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**Mountain Pine Beetle Program
Working Paper 2008-02**

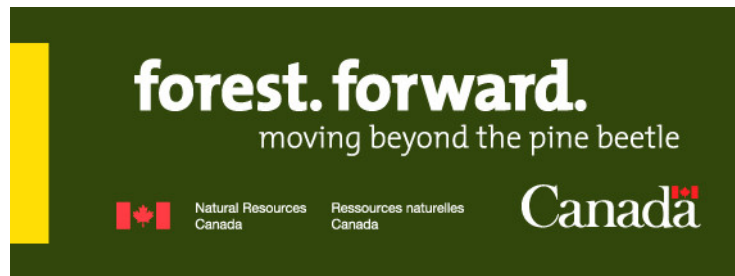
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Mountain Pine Beetle Project # 8.21

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Abstract

To mitigate the impact of the mountain pine beetle epidemic on the BC forest products sector, an understanding of the relationship between wood quality and time since infestation is needed. The proposed research project deals with the chemical and mechanical properties of mountain pine beetle-infested wood at various stages of attack. Using dynamic mechanical (DMA), and morphological (X-ray) analyses, the effect of mountain pine beetle on various wood components was determined and related to chemical composition. Chemical analysis did not show any significant differences in lignin content or sugar composition between stages of attack and location within the tree (top to bottom). Likewise, X-ray diffraction results showed similar crystallinity values (50 ± 1) for different samples. DMA analysis revealed some significant differences between the 1st and 2nd transition temperatures in the $\tan \delta$ curves of wood specimens conditioned at 60% humidity and 20°C; proposed to be the effect of variations in structure and composition between trees and within a tree rather than levels of attack.

Résumé

Pour réduire l'impact de l'épidémie de dendroctone du pin dans le secteur des produits forestiers de la Colombie-Britannique, il est nécessaire de comprendre la relation entre la qualité du bois et le temps qui s'est écoulé depuis l'infestation. Le projet de recherche proposé tient compte des propriétés mécaniques et chimiques du bois infesté par le dendroctone du pin à différents stades de l'infestation. Grâce à l'analyse mécanique dynamique (AMD) et à l'analyse morphologique (par rayons X), on a déterminé l'effet du dendroctone du pin sur différents éléments en bois et on a établi sa relation à la composition chimique. L'analyse chimique n'a pas montré de différence significative concernant le contenu en lignite ou la composition en sucre entre les stades de l'infestation ou selon l'emplacement à l'intérieur de l'arbre (du haut vers le bas). De la même manière, les résultats de la diffraction des rayons X ont montré des valeurs de cristallinité similaires (50 ± 1) pour les différents échantillons. L'analyse mécanique dynamique a révélé des différences considérables entre la 1^{re} et la 2^e des températures de transition observées dans les courbes $\tan \delta$ des échantillons de bois conditionnés à 60 % d'humidité et à 20 °C; elle suggère également qu'il s'agit de l'effet de variations dans la structure et la composition entre les arbres et à l'intérieur d'un arbre plutôt que de degrés d'infestation.

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1 Introduction

The extent of the mountain pine beetle infestation in British Columbia is of catastrophic proportions. Mild winters in the past few years, fire suppression and the remoteness of many initially attacked areas have allowed the beetle to thrive. From 2001 to 2002, the volume of beetle-attacked wood increased from 72 million m³ to 108 million m³. Approximately 4.2 million hectares of lodgepole pine stands out of the total 8 million hectares of mature pine in the central interior of the province is considered to be dead for 1-2 years (red attacked). Assuming 50% of mature pine (more than 80 years old) will be killed, and estimating that the shelf life of the mountain pine beetle-killed wood is 15 years, BC Ministry of Forests and Range plans to increase the Annual Allowable Cut (AAC) for the next 15 years. The increased AAC is not sustainable; therefore, it will need to be significantly scaled back after 15 years when the dead standing pine is deemed to have no commercial value. Even with this conservative estimate of affected mature pine and the increase in the AAC, there will be 200 million m³ of dead standing pine after 15 years.

The epidemic is causing BC companies to redirect planned timber development to absorb the immediate availability of mountain pine beetle-infested wood and the anticipated long-term losses in AAC. Typically, these companies will aggressively target high priority mountain pine beetle-infested timber for harvest during the 2004-2009 cut control period. However, the scope of the challenge presented by the epidemic will extend beyond the volume of timber harvested over the short-term to the development of value-added structural wood products produced from beetle-damaged stands over the long-term. Studies indicate that tree death is caused primarily by fungus-induced occlusion of the sapwood. The fungal infection spreads from the bark through the sapwood until it reaches the heartwood. Sapstain fungi do not grow in the heartwood due to high concentrations of diterpenoid resin acids (Martinez-Inigo et al. 1999; Dorado et al. 2000; Zheng et al. 1995). The blue-stained region always trails behind the leading edge of the infection (Solheim 1995). The infected region is preceded by a zone of occlusion. Once the zone of occlusion encircles the tree, the tree dies from lack of water transport (Solheim 1995; Martinez-Inigo et al. 1999; Parmeter et al. 1992; DeAngelis et al. 1986). The occlusive substance has not yet been identified; it may be a fungal metabolite (DeAngelis et al. 1986).

The progression of the beetle attack can be divided into three stages, based on tree foliation colour (Skakun et al. 2003). Green attack, the change from bright to dull green, occurs in the fall of the year following the attack. This stage marks the beginning of interrupted water transport. Red attack occurs as chlorophyll degrades due to cell death. By this time, most of the beetles have left the tree. Trees that have been dead for more than a year and have lost most of their foliage characterize grey attack. Green attack trees are mainly colonized by *Ophiostoma clavigerum* Harrington, *Ophiostoma montium* von Arx, *Ophiostoma minutum* Hausner and *Leptographium* species. The quantities of these fungi have often decreased by the red and grey attack stages, but other species of sapstaining fungi are also usually present (Kim et al. 2005). Red and grey attack trees are also vulnerable to infestation by other beetles which harbour decay fungi, among other things (Kim et al. 2005).

A recent study has reported that infested wood has lower lignin and hemicellulose contents as compared to sound wood (Woo et al. 2005). Structurally, lignin and to a lesser extent hemicelluloses, serve as an amorphous polymer matrix that consolidates the highly ordered fibrous network, providing adhesion between cellulose microfibrils and wood fibres. Studies have shown that sapstain fungi preferentially metabolize readily accessible non-structural wood components such as starch, proteins, triglycerides and fatty acids (Gao et al. 1994; Schirp et al. 2003). Loss of lignin and hemicellulose indicates co-infection by other fungi. However, the sapstain fungi may have an adverse effect on the mechanical properties of the sapwood because they grow through the ray parenchyma and invade the wood cell lumen (Gao et al. 1994; Ballard et al. 1982). Although the limited short term dry strength test results show either slightly increased or no change, the available data imply that in the long term, the sapstain could be an issue, especially the durability of wood products in service. The durability issue is especially important to ensure the performance of existing wood products and the development of new wood products from MPB wood as influenced by the time-since-death of the stands. Information on the physical and chemical properties of the wood can validate or check the definition of shelf life of 15 years given by the BC Forest Services and provide a more correct answer to the shelf life or durability of the wood.

2 Methods and Testing Procedures

Sample Preparation. Mountain pine beetle-attacked wood in green, red, and grey stages were collected from Clear Lake sawmill, Prince George BC in October 2004. Sampling was done during the trial when six to seven truckloads of logs from each condition were run through the sawmill. To ensure no cross contamination, mill cleanout was performed between each condition. Sample specimens were cut from the outer rings of several lodgepole pines at various stages of mountain pine beetle attack; these samples were mostly sapwood.

For thermal, X-ray and chemical analysis, sample boards were selected from 10 trees of each time-since beetle attack: < 6 months (“green”), 1 year (“red”), and 3 years (“grey”). In addition, samples were selected from three positions up the tree: bottom (B), middle (M) and top (T). The wood specimens were then machined to the appropriate size for dynamic mechanical analysis (DMA): 55 mm (length, longitudinal) x 12 mm (width, radial) x 2.5 mm (thickness, tangential). Samples were taken from blocks having the most discoloration to minimize errors according to nonuniform degradation. The samples were conditioned at 20° C and 65% relative humidity. All wood samples were then categorized according to Table 1, mixed and randomly selected for analysis. Then DMA, chemical analysis, and X-ray were conducted for all levels of attack. For chemical analysis, wood specimens were ground to 80 mesh using a Wiley mill, dried overnight at 105° C and stored under vacuum at 40° C.

Table 1. Mountain pine beetle attack sample designation

	Green	Red	Grey
Top	TN	TR	TG
Middle	MN	MR	MG
Bottom	BN	BR	BG

Four different pretreatments were applied for wood samples prior to DMA: 1. Simple conditioning at 60% humidity and 20°C for 1 week (Conditioned); 2. Acetone extraction followed by conditioning (ExC); 3. Soaking in water for 7 days followed by conditioning (SC); and 4. Soaking in water for 7 days and then oven drying overnight at 105 °C followed by conditioning (SDC). Samples were measured for moisture contents before testing. Moisture content of all tested samples was 11 ± 0.5 % dry basis.

Chemical Analysis. For each stage of attack, three samples from each position within the tree were combined and subjected to lignin and carbohydrate analyses. The ground samples were equilibrated to ambient temperature and extracted according to T264cm-97 with acetone. Klason and acid-soluble lignin were determined on the extracted wood according to T222 om-98. Carbohydrate content was determined by sugars analysis of the filtrate from the lignin content test. Arabinose, galactose, glucose, xylose, and mannose were analysed by ion chromatography. Replicates were performed for all analyses.

Mechanical Testing. For each stage of attack, two packages of 1" x 4" x 8' long material were obtained for mechanical testing. Small clear (3/4" x 3/4" x 18") test specimens were cut from the material. These specimens were straight grain and free of defects. The material was stored in a conditioning chamber at 20°C and 65% RH for more than 2 months prior to strength testing. Bending strength properties (MOR, MOE) tests were conducted on the specimens at a span of 18" under centre point loading. The MOE test involved installing a yoke to measure the mid span deflection relative to the supports. The resulting MOE estimates are apparent MOE values which included the influence of shear deformation. After the MOE testing, the test specimens were loaded to failure. The failure load and failure mode were recorded. Finally, the material was also tested for specific gravity and moisture content.

Dynamic Mechanical Analysis (DMA). Dynamic mechanical analysis was conducted using a TA Instruments Q800 DMA. A minimum of three replicates were run for each sample. Then DMA was performed using a dual-cantilever clamp and the sample chamber was purged with dry-nitrogen. As it is generally desirable to conduct DMA within the linear response region (Sun et al. 2007), the linear viscoelastic region (LVR) of the samples were determined before the dynamic test using strain sweep mode. Perpendicular samples were clamped on the radial surface and bending occurred in the tangential direction. Samples were oscillated perpendicular to the grain sinusoidally at

constant frequency (1 Hz) with increasing amplitude. The test was conducted on randomly selected samples at six different temperatures ranging from -145 to 150°C.

Single frequency, controlled strain/temperature ramp was selected as the dynamic analysis mode. All dynamic scans were collected from -145° to 150°C at a heating rate of 3°C min⁻¹, frequency of 1 Hz and strain level of 0.1%. The samples were rapidly cooled to -145°C using liquid nitrogen, and equilibrated for 5 minutes before commencing the scan.

Hygrothermal history. Samples were isothermally treated for 30 minutes at 40, 60, 80 and 100°C, cooled, and then scanned from room temperature to 270°C. Some samples were also subjected to several scans after the 1st dynamic test to see the effect of water absorption or desorption.

X-Ray diffraction (XRD). Two-dimensional X-ray diffraction data were collected from soaked and unsoaked dried samples using a Bruker AXS D8 Discover X-ray diffractometer. Samples were mounted with the grain perpendicular to the detector at a distance of 10 cm. The goniometer was tilted at an angle of 17° to allow collection of data in the range 4° < 2θ < 40°. The radiation source was Cu and the generator was set to 40 kV and 20 mA. Collection time was 100 sec. Data were collected from three pieces of each sample in order to obtain representative diffraction patterns. Twenty plots from these patterns were added together. After subtraction of the incoherent scattering (Krässig 1993), the diffraction patterns were normalized to the height of the 002 reflection, which occurs at 2θ ~22°. All samples were tested for crystallinity immediately before and after DMA. Some samples were also tested for crystallinity after relaxation in the conditioning room (60% humidity and 20°C).

Statistical analysis. Analysis of variances (ANOVA) was conducted between groups of data. The analysis followed single factor ANOVA for groups having different standard deviations. An assumption was made based on having independent variables (stages of attack) when comparing various locations in a tree; and independent variables (location) when comparing different stages of attack. That is, we assumed each type of attack is caused based on its own environmental and growth conditions and does not reflect any other part or stage of attack property. Analysis included chemical analysis data, and DMA data for the 1st and 2nd transition temperatures captured from tan δ curves. Significance of the difference between groups was analyzed based on the Fisher (F) statistic calculated compared to F critical for 1% probability of having test errors.

3 Results

Small Clear Mechanical performance. Table 2 lists the results of bending tests performed on the green, red and grey mountain pine beetle-attacked specimens. The average values of MOE and MOR decrease slightly with increased time-since attack. In almost all cases the classical failure mode of small clear bending test was observed – compression failure of the fibre in the compression leading to eventual breakage on the tension side. Additional research is required to study the strength properties of the resource to develop a clear understanding of the interacting relationship of the wood/lumber properties, mode of failure, age since attack, presence of fungal, and the bio-geoclimatic zone of the resource.

Table 2. Summary statistics for specific gravity SG, MOE and MOR for mountain pine beetle-attacked wood.

Group (N)	Green (23)			Red (14)			Grey (14)		
	SG	MOE (GPa)	MOR (MPa)	SG	MOE (GPa)	MOR (MPa)	SG	MOE (GPa)	MOR (MPa)
Average	0.485	14.9	87.1	0.465	13.9	82.0	0.478	13.2	81.6
Stdev	0.033	2.1	11.2	0.029	1.7	10.1	0.035	2.1	9.9
Max	0.552	19.7	104.8	0.533	16.2	93.9	0.521	18.0	97.8
Min	0.417	10.9	50.8	0.434	10.7	57.7	0.401	10.2	65.5
Cov		0.14	0.13		0.12	0.12		0.16	0.12

Chemical analysis of small wood specimens. The results of klason lignin and sugar analysis are shown in Table 3. Standard deviations have been reported in the parentheses with the number of replications. ANOVA analysis did not show any significant differences between stages of attack and height of attack for any of the carbohydrate monomers, nor lignin. Although mannose and xylose show some differences between stages of attack, they do not exhibit a uniform trend of increase or decrease. This is because of the high standard deviations, which shifts the data distribution from the mean. Variation in chemical analysis among stages of attack can be discussed based on non-uniform degradation. According to Blanchette and Abad (1988) chemical analysis is a misleading method to analyze decayed wood because degradation is not uniformly distributed. In this study, we tried to prepare samples from attacked areas of wood blocks, but it seems that degradation is microscopic rather than macroscopic. Ballard et al. (1982), in a microscopic study of mountain pine beetle-attacked logepole pine, revealed that the attacked tree is dead before its needles turn brown. They proposed that logepole pine has the ability to maintain water in the needles for a long period of time. That means that tree would have been dead at green stages of attack, and the transition from green to red, and then grey is because of the resistance of the needles. Our results could confirm

the proposition, which indicates no composition changes among stages of attack. It, however, could change between sound and attacked wood (Woo et al. 2005).

Table 3. Lignin and Carbohydrate Composition of Wood Samples MPB = mountain pine beetle

MPB	Arabinose	Galactose	Xylose	Mannose	Glucose	Klason lignin	ASL
TG	1.29(0.13)	1.86(0.07)	6.07(0.23)	12.06(0.31)	45.98(0.77)	30(0.38)	0.54(0.0)
MG	1.28(0.03)	1.6(0.01)	5.39(0.04)	11.64(0.09)	46.7(0.30)	30.1(1.13))	0.53(0.0)
BG	1.08(0.02)	1.85(0.06)	5.74(0.08)	11.34(0.21)	46.61(0.15)	30.1(0.95)	0.53(0.0)
TR	1.22(0.08)	1.61(0.07)	5.99(0.25)	10.97(0.45)	44.42(0.02)	31.1(0.26)	0.54(0.0)
MR	1.24(0.02)	1.69(0.01)	6.11(0.34)	11.3(0.66)	46.17(0.65)	29.1(0.38)	0.57(0.0)
BR	1.17(0.05)	1.59(0.05)	5.38(0.13)	11.78(0.30)	46.75(1.29)	28.9(1.07)	0.58(0.0)
TN	1.3 (0.00)	1.55(0.09)	5.96(0.10)	11.72(0.42)	47.03(1.24)	29.2(0.38)	0.59(0.0)
MN	1.25(0.05)	1.38(0.02)	5.98(0.39)	11.85(0.64)	47.36(0.98)	28.3(0.38)	0.53(0.0)
BN	1.24(0.05)	1.66(0.08)	5.9(0.20)	11.17(0.27)	47.17(1.74)	29.4(0.50)	0.54(0.0)

DMA analysis. Prior to DMA analysis the LVR had to be determined for the mountain pine beetle samples. Figure 1 shows the result of the LVR analysis on samples at different temperatures. The temperature range chosen covered the entire temperature range used for the DMA tests. The LVR is where the modulus remains constant to changes in strain. The LVR limit was the same range for all of the tested temperatures (0.1% to 0.25 %). As a consequence of this analysis, and comparison to other studies (Sun et al, 2007), a strain of 0.1 % was chosen for the DMA tests.

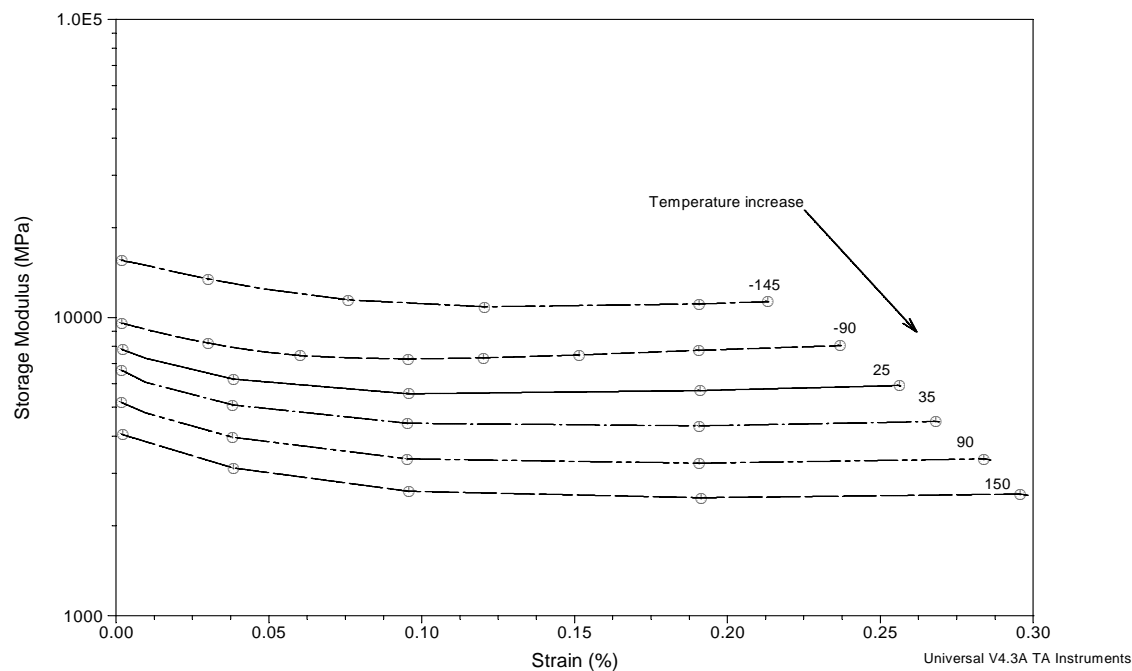


Figure 1. Linear viscoelastic region (LVR) determination of mountain pine beetle wood samples (temperatures in °C)

Figure 2 illustrates the $\tan \delta$ DMA results for all nine levels of attack for conditioned samples. All other data, a total of 144 tests, exhibited the similar graphs. Two distinctive transitions are observed on the curves with intensity below 0.1. The $\tan \delta$ peaks of both transitions showed variations between levels of attack, based on stage of attack or location within the tree. The transitions exhibited almost the same broadness interval. The lower temperature, 2nd transition temperature, is related to methylol groups (Obataya et al. 2001). Methylol groups are in lignin polymers, as well as xylan hemicelluloses. Since the xylan composition is only 5%-10 % of total hemicelluloses in softwoods, lignin would be the only major source for methylol groups. Thus any difference observed in the 2nd transition would imply a probable difference in lignin structure. The higher temperature 1st transition has been discussed as the glass transition temperature of lignin in the presence of moisture (Salmen 1984, Salmen and Olsson 1998). In fact, Sun et al. (2007) concluded that there is no transition for completely dry wood between room temperature and 150° C. They suggest that any transitions below 150°C likely arise from the presence of water molecules. Since our wood samples had an 11% moisture content, the transitions may be assigned to the glass transition of water plasticized lignin. Therefore, any differences in the transition temperature again suggests some changes of lignin structure.

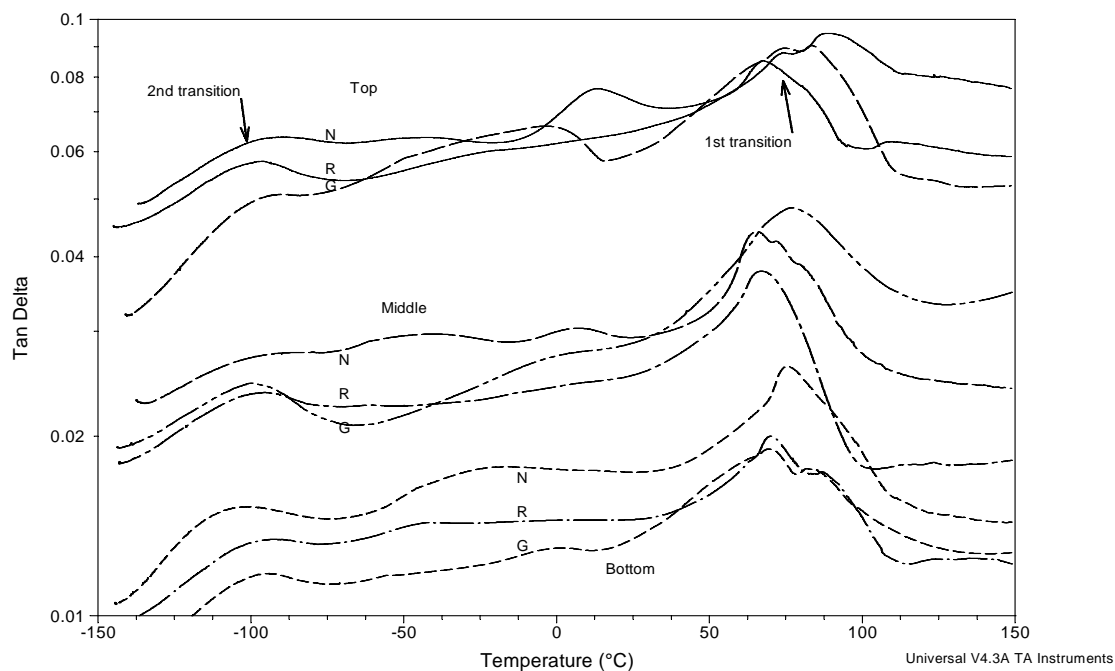


Figure 2. DMA (Tan δ curves) analysis of mountain pine beetle wood

The ANOVA analysis of the DMA data showed some differences between the conditioned samples without any other pretreatment. The results, however, were different from one treatment to the other. Tables 4 and 5 show the result of the ANOVA analysis. The analysis reveals that only some of the transition temperatures have significant differences (95% levels of confidence). Table 4 exhibits significant differences for the 1st transition on the top of tree samples and the 2nd transition in the middle part of the tree samples. Table 5 shows differences for the 1st transition between red and grey stages of attack, as well as with the 2nd transition of the green attack stage.

Table 4. ANOVA analysis of MPB DMA data based on tree position; analysis of 1st and 2nd transition temperature differences

Degree of freedom : 2 (between groups), 6 (within groups), 8 (total) F critical = 9.55						
Source Analysis Factors	1 st transition temperature			2 nd transition temperature		
	Top	Middle	Bottom	Top	Middle	Bottom
F calculated	112.7	19.22	1.62	0.47	551.66	0.60
Probability	0.002	0.190	0.270	0.580	0.0001	0.670

Table 5. ANOVA analysis of MPB DMA data based on stage of attack; analysis for 1st and 2nd transition temperature differences

Degree of freedom : 2 (between groups), 6 (within groups), 8 (total) F critical = 9.55						
Source Analysis factors	1 st transition temperature			2 nd transition temperature		
	Green	Red	Gray	Green	Red	Gray
F calculated	1.29	51.64	41.85	123.69	0.88	10.47
Probability	0.390	0.005	0.006	0.001	0.500	0.044

It seems that for all of the data the 1st transition is more affected than the 2nd transition. Also, the transition is more related to the position within the tree than stage of attack, which agrees with the result of chemical analysis regarding nonuniform degradation. The observation of different relaxation temperatures might be related to variation and heterogeneity of the amorphous polymers in the MPB wood specimens. The variation in the tan δ transition can be rationalized on the basis of differences in both density and frequency of cross-linking within the wood's amorphous components, lignin and hemicellulose (Salmen 1984). The height of the main tan δ peak is a relative measure of the amount of material taking part in the transition. Variation in the height of the tan δ peaks may reflect the change in content of amorphous wood components in the different samples (Backman and Lindberg 2001).

Hygrothermal history. Isothermal treatment at 40, 60 and 80°C for 30 minutes did not have an effect on the DMA profile; however, heat treatment at 100°C and above caused

complete desiccation and a change to the 1st transition. Figure 3 demonstrates that 100°C isothermal treatments caused changes in the $\tan \delta$ relaxation and shifted it to a higher temperature region. The temperature transition of wood softening depends on the amount of water present because water acts as a plasticizer (Salmen and Back 1977). Plasticization causes a reduction in the energy required to initiate chain mobility for the amorphous components of the wood. Consequently, the precise temperature at which the relaxations are found in the wood spectra can be expected to vary with moisture content (Salmen and Olsson 1998). It should be mentioned that transition relaxation in the thermally treated samples at very low moisture content were barely detectable.

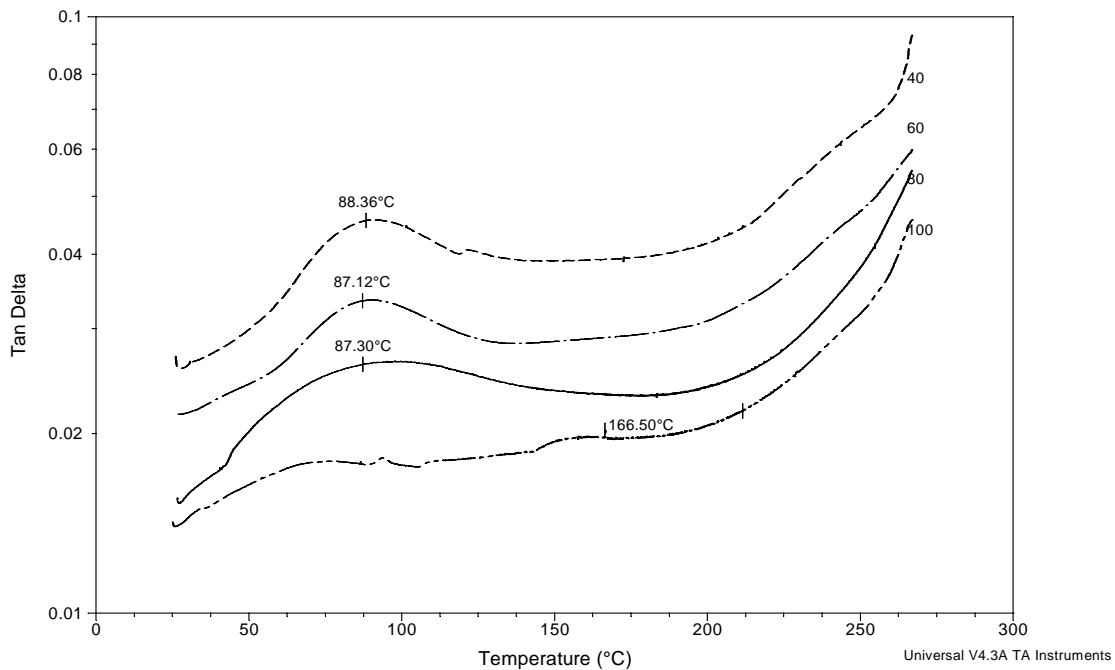


Figure 3. Hygrothermal effect. DMA scans of BG specimens after isothermal desiccation at 40, 60, 80 and 100°C (3°C/min heating rate, 1 Hz frequency and 0.1% strain).

The DMA scans were also collected on the same samples after being stored at 60% humidity and 20°C for one month after the first scan. These data showed the identical $\tan \delta$ profiles as the initial run. The observed similarity between these scans suggests repeatability of wood softening at the same transition. However, as expected when a third scan was collected immediately after the second scan, a lower storage modulus was observed and the transition was not apparent.

DMA at different conditions. The result of ANOVA analysis did not show any significant differences for $\tan \delta$ between stage and height of attack when wood samples were soaked, soak-dried, and extracted before conditioning at 60% RH and 20°C. Figure 4 illustrates the results obtained for the 1st transition for the different pretreatments.

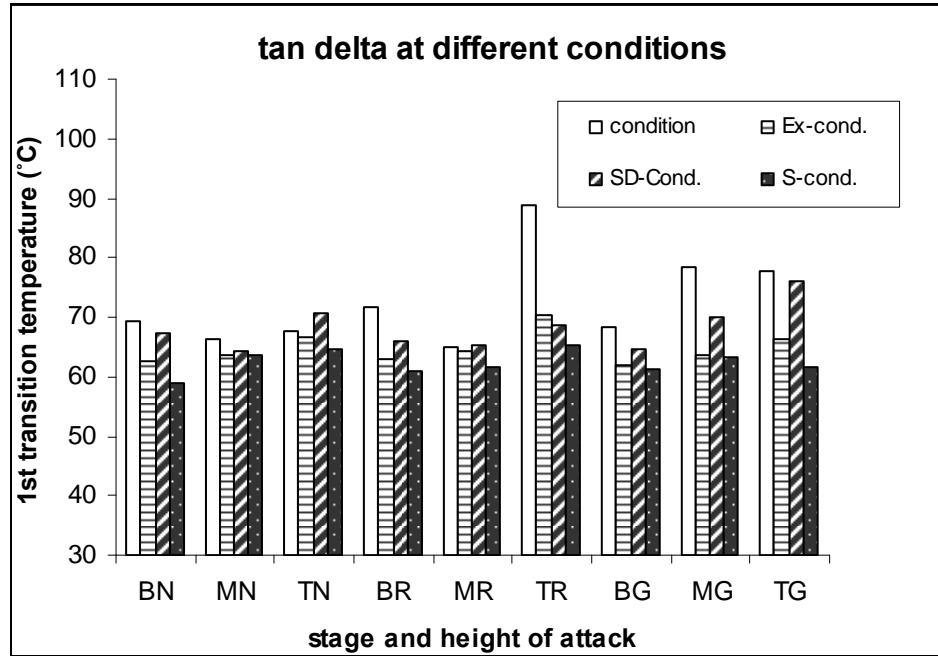


Figure 4. Analysis of $\tan \delta$ results for the various pretreatments

It can be seen that the $\tan \delta$ is the highest value for the conditioned samples regardless of location within the tree or stage of attack, followed by soaked-dried-conditioned, extracted-conditioned, and soaked-conditioned. Exceptions are Top-Normal (TN), which exhibits soaked-conditioned as the highest $\tan \delta$; and Top-red (TR) in which extracted-conditioned exceeds soaked-dried-conditioned. As mentioned, the 1st transitions are attributed mostly to the lignin component. Glass transition of amorphous polymers like lignin is influenced by the main-chain flexibility, the nature of the side groups, and the presence of plasticizers (Bower 2002). All samples had the same moisture content before DMA; therefore, the same plasticizer effect. However, each pretreatment did contribute to a difference in thermal and physical history, both of which can effect transition temperatures (Chartoff et al. 1994). Acetone extraction of samples will remove extractives as well as some polysaccharides (Whistler and Chen 1991). Likewise, water soaking, which will remove water soluble materials, swells wood and saturates all of the amorphous regions. Then, depending on how the water is removed, fast (oven drying) or slow (conditioning), the physical environment may differ; kinetic versus thermodynamic arguments. Further conditioning of oven dried samples might reinforce the absorption of water into the amorphous regions rather than natural hysteresis. Soaked-conditioned

samples however, have sufficient time to loose excess water and keep the already saturated amorphous regions without stress. It means that soaked-dried-conditioned samples might exhibit properties of less amorphous domains, which agrees with the $\tan \delta$ results. Finally, it can be concluded that the non-pretreated conditioned samples should exhibit a higher level of amorphous material than the pretreated samples.

Figure 5 shows the $\tan \delta$ results for the 2nd transition temperature at different conditions. It seems that the different pretreatments have less effect on the 2nd transition than on the 1st transition (Fig. 4). The order of highest to the lowest temperature is also different as compared to the 1st transitions. Here, the extracted-conditioned shows the lowest 2nd transition temperature. As mentioned, methylol groups have been reported as being responsible for the 2nd transitions (Obataya et al. 2001). Methylol groups might be affected by acetone extraction and heat drying, which could shift the temperature.

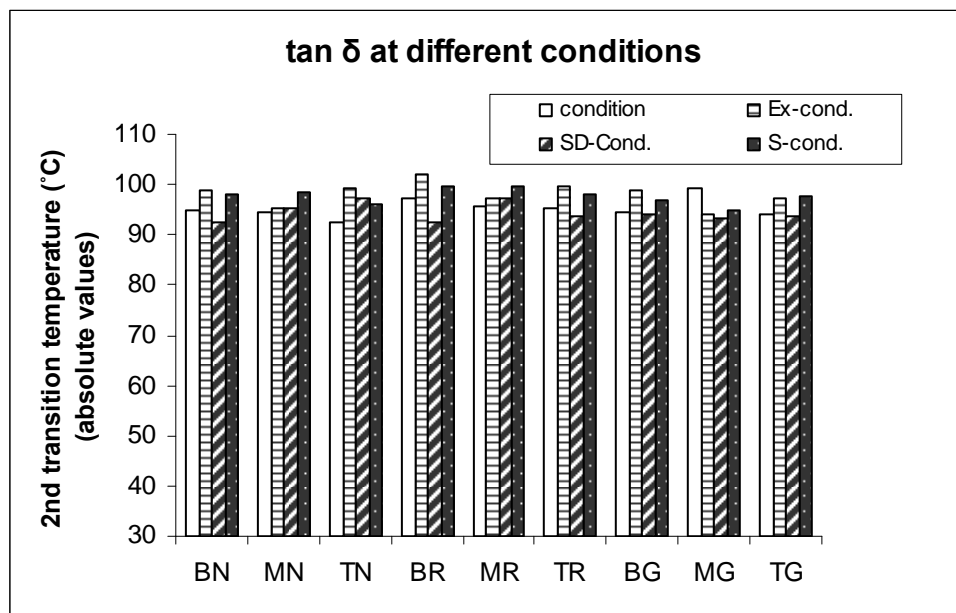


Figure 5. Analysis of $\tan \delta$ results for the various pretreatments

X-ray Analysis. Figure 6 shows a typical 2θ plot for an x-ray diffraction pattern from a wood sample. Degree of crystallinity (X_c) was calculated by the ratio of the areas of corresponding to crystalline region (peaks) and amorphous regions in the diffractograms. Considering stages of attack, tree height and all four types of wood pretreatment, the X_c is the same for all samples (50 ± 1). This implies there is no significant difference in the wood polymers in the different levels of attack, consistent with the chemical analysis.

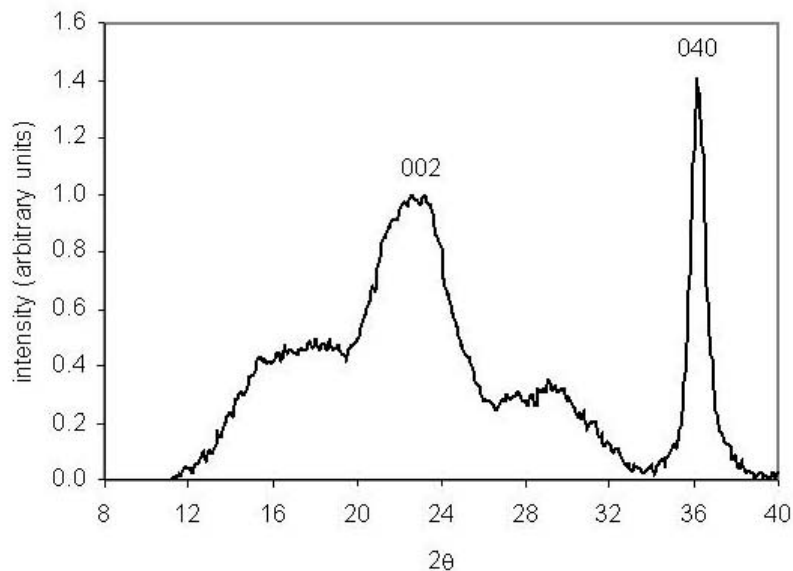


Figure 6. 2θ Plot of an XRD pattern from a green attack wood sample.

It was found that heating samples (150°C) in the DMA chamber causes a drop in crystallinity to ~ 40 . However, this decrease is temporary and analysis after three weeks of relaxing the specimen in a conditioned environment revealed the same initial degree of crystallinity. It seems heating the wood specimens up to 150°C and applying a controlled strain affects intermolecular interactions, perhaps introducing stress between microfibrils and matrix substances. Upon removal of the applied strain, the wood constituents are able to slowly relax back to the initial chemical/physical environment. This is an interesting phenomenon and we are continuing to investigate the effect of thermal processing on the wood polymer environment.

4 Conclusion

Statistical analysis was conducted on the results obtained from both chemical and dynamic mechanical analyses of mountain pine beetle-attacked wood. The results of chemical analysis did not show any significant differences between stages of attack and location within the tree (top to bottom). Likewise, X-ray diffraction results showed similar crystallinity values (50 ± 1) for different samples. The DMA analysis revealed some significant differences between the 1st and 2nd transition temperatures in the $\tan \delta$ curves of wood specimens conditioned at 60% humidity and 20°C ; these differences are proposed to be the effect of variations in structure and composition between trees and within a tree rather than levels of attack. Subjecting the wood specimens to different pretreatments (solvent extraction; water soak; water soak-dry) prior to conditioning did not have any significant effect on the DMA results. Based on these analyses, it seems that

there is no significant chemical or structural difference between the mountain pine beetle-attacked lodgepole pine investigated in this study as a function of time-since-attack.

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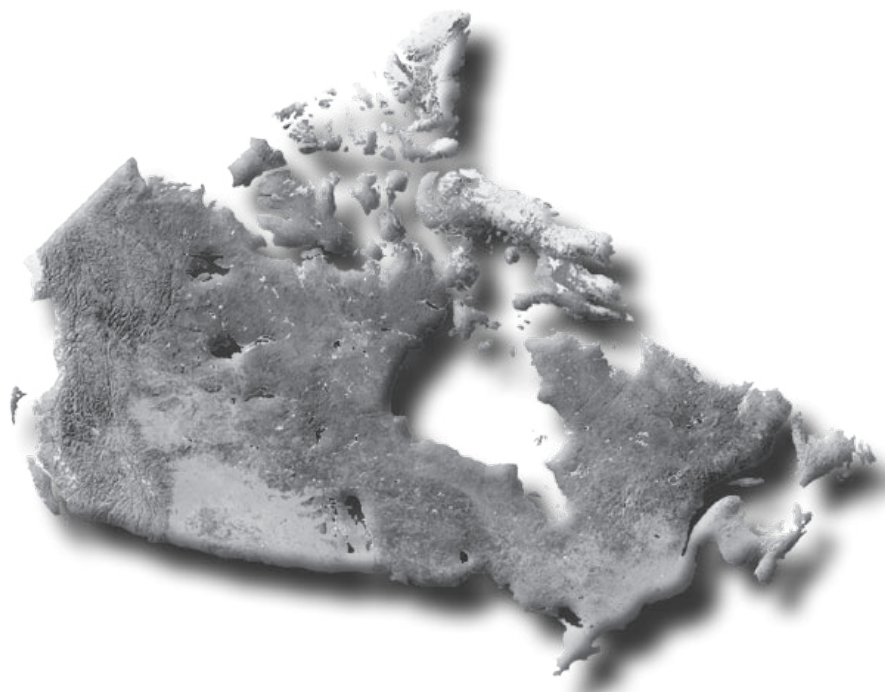
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