test (AWPA E24-06) was conducted to check the efficacy of adding didecyldimethyl ammonium chloride (DDAC), a co-biocide much used in wood industries to the borate.

6.1.2 The simulation of an extreme exposure situation for mould growth:

To simulate a worst case situation for CLT in construction and to see its effects on spruce lumber, an environmental chamber was pre-conditioned to 28-30⁰ C and 90-95% R.H. Wooden joints with angle brackets were placed so mould development could be monitored and effects of borate as pre-treatment or factory treatment compared. Some joints were kept untreated and some joints were partially treated, i.e., one part was treated with either glycerol borate or copper borate, but the opposite face was not treated. From visual observations, it was found that mould started to grow in the untreated parts of the joints in only two weeks, but no significant mould was visible in the treated part up to 8 weeks for both formulations (Fig. 11). After 15 weeks, significant mould had developed also in the treated parts. However, it was found from visual observation that the density of mould in glycerol treated parts was higher than that for copper borate treated parts (Fig. 12).





Fig. 11: Treated (left) & untreated (right) parts of joints after 15 weeks(Top - copper borate treated & Bottom - glycerol borate treated).



Fig. 12: Comparison between glycerol borate treated(left) and copper borate treated (right) joints (Top- 8 weeks, Bottom- 15 weeks)

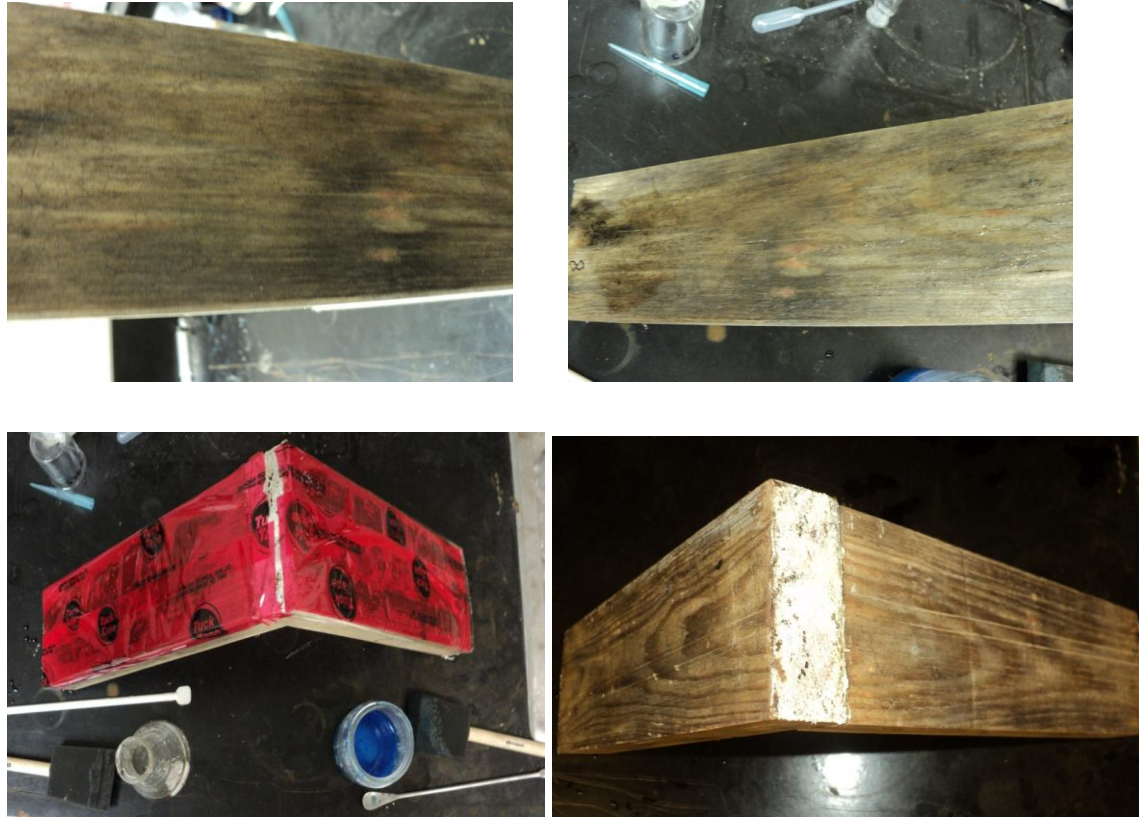


Fig. 13: Mouldy parts (top) of an untreated joint treated by Glycerol borate and copper borate and enclosed by Tuck® tape (bottom left) and after keeping 12 weeks of exposure condition (bottom right)

To see the remedial effect of the borate formulations, an untreated joint (kept in moist condition for 3 months) with two extremely mouldy parts was selected for borate treatment (Fig. 13). One part was treated with copper borate and the other with glycerol borate. They were enclosed by Tuck® tape, a sheathing tape to ensure the highly concentrated borate solutions remained in place and no surface drying occurred. After 4 weeks, the tape was removed and from visual observation no change of color or decrease in mouldy condition was observed. It was kept for 8 more weeks and there was no spread of mould found from visual observation. However, all the tests in this section are preliminary in nature and they demand further research.

6.1.3 Efficacy test with borate formulations and didecyldimethyl ammonium chloride (DDAC)

6.1.3.1 Materials and method

Didecyldimethyl ammonium chloride (DDAC) is mainly a surfactant which has pesticidal properties and hence is used in the wood industry to protect lumber from mould and sapstain. It is used with borate to protect borate treated wood from mould during long diffusion periods as borate is not as much effective against staining fungi, especially mould as it is against decay fungi (Morris et al. 1996; Byrne 1990). It is also believed to increase penetration of borate by decreasing the surface tension and improving the wettability of wood (Morris et al. 1996). To see the efficacy of borate formulations along with the addition of DDAC against mould, spruce samples (6 replicates each- 3 by 4 in.) were treated with glycerol borate, copper borate and copper borate with 0.5% DDAC and placed in the mould chamber according to standard AWPA E24-06. The chamber is maintained at 32⁰ C and was prepared with 4 kinds of mould spores:

1. *Aureobasidium pullulans* MAD MDX-18
2. *Aspergillus brasiliensis* FPL - W56
3. *Penicillium spp.* UT002
4. *Alternaria spp.* UT003

The vulnerable condition inside the mould chamber was confirmed by the medium to heavy mould growth on untreated southern pine samples (05 replicates) after 2 weeks of exposure. After keeping the spruce samples inside the mould chamber for 2, 4, 6 and 8 weeks, they were checked for mould intensity and weighed to determine the moisture content. The samples were visually rated for the extent and intensity of mould growth as per the scales in the table 12.

Table 12: Scales used for rating the mould growth on the samples exposed in the mould chamber

Rating	Description
0	No visible growth
1	Mould covering up to 10% of surfaces providing growth is not so intense or colored as to obscure the sample color over more than 5% of surfaces
2	Mould covering between 10% and 30% of surfaces providing growth is not so intense

	or colored as to obscure the sample color or more than 10% of surfaces
3	Mould covering between 30% and 70% of surfaces providing growth is not so intense or colored as to obscure the sample color on more than 30% of surfaces
4	Mould on greater than 70% of surfaces providing growth is not so intense or colored as to obscure the sample color or more than 70% of surfaces
5	Mould on 100% of surfaces or with less than 100% coverage and with intense or colored growth obscuring greater than 70% of the sample color

6.1.3.2 Results and discussion

Table 13: Mould test results with spruce samples

Formulations	After 2 weeks			After 4 weeks			After 6 weeks			After 8 weeks		
	AVG. MC	AVG. Rating	SD	AVG MC	AVG. Rating	SD	AVG MC	AVG. Rating	SD	AVG MC	AVG. Rating	SD
GLY BOR	41%	0.67	0.516	38 %	1.67	0.516	38%	3.0	0.894	37%	3.67	0.516
Cu BOR	42%	0.17	0.408	41%	0.83	0.408	42%	1.83	0.753	39%	2.17	0.753
Cu BOR/DDAC	40%	0.33	0.516	49%	0.83	0.753	44%	1.67	0.516	45%	1.83	0.408
CONTROL (spruce)	33%	0.33	0.516	36%	0.67	0.516	45%	1.5	0.548	44%	2.67	0.516

After 2 weeks, no significant mould was visible except in the samples treated with glycerol borate which could be rated 1 for mould. The moisture content reached around 40% for all treated samples while the untreated samples were at a lower MC. After 4 weeks, mould had spread on all samples. The samples treated with copper borate and copper borate plus DDAC showed rating 1 of mould whereas the samples treated with glycerol borate had been substantially mouldy up to rating 3. The mould situation of glycerol borate treated samples deteriorated (up to rating 4) after 6 and 8 weeks. This may have happened due to the hygroscopic nature of glycerol borate which can absorb moisture from the air, but this was not be reflected in the average moisture content because of the frequent evaporation and

condensation inside the chamber. The untreated samples (spruce) did not take up much moisture at the beginning. However, when the moisture content reached more than 40% mould grew very rapidly (up to rating 5) on the replicates. After 6 weeks, the copper borate treated and copper borate plus DDAC treated samples had similar ratings, but after 8 weeks, the copper borate plus DDAC treated samples showed better rating (2 compared to 3) than copper borate treated samples and also other samples. Overall, spruce is a species which is hard to absorb water even in moist condition, but if water can somehow penetrate, it would be difficult to dry. So, it was observed that as the water does not stay longer on the surface of spruce samples, the chance of getting mould is less until the surface moisture content rises to around 40% and addition of DDAC with copper borate formulation showed better protection against mould. Fig. 14 shows the comparison between different formulations at different time periods.

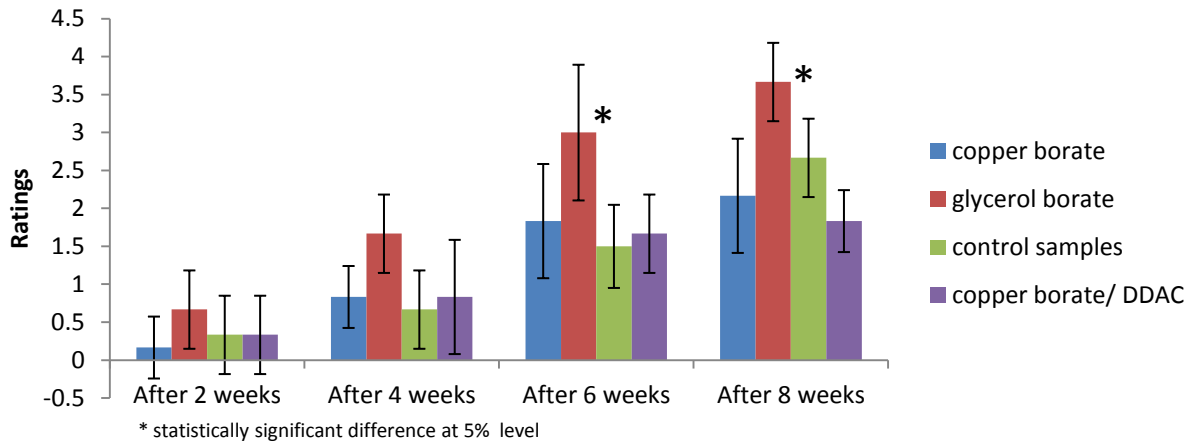


Fig. 14: Comparison of mould test performance of different formulations

It can be noted here that spruce samples were used (standard method suggests sapwood of pine species which is vulnerable to mould growth) to see the efficacy of the formulations as well as the resistance of spruce towards mould growth. This may be the reason why only glycerol borate treated samples showed significant difference at 5% level. After 8 weeks, the average performance of copper borate plus DDAC formulation was better than the others, but continuing for longer time of exposure might prove the efficacy clearly.

6.2 Fire resistant property

For building envelope components, the fire retardant property of borate can be very useful. In a fire test experiment by ASTM D3806-98(2011):2-foot tunnel method lumbars treated by glycerol borate and copper borate showed good results (less distance and depth travelled by fire)

as in Fig. 15 . The glycerol borate showed better results than copper borate. This may be because the viscous nature of glycerol borate formulation can keep more boron on the surface which helps to increase the fire retardant property. This experiment was preliminary in nature and further investigation is needed.

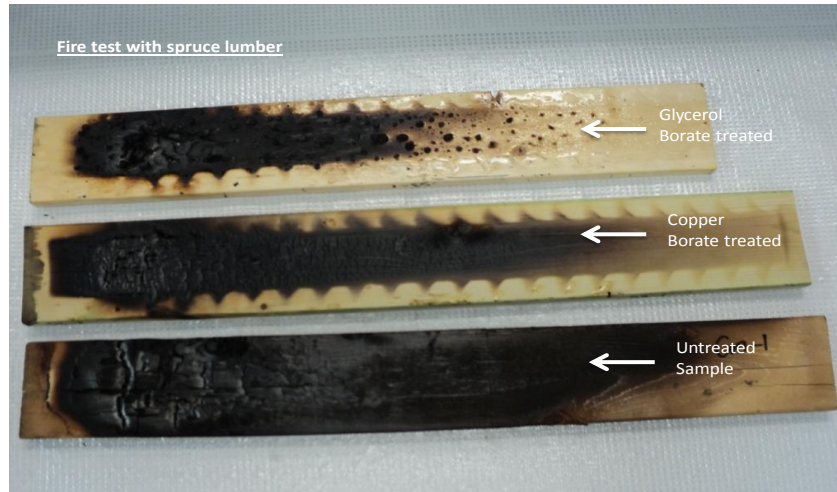


Fig. 15: Fire test ASTM D3806-98(2011) result for different formulations compared to untreated sample

CHAPTER 7 SUMMARY, RECOMMENDATIONS & FUTURE RESEARCH

7.1 Summary

In this dissertation, factors affecting diffusion of borate were evaluated to understand their influence over the treatment process which can facilitate the borate treatment process at a commercial level. The concentration data within the samples were assayed and evaluated by analysis of variance (ANOVA) to understand the significance of the variables. Diffusion coefficients were measured to evaluate the comparison between variables quantitatively. Finally, mould tests were done to compare the efficacy of the formulations as well as to evaluate the significance of addition of DDAC to one formulation. The following results are notable.

- The grain direction and moisture content affect boron distribution more significantly than species and formulation.
- Longitudinal diffusion is much higher than radial and tangential diffusion and diffusion in the radial direction is higher than in the tangential direction. This is more obvious at higher moisture content. Effective (more than 0.2% BAE) diffusion can be achieved at maximum distance (ca. 4 cm) in the longitudinal direction with both formulations at 70% moisture content (in 21 days). After this time there was still a high concentration of borate present near the surface and over a longer test period, borate should diffuse much further at levels high enough to stop or prevent decay.
- Boron diffusion extent was always higher with higher wood moisture content. However, this was more obvious in the longitudinal direction and least obvious in the tangential direction.
- Spruce samples showed slightly better diffusion results than Douglas-fir samples.
- Between the two formulations, copper borate had better diffusion extent than glycerol borate especially in the longitudinal direction and at higher moisture content.
- From the measured average diffusion co-efficients it was observed that average longitudinal diffusion co-efficients were about 7 times higher than tangential diffusion co-efficients and about 3 times higher than radial diffusion co-efficients. Average diffusion co-efficients at 70% moisture content and 50% moisture content respectively were around 8 times and 3 times higher than at 30% moisture content. Copper borate had

higher diffusion coefficients than glycerol borate, but for species no significant differences were seen.

- Under high humidity exposure situations, the formulations show some resistance to mould growth and copper borate showed better results than glycerol borate.
- The fire retardant property of borate was observed in a preliminary test in which glycerol borate performing better than copper borate treatment.
- From the average results in the standard mould test, use of copper borate formulation shows marginal potential to prevent mould growth compared to untreated samples and addition of DDAC with the formulation may give better protection whereas spruce itself has some mould resistance as it is hard to get wet.

7.2 Recommendations

The following recommendations are made as the outcomes of this research.

- Lumber can be treated as pretreatment to protect building envelope components to protect them from decay and pests, because if there is water accumulation inside the structure, borate can easily diffuse into the vulnerable area due to concentration differences. This is also true for engineered wood products with solid lumber such as CLT. In that case, treating wood before fabrication can minimize the uncertainty of boron distribution and can give more control to the manufacturer to protect vulnerable components.
- 'In situ' treatment can be a good idea, but the high concentration should be kept on the surface by bandage or the component should be wet enough to start boron diffusion.
- To protect from mould, addition of a co-biocide such as DDAC appears to be more effective.
- From measured potential volumes which can be protected by applying 0.39 kg/m^2 of preservative by topical application, it was observed that copper borate treated spruce samples showed better protection than glycerol borate in most cases. It is also noteworthy that high concentrations of borate were still present near the treated surfaces of the samples after 21 days of diffusion which could possibly diffuse deeper if the samples were kept in the same moisture content condition for a longer period of time.

- The use of glycerol can increase the hygroscopicity and is reported to improve the check resistant property of wood, but it may also draw moisture into the surface which can cause mould to grow easily.
- The borate bandage can help the high concentration of the borate to remain in a specific area that needs protection. It can also help the borate to get proper surface sorption by preventing surface drying of the lumber.

7.3 Future research

The following points are noted through the research which may be subject to further investigation

- The special feature of water in wood is its equilibrium condition with its environment which changes frequently. The understanding of this dynamic is an important way to understand the moisture ingress, its movement and initiation of decay because of this. The vulnerability of wood initiates with water accumulation while the diffusion of boron depends on the availability of the moisture inside the wood. Hence, more knowledge about this subject matter can facilitate the proper application of borates to the vulnerable areas of the building envelope.
- In general, sapwood is more permeable than heartwood because of its lower pit aspiration and less incrustation with extractives (Siau 1984). However, there is inadequate literature present about the effects of sapwood and heartwood on the diffusion process which can contribute to significant information about the chance of biodegradation as well as the proper application of diffusible wood preservatives.
- Temperature effects of borate treatment on CLT panel should be investigated, because studies (Ra et al 2001; Warren et al 1968; Smith and Williams 1969) show that treatment temperature is a very important factor in case of diffusible preservatives.
- In a preliminary experiment, the CLT panel in the exposure condition (28-30⁰ C and 90-95% R.H) shows its vulnerability to mould growth. When exposed in the horizontal position, mould grows mainly near the edge joints of the panel especially on the parts more exposed; in the vertical position, mould tends to grow near the end grain of the panel (Fig. 16). Further research can confirm these results which can elucidate the vulnerable points in the CLT panel for mould growth. Currently, there is more research going on in our lab regarding CLT panels treated by different application methods such

as pressure treatment, topical application, boron rod, etc and using different commercial products such as boracol[®] and boron rod which can acquire recommendations about the effective treatment of CLT structures by borate preservatives to protect them from biodegradation.



Fig. 16: CLT panel after exposed to extreme condition in two position: left - horizontal, right- vertical.

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Appendices

APPENDIX A: ALL BORATE DISTRIBUTION DATA (GRAPHS) WITH STANDARD DEVIATIONS

At 30% Moisture Content

Fig. 1.1 Glycerol borate treated samples

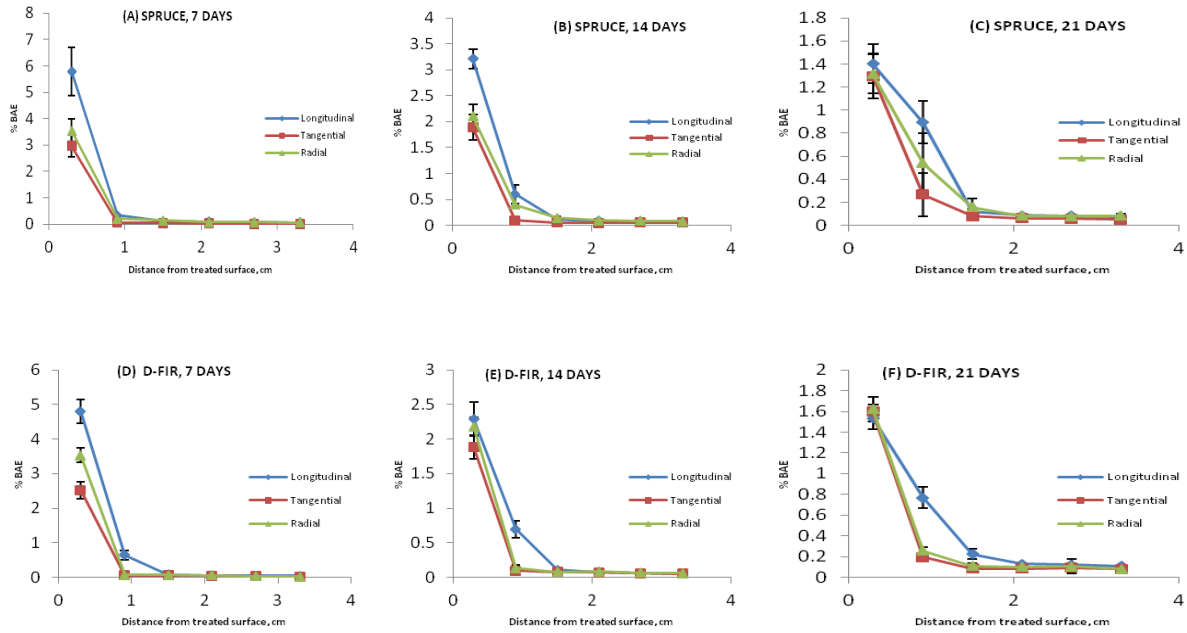
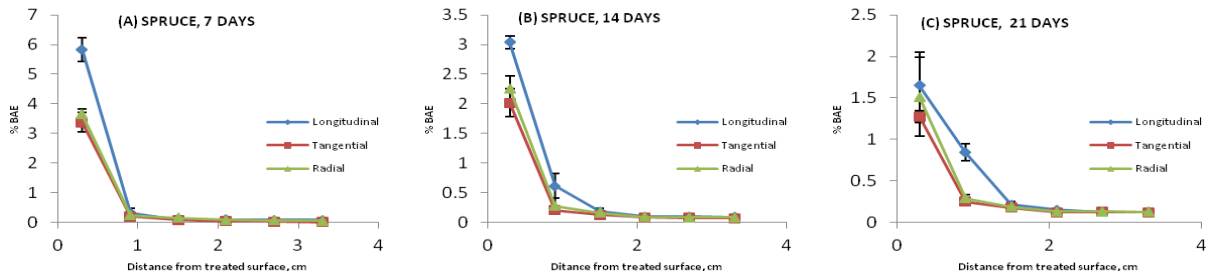
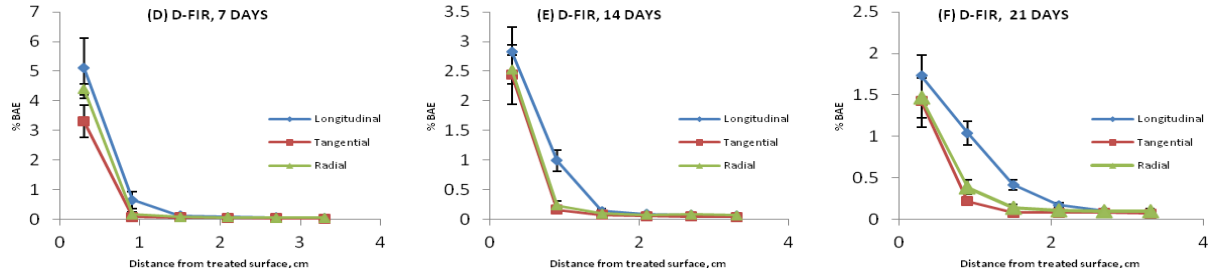


Fig. 1.2 Copper borate treated samples





At 50% Moisture Content

Fig. 1.3 Glycerol borate treated samples

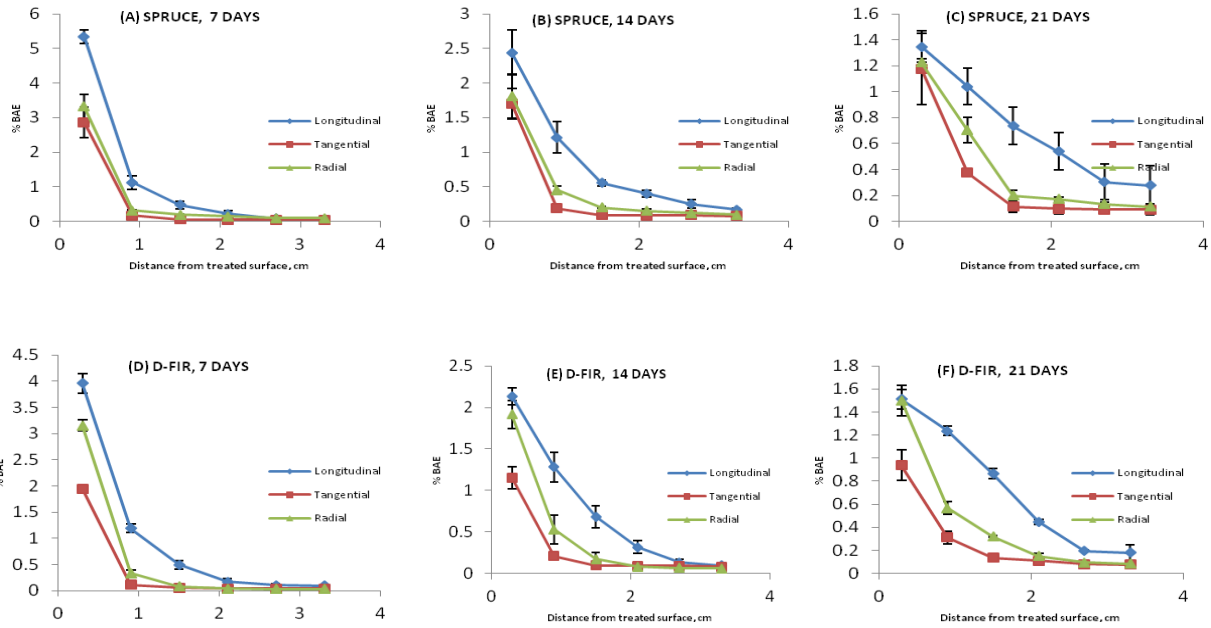
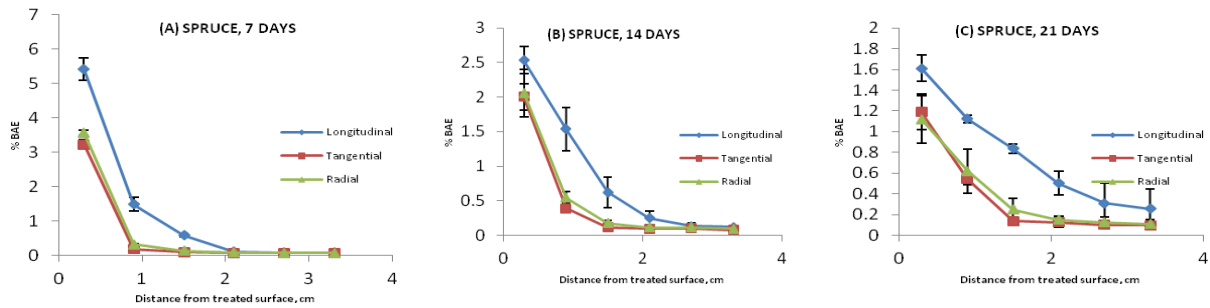
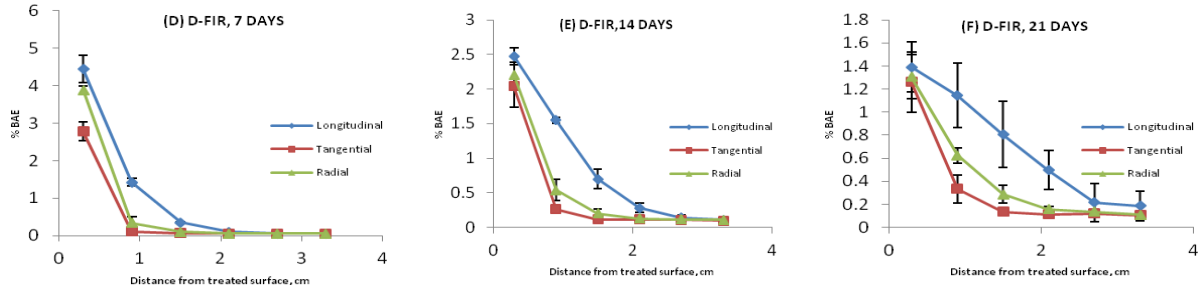


Fig. 1.4 Copper borate treated samples





At 70% Moisture Content

Fig. 1.5 Glycerol borate treated samples

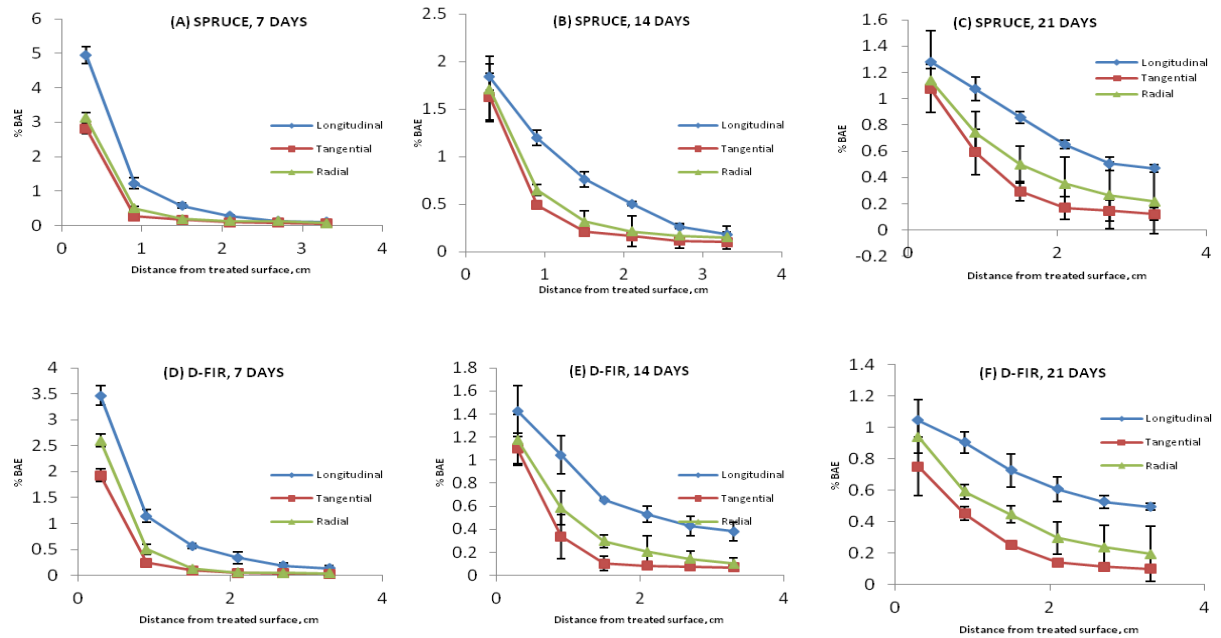
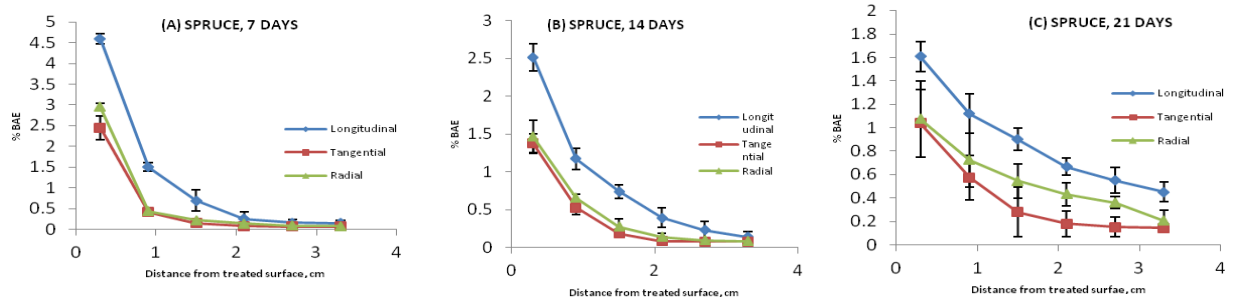
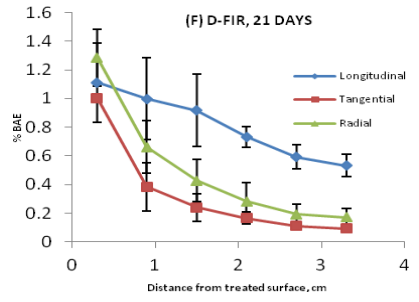
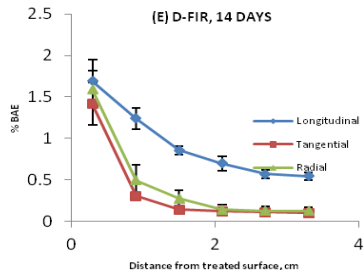
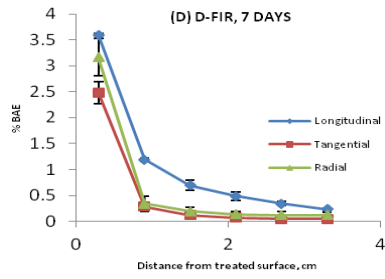


Fig. 1.6 Copper borate treated samples





APPENDIX B: ANOVA TABLES

Univariate Analysis of Variance

Independent Variables:

- Species (VAR00001)
- Formulation (VAR00002)
- Grain Direction (VAR00003)
- Moisture Content (VAR00004)

Dependent Variable : Concentration (VAR00007)

Tests of Between-Subjects Effects

Table 2.1 : DATA FOR DEPTH = 0.3 cm ; PERIOD = 7 days

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	125.272 ^a	35	3.579	44.301	.000
Intercept	1401.545	1	1401.545	17347.525	.000
VAR00001 (Species)	8.194	1	8.194	101.420	.000
VAR00002 (Formulation)	3.768	1	3.768	46.639	.000
VAR00003 (Grain direction)	80.515	2	40.257	498.283	.000
VAR00004 (Moisture content)	17.632	2	8.816	109.117	.000
VAR00001 * VAR00002	3.036	1	3.036	37.579	.000
VAR00001 * VAR00003	4.514	2	2.257	27.933	.000
VAR00001 * VAR00004	1.413	2	.706	8.742	.000

VAR00002 * VAR00003	1.027	2	.514	6.357	.003
VAR00002 * VAR00004	.199	2	.100	1.232	.298
VAR00003 * VAR00004	1.607	4	.402	4.973	.001
VAR00001 * VAR00002 * VAR00003	.522	2	.261	3.231	.045
VAR00001 * VAR00002 * VAR00004	.629	2	.315	3.895	.025
VAR00001 * VAR00003 * VAR00004	1.095	4	.274	3.389	.013
VAR00002 * VAR00003 * VAR00004	.738	4	.184	2.283	.069
VAR00001 * VAR00002 * VAR00003 * VAR00004	.384	4	.096	1.187	.324
Error	5.817	72	.081		
Total	1532.634	108			
Corrected Total	131.089	107			

a. R Squared = .956 (Adjusted R Squared = .934)

Table 2.2 : DATA FOR DEPTH = 0.9 cm ; PERIOD = 7 days

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	22.371 ^a	35	.639	56.512	.000
Intercept	29.339	1	29.339	2593.941	.000
VAR00001 (Species)	.001	1	.001	.101	.751

VAR00002 (Formulation)	.035	1	.035	3.091	.083
VAR00003 (Grain direction)	14.133	2	7.067	624.787	.000
VAR00004 (Moisture content)	4.120	2	2.060	182.120	.000
VAR00001 * VAR00002	.108	1	.108	9.536	.003
VAR00001 * VAR00003	.077	2	.038	3.401	.039
VAR00001 * VAR00004	.028	2	.014	1.255	.291
VAR00002 * VAR00003	.318	2	.159	14.038	.000
VAR00002 * VAR00004	.100	2	.050	4.429	.015
VAR00003 * VAR00004	2.238	4	.559	49.468	.000
VAR00001 * VAR00002 * VAR00003	.015	2	.008	.677	.512
VAR00001 * VAR00002 * VAR00004	.139	2	.070	6.160	.003
VAR00001 * VAR00003 * VAR00004	.538	4	.134	11.884	.000
VAR00002 * VAR00003 * VAR00004	.398	4	.100	8.804	.000
VAR00001 * VAR00002 * VAR00003 * VAR00004	.122	4	.031	2.706	.037
Error	.814	72	.011		
Total	52.524	108			
Corrected Total	23.185	107			

a. R Squared = .965 (Adjusted R Squared = .948)

Table 2.3 : DATA FOR DEPTH = 1.5 cm ; PERIOD = 7 days

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	4.660 ^a	35	.133	36.114	.000
Intercept	5.478	1	5.478	1485.816	.000
VAR00001 (Species)	.004	1	.004	1.111	.295
VAR00002 (Formulation)	.001	1	.001	.171	.680
VAR00003 (Grain direction)	1.885	2	.943	255.666	.000
VAR00004 (Moisture content)	1.310	2	.655	177.596	.000
VAR00001 * VAR00002	.033	1	.033	8.838	.004
VAR00001 * VAR00003	.019	2	.010	2.589	.082
VAR00001 * VAR00004	.066	2	.033	8.944	.000
VAR00002 * VAR00003	.064	2	.032	8.615	.000
VAR00002 * VAR00004	.008	2	.004	1.115	.334
VAR00003 * VAR00004	.831	4	.208	56.371	.000
VAR00001 * VAR00002 * VAR00003	.019	2	.010	2.643	.078
VAR00001 * VAR00002 * VAR00004	.030	2	.015	4.100	.021
VAR00001 * VAR00003 * VAR00004	.120	4	.030	8.104	.000

VAR00002 * VAR00003 * VAR00004	.119	4	.030	8.095	.000
VAR00001 * VAR00002 * VAR00003 * VAR00004	.151	4	.038	10.263	.000
Error	.265	72	.004		
Total	10.404	108			
Corrected Total	4.926	107			

a. R Squared = .946 (Adjusted R Squared = .920)

Table 2.4 : DATA FOR DEPTH = 2.1 cm ; PERIOD = 7 days

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	1.468 ^a	35	.042	19.582	.000
Intercept	1.688	1	1.688	788.295	.000
VAR00001 (Species)	.012	1	.012	5.385	.023
VAR00002 (Formulation)	.011	1	.011	5.269	.025
VAR00003 (Grain direction)	.324	2	.162	75.696	.000
VAR00004 (Moisture content)	.452	2	.226	105.499	.000
VAR00001 * VAR00002	.000	1	.000	.112	.739
VAR00001 * VAR00003	.011	2	.006	2.634	.079
VAR00001 * VAR00004	.105	2	.052	24.419	.000
VAR00002 * VAR00003	.035	2	.017	8.068	.001

VAR00002 * VAR00004	.013	2	.006	2.927	.060
VAR00003 * VAR00004	.181	4	.045	21.120	.000
VAR00001 * VAR00002 * VAR00003	.039	2	.020	9.167	.000
VAR00001 * VAR00002 * VAR00004	.013	2	.006	3.026	.055
VAR00001 * VAR00003 * VAR00004	.080	4	.020	9.367	.000
VAR00002 * VAR00003 * VAR00004	.095	4	.024	11.049	.000
VAR00001 * VAR00002 * VAR00003 * VAR00004	.098	4	.024	11.394	.000
Error	.154	72	.002		
Total	3.310	108			
Corrected Total	1.622	107			

a. R Squared = .905 (Adjusted R Squared = .859)

Table 2.5 : DATA FOR DEPTH = 2.7 cm ; PERIOD = 7 days

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	.716 ^a	35	.020	26.565	.000
Intercept	.865	1	.865	1122.627	.000
VAR00001 (Species)	.008	1	.008	10.204	.002
VAR00002 (Formulation)	.001	1	.001	1.519	.222

VAR00003 (Grain direction)	.104	2	.052	67.649	.000
VAR00004 (Moisture content)	.217	2	.109	141.127	.000
VAR00001 * VAR00002	.001	1	.001	.886	.350
VAR00001 * VAR00003	.011	2	.005	6.934	.002
VAR00001 * VAR00004	.061	2	.031	39.737	.000
VAR00002 * VAR00003	.035	2	.017	22.520	.000
VAR00002 * VAR00004	.007	2	.004	4.711	.012
VAR00003 * VAR00004	.063	4	.016	20.308	.000
VAR00001 * VAR00002 * VAR00003	.011	2	.005	6.840	.002
VAR00001 * VAR00002 * VAR00004	.003	2	.001	1.797	.173
VAR00001 * VAR00003 * VAR00004	.064	4	.016	20.763	.000
VAR00002 * VAR00003 * VAR00004	.078	4	.019	25.218	.000
VAR00001 * VAR00002 * VAR00003 * VAR00004	.053	4	.013	17.347	.000
Error	.055	72	.001		
Total	1.637	108			
Corrected Total	.772	107			

a. R Squared = .928 (Adjusted R Squared = .893)

Table 2.6 : DATA FOR DEPTH = 3.3 cm ; PERIOD = 7 days

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	.471 ^a	35	.013	20.204	.000
Intercept	.624	1	.624	937.483	.000
VAR00001 (Species)	.005	1	.005	7.440	.008
VAR00002 (Formulation)	7.002E-6	1	7.002E-6	.011	.919
VAR00003 (Grain direction)	.065	2	.033	48.959	.000
VAR00004 (Moisture content)	.127	2	.063	95.150	.000
VAR00001 * VAR00002	.003	1	.003	4.964	.029
VAR00001 * VAR00003	.008	2	.004	6.087	.004
VAR00001 * VAR00004	.042	2	.021	31.453	.000
VAR00002 * VAR00003	.021	2	.010	15.721	.000
VAR00002 * VAR00004	.003	2	.001	2.230	.115
VAR00003 * VAR00004	.042	4	.010	15.660	.000
VAR00001 * VAR00002 * VAR00003	.006	2	.003	4.349	.016
VAR00001 * VAR00002 * VAR00004	.008	2	.004	5.949	.004
VAR00001 * VAR00003 * VAR00004	.056	4	.014	21.062	.000
VAR00002 * VAR00003 * VAR00004	.041	4	.010	15.378	.000

VAR00001 * VAR00002 * VAR00003 * VAR00004	.044	4	.011	16.635	.000
Error	.048	72	.001		
Total	1.143	108			
Corrected Total	.519	107			

a. R Squared = .908 (Adjusted R Squared = .863)

Table 2.7 : DATA FOR DEPTH = 0.3 cm ; PERIOD = 14 days

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	27.553 ^a	35	.787	13.626	.000
Intercept	433.271	1	433.271	7499.456	.000
VAR00001 (Species)	.806	1	.806	13.953	.000
VAR00002 (Formulation)	1.993	1	1.993	34.493	.000
VAR00003 (Grain direction)	7.938	2	3.969	68.697	.000
VAR00004 (Moisture content)	12.004	2	6.002	103.888	.000
VAR00001 * VAR00002	.771	1	.771	13.337	.000
VAR00001 * VAR00003	1.145	2	.572	9.909	.000
VAR00001 * VAR00004	.460	2	.230	3.978	.023
VAR00002 * VAR00003	.070	2	.035	.609	.547
VAR00002 * VAR00004	.125	2	.062	1.080	.345
VAR00003 * VAR00004	.332	4	.083	1.436	.231

VAR00001 * VAR00002 * VAR00003	.140	2	.070	1.210	.304
VAR00001 * VAR00002 * VAR00004	.040	2	.020	.347	.708
VAR00001 * VAR00003 * VAR00004	.566	4	.141	2.448	.054
VAR00002 * VAR00003 * VAR00004	.564	4	.141	2.442	.054
VAR00001 * VAR00002 * VAR00003 * VAR00004	.600	4	.150	2.596	.043
Error	4.160	72	.058		
Total	464.983	108			
Corrected Total	31.712	107			

a. R Squared = .869 (Adjusted R Squared = .805)

Table 2.8 : DATA FOR DEPTH = 0.9 cm ; PERIOD = 14 days

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	18.425 ^a	35	.526	32.164	.000
Intercept	39.913	1	39.913	2438.720	.000
VAR00001 (Species)	.023	1	.023	1.426	.236
VAR00002 (Formulation)	.189	1	.189	11.530	.001
VAR00003 (Grain direction)	13.440	2	6.720	410.604	.000
VAR00004 (Moisture content)	2.935	2	1.468	89.672	.000

VAR00001 * VAR00002	.004	1	.004	.220	.641
VAR00001 * VAR00003	.151	2	.076	4.615	.013
VAR00001 * VAR00004	.099	2	.049	3.020	.055
VAR00002 * VAR00003	.143	2	.072	4.378	.016
VAR00002 * VAR00004	.090	2	.045	2.738	.071
VAR00003 * VAR00004	1.041	4	.260	15.905	.000
VAR00001 * VAR00002 * VAR00003	.062	2	.031	1.881	.160
VAR00001 * VAR00002 * VAR00004	.070	2	.035	2.130	.126
VAR00001 * VAR00003 * VAR00004	.131	4	.033	1.994	.105
VAR00002 * VAR00003 * VAR00004	.016	4	.004	.239	.915
VAR00001 * VAR00002 * VAR00003 * VAR00004	.032	4	.008	.485	.747
Error	1.178	72	.016		
Total	59.516	108			
Corrected Total	19.603	107			

a. R Squared = .940 (Adjusted R Squared = .911)

Table 2.9 : DATA FOR DEPTH = 1.5 cm ; PERIOD = 14 days

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	6.018 ^a	34	.177	36.292	.000
Intercept	7.663	1	7.663	1571.160	.000
VAR00001 (Species)	.002	1	.002	.499	.482
VAR00002 (Formulation)	.020	1	.020	4.075	.047
VAR00003 (Grain direction)	3.065	2	1.532	314.177	.000
VAR00004 (Moisture content)	1.394	2	.697	142.941	.000
VAR00001 * VAR00002	.001	1	.001	.213	.646
VAR00001 * VAR00003	.021	2	.010	2.119	.128
VAR00001 * VAR00004	.025	2	.012	2.560	.085
VAR00002 * VAR00003	.013	2	.007	1.351	.266
VAR00002 * VAR00004	.001	2	.001	.145	.865
VAR00003 * VAR00004	1.191	4	.298	61.045	.000
VAR00001 * VAR00002 * VAR00003	.003	2	.001	.306	.737
VAR00001 * VAR00002 * VAR00004	.026	2	.013	2.624	.080
VAR00001 * VAR00003 * VAR00004	.020	4	.005	1.015	.406
VAR00002 * VAR00003 * VAR00004	.011	4	.003	.561	.692

VAR00001 * VAR00002 * VAR00003 * VAR00004	.020	3	.007	1.350	.265
Error	.341	70	.005		
Total	14.490	105			
Corrected Total	6.360	104			

a. R Squared = .946 (Adjusted R Squared = .920)

Table 2.10 : DATA FOR DEPTH = 2.1 cm ; PERIOD = 14 days

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	2.459 ^a	35	.070	22.182	.000
Intercept	3.363	1	3.363	1061.964	.000
VAR00001 (Species)	.001	1	.001	.392	.533
VAR00002 (Formulation)	.004	1	.004	1.175	.282
VAR00003 (Grain direction)	1.001	2	.501	158.060	.000
VAR00004 (Moisture content)	.673	2	.337	106.335	.000
VAR00001 * VAR00002	.028	1	.028	8.852	.004
VAR00001 * VAR00003	.017	2	.008	2.613	.080
VAR00001 * VAR00004	.021	2	.010	3.273	.044
VAR00002 * VAR00003	.004	2	.002	.585	.560
VAR00002 * VAR00004	.004	2	.002	.635	.533
VAR00003 * VAR00004	.566	4	.141	44.669	.000

VAR00001 * VAR00002 * VAR00003	.015	2	.008	2.444	.094
VAR00001 * VAR00002 * VAR00004	.024	2	.012	3.851	.026
VAR00001 * VAR00003 * VAR00004	.049	4	.012	3.861	.007
VAR00002 * VAR00003 * VAR00004	.034	4	.009	2.711	.037
VAR00001 * VAR00002 * VAR00003 * VAR00004	.017	4	.004	1.348	.261
Error	.228	72	.003		
Total	6.050	108			
Corrected Total	2.687	107			

a. R Squared = .915 (Adjusted R Squared = .874)

Table 2.11 : DATA FOR DEPTH = 2.7 cm ; PERIOD = 14 days

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	1.156 ^a	35	.033	18.156	.000
Intercept	1.874	1	1.874	1029.745	.000
VAR00001 (Species)	.006	1	.006	3.331	.072
VAR00002 (Formulation)	.000	1	.000	.180	.673
VAR00003 (Grain direction)	.302	2	.151	83.120	.000
VAR00004 (Moisture content)	.303	2	.151	83.133	.000

VAR00001 * VAR00002	.018	1	.018	9.917	.002
VAR00001 * VAR00003	.027	2	.013	7.357	.001
VAR00001 * VAR00004	.065	2	.032	17.779	.000
VAR00002 * VAR00003	.001	2	.000	.180	.836
VAR00002 * VAR00004	.001	2	.001	.328	.721
VAR00003 * VAR00004	.278	4	.070	38.219	.000
VAR00001 * VAR00002 * VAR00003	.009	2	.004	2.427	.095
VAR00001 * VAR00002 * VAR00004	.013	2	.007	3.600	.032
VAR00001 * VAR00003 * VAR00004	.105	4	.026	14.468	.000
VAR00002 * VAR00003 * VAR00004	.024	4	.006	3.273	.016
VAR00001 * VAR00002 * VAR00003 * VAR00004	.004	4	.001	.589	.672
Error	.131	72	.002		
Total	3.161	108			
Corrected Total	1.287	107			

a. R Squared = .898 (Adjusted R Squared = .849)

Table 2.12 : DATA FOR DEPTH = 3.3 cm ; PERIOD = 14 days

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	.927 ^a	35	.026	23.180	.000
Intercept	1.367	1	1.367	1196.485	.000
VAR00001 (Species)	.013	1	.013	11.667	.001
VAR00002 (Formulation)	.002	1	.002	1.890	.173
VAR00003 (Grain direction)	.181	2	.090	79.083	.000
VAR00004 (Moisture content)	.208	2	.104	90.824	.000
VAR00001 * VAR00002	.016	1	.016	14.354	.000
VAR00001 * VAR00003	.046	2	.023	20.205	.000
VAR00001 * VAR00004	.077	2	.038	33.644	.000
VAR00002 * VAR00003	.002	2	.001	.672	.514
VAR00002 * VAR00004	.001	2	.000	.230	.795
VAR00003 * VAR00004	.193	4	.048	42.126	.000
VAR00001 * VAR00002 * VAR00003	.007	2	.003	3.059	.053
VAR00001 * VAR00002 * VAR00004	.018	2	.009	7.927	.001
VAR00001 * VAR00003 * VAR00004	.147	4	.037	32.186	.000
VAR00002 * VAR00003 * VAR00004	.013	4	.003	2.809	.032

VAR00001 * VAR00002 * VAR00003 * VAR00004	.004	4	.001	.904	.466
Error	.082	72	.001		
Total	2.377	108			
Corrected Total	1.009	107			

a. R Squared = .918 (Adjusted R Squared = .879)

Table 2.13 : DATA FOR DEPTH = 0.3 cm ; PERIOD = 21 days

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	5.946 ^a	35	.170	3.850	.000
Intercept	184.844	1	184.844	4188.870	.000
VAR00001 (Species)	.012	1	.012	.276	.601
VAR00002 (Formulation)	.274	1	.274	6.205	.015
VAR00003 (Grain direction)	1.031	2	.516	11.684	.000
VAR00004 (Moisture content)	2.443	2	1.222	27.682	.000
VAR00001 * VAR00002	.032	1	.032	.729	.396
VAR00001 * VAR00003	.278	2	.139	3.147	.049
VAR00001 * VAR00004	.477	2	.238	5.402	.007
VAR00002 * VAR00003	.129	2	.065	1.467	.237
VAR00002 * VAR00004	.034	2	.017	.387	.681
VAR00003 * VAR00004	.050	4	.013	.285	.887

VAR00001 * VAR00002 * VAR00003	.093	2	.046	1.049	.356
VAR00001 * VAR00002 * VAR00004	.157	2	.079	1.780	.176
VAR00001 * VAR00003 * VAR00004	.335	4	.084	1.898	.120
VAR00002 * VAR00003 * VAR00004	.415	4	.104	2.353	.062
VAR00001 * VAR00002 * VAR00003 * VAR00004	.185	4	.046	1.050	.388
Error	3.177	72	.044		
Total	193.967	108			
Corrected Total	9.123	107			

a. R Squared = .652 (Adjusted R Squared = .482)

Table 2.14 : DATA FOR DEPTH = 0.9 cm ; PERIOD = 21 days

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	9.412 ^a	35	.269	13.333	.000
Intercept	47.823	1	47.823	2371.061	.000
VAR00001 (Species)	.037	1	.037	1.822	.181
VAR00002 (Formulation)	.003	1	.003	.173	.679
VAR00003 (Grain direction)	6.966	2	3.483	172.678	.000
VAR00004 (Moisture content)	.815	2	.407	20.204	.000

VAR00001 * VAR00002	.000	1	.000	.022	.884
VAR00001 * VAR00003	.039	2	.019	.961	.387
VAR00001 * VAR00004	.178	2	.089	4.405	.016
VAR00002 * VAR00003	.085	2	.042	2.096	.130
VAR00002 * VAR00004	.060	2	.030	1.479	.235
VAR00003 * VAR00004	.344	4	.086	4.263	.004
VAR00001 * VAR00002 * VAR00003	.233	2	.116	5.770	.005
VAR00001 * VAR00002 * VAR00004	.016	2	.008	.402	.670
VAR00001 * VAR00003 * VAR00004	.230	4	.057	2.851	.030
VAR00002 * VAR00003 * VAR00004	.196	4	.049	2.433	.055
VAR00001 * VAR00002 * VAR00003 * VAR00004	.211	4	.053	2.617	.042
Error	1.452	72	.020		
Total	58.687	108			
Corrected Total	10.865	107			

a. R Squared = .866 (Adjusted R Squared = .801)

Table 2.15 : DATA FOR DEPTH = 1.5 cm ; PERIOD = 21 days

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	7.974 ^a	35	.228	19.579	.000
Intercept	14.794	1	14.794	1271.465	.000
VAR00001 (Species)	.003	1	.003	.236	.629
VAR00002 (Formulation)	.032	1	.032	2.707	.104
VAR00003 (Grain direction)	4.027	2	2.014	173.046	.000
VAR00004 (Moisture content)	2.322	2	1.161	99.765	.000
VAR00001 * VAR00002	.008	1	.008	.727	.397
VAR00001 * VAR00003	.022	2	.011	.962	.387
VAR00001 * VAR00004	.068	2	.034	2.938	.059
VAR00002 * VAR00003	.050	2	.025	2.152	.124
VAR00002 * VAR00004	.006	2	.003	.264	.769
VAR00003 * VAR00004	1.275	4	.319	27.403	.000
VAR00001 * VAR00002 * VAR00003	.016	2	.008	.680	.510
VAR00001 * VAR00002 * VAR00004	.016	2	.008	.709	.496
VAR00001 * VAR00003 * VAR00004	.052	4	.013	1.113	.357
VAR00002 * VAR00003 * VAR00004	.021	4	.005	.457	.767

VAR00001 * VAR00002 * VAR00003 * VAR00004	.054	4	.014	1.166	.333
Error	.838	72	.012		
Total	23.606	108			
Corrected Total	8.811	107			

a. R Squared = .905 (Adjusted R Squared = .859)

Table 2.16 : DATA FOR DEPTH = 2.1 cm ; PERIOD = 21 days

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	4.118 ^a	35	.118	20.927	.000
Intercept	6.884	1	6.884	1224.420	.000
VAR00001 (Species)	.005	1	.005	.939	.336
VAR00002 (Formulation)	.016	1	.016	2.826	.097
VAR00003 (Grain direction)	1.852	2	.926	164.716	.000
VAR00004 (Moisture content)	1.383	2	.691	122.952	.000
VAR00001 * VAR00002	.000	1	.000	.039	.844
VAR00001 * VAR00003	.007	2	.004	.655	.523
VAR00001 * VAR00004	.012	2	.006	1.052	.355
VAR00002 * VAR00003	.004	2	.002	.395	.675
VAR00002 * VAR00004	.006	2	.003	.518	.598
VAR00003 * VAR00004	.775	4	.194	34.479	.000

VAR00001 * VAR00002 * VAR00003	.014	2	.007	1.211	.304
VAR00001 * VAR00002 * VAR00004	.010	2	.005	.869	.424
VAR00001 * VAR00003 * VAR00004	.019	4	.005	.867	.488
VAR00002 * VAR00003 * VAR00004	.004	4	.001	.181	.947
VAR00001 * VAR00002 * VAR00003 * VAR00004	.010	4	.003	.450	.772
Error	.405	72	.006		
Total	11.407	108			
Corrected Total	4.523	107			

a. R Squared = .910 (Adjusted R Squared = .867)

Table 2.17 : DATA FOR DEPTH = 2.7 cm ; PERIOD = 21 days

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	2.210 ^a	35	.063	10.080	.000
Intercept	3.961	1	3.961	632.224	.000
VAR00001 (Species)	.013	1	.013	2.068	.155
VAR00002 (Formulation)	.011	1	.011	1.834	.180
VAR00003 (Grain direction)	.723	2	.362	57.724	.000
VAR00004 (Moisture content)	.857	2	.428	68.358	.000

VAR00001 * VAR00002	.006	1	.006	.960	.330
VAR00001 * VAR00003	.004	2	.002	.323	.725
VAR00001 * VAR00004	.010	2	.005	.767	.468
VAR00002 * VAR00003	.001	2	.000	.057	.945
VAR00002 * VAR00004	.000	2	.000	.037	.964
VAR00003 * VAR00004	.513	4	.128	20.473	.000
VAR00001 * VAR00002 * VAR00003	.002	2	.001	.173	.842
VAR00001 * VAR00002 * VAR00004	.014	2	.007	1.109	.336
VAR00001 * VAR00003 * VAR00004	.041	4	.010	1.619	.179
VAR00002 * VAR00003 * VAR00004	.004	4	.001	.167	.954
VAR00001 * VAR00002 * VAR00003 * VAR00004	.011	4	.003	.454	.769
Error	.451	72	.006		
Total	6.622	108			
Corrected Total	2.661	107			

a. R Squared = .831 (Adjusted R Squared = .748)

Table 2.18 : DATA FOR DEPTH = 3.3 cm ; PERIOD = 21 days

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	1.683 ^a	35	.048	8.210	.000
Intercept	3.039	1	3.039	518.893	.000
VAR00001 (Species)	.005	1	.005	.821	.368
VAR00002 (Formulation)	.002	1	.002	.396	.531
VAR00003 (Grain direction)	.608	2	.304	51.942	.000
VAR00004 (Moisture content)	.571	2	.286	48.763	.000
VAR00001 * VAR00002	.001	1	.001	.131	.719
VAR00001 * VAR00003	.000	2	.000	.039	.962
VAR00001 * VAR00004	.006	2	.003	.475	.624
VAR00002 * VAR00003	.000	2	.000	.018	.982
VAR00002 * VAR00004	.002	2	.001	.177	.838
VAR00003 * VAR00004	.442	4	.111	18.878	.000
VAR00001 * VAR00002 * VAR00003	.003	2	.001	.225	.799
VAR00001 * VAR00002 * VAR00004	.010	2	.005	.851	.431
VAR00001 * VAR00003 * VAR00004	.027	4	.007	1.140	.345
VAR00002 * VAR00003 * VAR00004	.003	4	.001	.121	.974

VAR00001 * VAR00002 * VAR00003 * VAR00004	.003	4	.001	.115	.977
Error	.422	72	.006		
Total	5.144	108			
Corrected Total	2.105	107			

a. R Squared = .800 (Adjusted R Squared = .702)