Journal of Korea TAPPI
Vol. 41. No. 1, 2009
Printed in Korea

Quality of Mountain Pine Beetle Infested Fibers: Implications on the Production of Pulp and Paper Products

Yong Sik Kim[*1], Alice Obermajer[*2], Reza Korehei[*1], John F. Kadla[*1], and Byung-Ho Yoon[†]

(Received January 28, 2009: Accepted February 25, 2009)

ABSTRACT

Mountain Pine Beetle (MPB) attacked pine was evaluated for pulp quality, chemical and physical properties, and bleachability. Chemical analyses showed that the dehydroabietic acid and total acetone extractives content were higher in the year 3 (grey) attacked MPB chips and lower in the year 5 (grey) attacked MPB chips as compared to a typical SPF (spruce/pine/fir) reference. Lignin and carbohydrate content of the MPB wood chips were comparable to the SPF. Similarly, there was little difference in kappa number, pulp yield and liquor consumption between the 3 and 5 year MPB attacked wood. Likewise there was no significant difference in the resulting tear strength, burst, or tensile strength. There appeared to be an improved bleaching response in the MPB attacked pulp as compared to the SPF reference, but this was accompanied by a slightly lower bleached pulp yield and higher bleach filtrate COD and solids content.

Keywords : *bleached and kraft pulp, chemical and mechanical properties, lodgepole pine, mountain pine beetle (MPB)*

1. Introduction

The largest epidemic caused by the mountain pine beetle (MPB), *Dendroctonus ponderosae hopkins*, in Canada's history is compelling BC's forest industry to utilize large volumes of dead and dying Lodgepole Pine, *Pinus contorta* (1-3). 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 (4-6). 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 (4,7-9). It was reported that the morphology and chemistry of MPB killed wood was altered (16). However, the impact of MPB attack on wood processability and pulp properties have not been thoroughly investigated. It is known that the fungi

• Changgang Institute of Paper Science and Technology, Kangwon National University, 200-701 Chunchon, South Korea
*1 Biomaterials Chemistry, Faculty of Forestry, University of British Columbia, Vancouver, BC, Canada
*2 Research and Development Group, Canfor Pulp Limited Partnership, Vancouver, BC Canada
† Corresponding Author: E-mail; bhyoon@kangwon.ac.kr

associated with MPB causes a blue stain in the sapwood (7-8,10-13) and extensive checking, which lowers the commercial value of the MPB infested wood for use in lumber and pulp and paper products (14-15).

To mitigate the impact of the MPB epidemic on the BC forest products sector, an understanding of the relationship among pulp processing, quality, and time since infestation is required. This work will comprehensively investigate the effect of MPB infected wood on chemical pulp processing efficiency and the consequential fibers quality at various times since beetle attack in the grey stage. Using chemical and physical analyses, the effect of MPB time of attack on the pulping and bleaching will be determined. As a result, this study will help predict the infestation time span within which some of the processing and pulp properties of wood can still be considered useful as well as potentially discovering new value-added process streams. Therefore, the aim of this study is to integrate fundamental and practical information to develop strategies for the extraction of the highest value from MPB infested wood while minimizing detrimental effects during processing.

2. Materials and Methods

2.1 Chip samples

Two truckloads of three year old MPB grey attacked stems from the Chedakuz area and two truckloads of five year old MPB grey attacked stems from the Vantyne area were delivered to Plateau sawmill near, B.C. Both areas have an identical biogeoclimatic zone. The two truckloads each of full-length stems were sawn into dimensional lumber. The two truckloads of 3 year attack were run first, followed by the 2 truckloads of 5 year attack. Chips (approximately 50 kg each) were collected directly off the chip/canter. For the SPF (spruce, pine and douglas fir) reference, chips were collected from the chip bin during processing of normal timber. A grab sample of 400 g for chemical analysis was taken prior to screening.

Chip screening was done on a Linden Chip Classifier according to standard mill method. Batch weight (10 L volume) and fraction weights were recorded. Fractions of 10 mm bar, 16 mm round, 7 mm round, 2 mm bar, and fines were collected after 10 minutes of agitation. Bark was removed from each fraction prior to weighing. Chip mixtures of 70% 16 mm round and 30% 7 mm round fractions were prepared for the pulping study and stored in a cooler at 5℃.

2.2 Chemical compositions

2.2.1 Total extractives and lignin content

Acetone extractives of wood chip and pulp samples were determined by weight according to TAPPI T280 pm-99. Klason and acid-soluble lignin were determined on extractive free wood chips according to TAPPI Method T222 om-98 and TAPPI Useful Method UM250, respectively.

2.2.2 Carbohydrate content

The amount of carbohydrate was determined by sugar analysis of the Klason lignin filtrate using High Performance Anion Exchange Liquid Chromatography. The HPLC system (Dionex DX-500, Dionex, CA, USA) was equipped with an ion-exchange PA1 (Dionex) column, a pulsed amperometric detector with a gold electrode, and a Spectra AS3500 auto-injector (Spectra-Physics, CA, USA). Prior to injection, samples of 20 μL volume were filtered through a 0.45 μm HV filter (Millipore, MA, USA) containing fructose as an internal standard. The column was equilibrated with 0.25 M NaOH (Fisher Scientific, Nepean Ont.) and eluted with de-ionized water at a flow rate of 1.0 $mL·min^{-1}$.

2.2.3 Resin acid content

The wood chip samples were prepared by air-drying and grinding in a Wiley mill to 1 mm mesh. Aqueous extractions for wood and pulp samples were carried out at pH 11 for 24 h at 80℃. Fibers to water ratio was 1.5 g to 150 mL. Analysis of dehydroabietic acid was conducted using a Shimadzu SCL-10A HPLC with LC-10AD pump and SPD-10AV variable wavelength UV/VIS detector at 200 nm. The sample extracts were injected in duplicate at pH 11 into a 20 µL injection port with a 0.45 µm PTFE pre-filter and were carried with a mobile phase of 65% acetonitrile and 35% acetic acid at 2.0 $mL\cdot min^{-1}$ through a Whatman Universal RP guard column, Zorbax Rx-C8 guard column, and a Zorbax Rx-C8 4.6 mm × 250 mm reverse phase column.

2.3 Chemical pulping properties

2.3.1 Kraft pulping

SPF, MPB 3 year and MPB 5 year attacked chips underwent kraft pulping to a Kappa target of 30 ± 2 in a batch digester. Chemical charge was 16 % on OD wood based on AA (active alkali) grams of Na_2O required; liquid to wood ratio was 4:1. Optimization involved variations of H factor in the range of 1400 to 2000 and chemical charge in the range of 13 % to 18 % AA. Chips were pre-steamed for 20 minutes at 60°C at atmospheric pressure. The cooking cycle involved impregnation for 35 minutes to 115℃ followed by ramping to 168℃ and 120 psi. Pulps were disintegrated, washed, and screened. Optimized pulps were combined for further analysis, including chemical compositions, fibers and strength properties, and bleaching response.

2.3 2 Kappa number

Kappa number was determined according to TAPPI Standard Method T236 cm-85. The test was performed on a Radiometer KTS1 Semi Automatic Kappa Analyser which carries out the reagent addition, mixing, and potentiometric titration to 270 mV.

2.4 Black liquor analysis

Density was measured with a hydrometer. Solids content was determined gravimetrically. Residual effective and AA were determined by barium carbonate precipitation and potentiometric titration with hydrochloric acid to pH 8.3.

2.5 Chemical pulp strength properties

Analysis of fibers length and coarseness was done on a Kajaani FS-200. Lab scale PFI refining was done at 0, 3000, 6000, and 9000 revolutions for the evaluation of pulp strength as well as physical and optical properties by Standard TAPPI Methods.

2.6 Pulp bleaching

SPF and MPB grey attacked pulps underwent D_oEpD_1 bleaching. Process conditions are shown in Table 1. Bleaching response was monitored by brightness, Kappa number, and chemical consumption. Dissolved solids and chemical oxygen demand (COD) of the bleach filtrates were measured (CPPA standard H.3) in order to evaluate impact on the mill's effluent treatment system. To investigate impact on yield, alkali

Table 1. Bleaching conditions

D_o	
Kappa Factor	0.17
Consistency (%)	3
Time (min)	20
Temperature (°C)	50
pH initial	4
E_p	
H_2O_2 (%)	0.55
NaOH (%)	1.80
Consistency (%)	10
Time (min)	90
Temperature (°C)	82
pH initial	11
D_1	
ClO_2 (%)	1.30
Consistency (%)	10
Time (min)	120
Temperature (°C)	68
pH initial	3

solubility (S18) of the bleached pulps was determined according to TAPPI T235 cm-00.

3. Results and Discussion

3.1 Chemical composition of chip samples

Table 2 shows the lignin and carbohydrate composition of the SPF and MPB attacked wood samples. The 5 year attacked wood had slightly lower lignin content than the 3 year attacked wood; however, the sugar analysis results were essentially the same. According to Woo et al. (15), MPB killed wood generally has lower carbohydrate, lignin, and extractives content compared to healthy wood. However, this trend was not shown clearly in Tables 2 and 3.

Table 3 displays the extractives content and fibers properties of the wood chip samples. Moisture content of the MPB attacked chips are lower in comparison to the SPF by 20%. The low moisture content in the MPB attacked chip was similar to that observed by Woo et al. (16) and Chow and Obermajer (17). The 5 year attacked fibers had a 23% higher caustic solubility than the 3 year attacked fibers, which indicates higher presence of decay with increasing time since beetle attack. The 3 year attacked wood chips had a 32% higher amount of total extractives and 83% higher resin acid content than the 5 year attacked wood chips. The higher extractives would offer improved protection from decay organisms. The grey attacked wood chips had lower bulk density and pH than the SPF reference which may be due to higher resin content.

3.2 Chemical pulping properties

Fig. 1 shows optimization of chemical charge at constant 1600 H factor and shows that 16% active alkali is required to reach a 30 Kappa for the MPB attacked fibers. Table 4 shows the pulping properties of the wood chip samples pulped under optimal conditions. The Kappa numbers of the MPB attacked wood fell

Table 2. Lignin and carbohydrate composition of wood chips

Chip Sample	Wood Chemical Composition						
	Klason Lignin, %	Acid soluble lignin, %	Arabinose, %	Galactose, %	Glucose, %	Xylose, %	Mannose, %
SPF	26.4	0.51	1.52	3.13	50.6	5.88	12.51
3 year attack	26.3	0.52	1.80	2.86	50.3	6.02	13.73
5 year attack	24.7	0.52	1.67	3.02	50.7	6.05	12.54

Table 3. Extractives content and fibers properties of wood chip samples

Chip sample	pH	Moisture content (%)	Bulk density (g/cm^3)	Acetone extractives (w %)	Resin acid (mg/kg)	Decay (% solubility)
SPF	4.98	48	0.1306	2.54	1292	14.0
3 year attack	4.56	40	0.1193	3.04	1844	12.1
5 year attack	4.55	40	0.1256	2.31	1006	14.9

Table 4. Pulping properties and black liquor analysis of SPF and MPB grey attacked wood

Sample	Yield (%)	Reject (%)	Kappa number	Black liquor density (g/cm^3)	Black liquor solid (%)	Residual alkali (%)	Dehydroabietic acid (ppm)
SPF	45.7	0.23	36	1.080	16.44	16.80	168.9
3 year attack	46.4	0.11	31	1.080	17.39	17.60	290.6
5 year attack	45.7	0.26	30	1.075	17.38	17.50	207.4

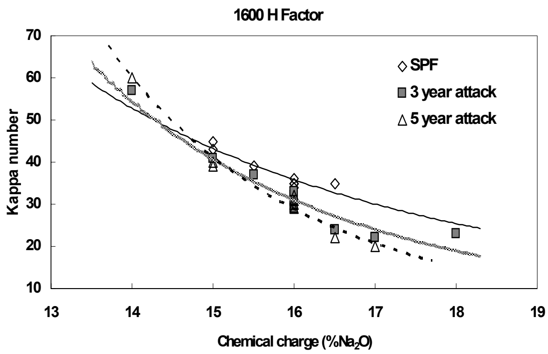

Fig. 1. Kappa number versus chemical charge at 1600 H factor.

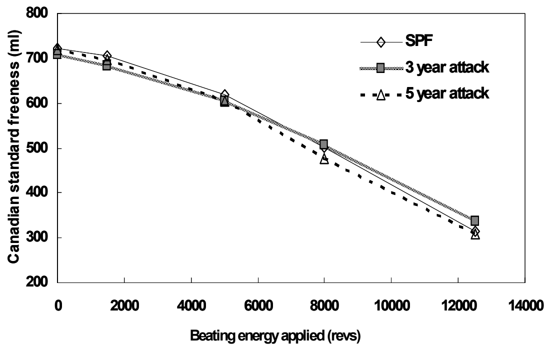

Fig. 2. Canadian standard freeness versus refining energy.

within the target range of 30 ± 2. The MPB attacked wood pulped to a higher yield than the SPF as a function of Kappa number.

The black liquor from pulping MPB infested wood had 6% higher solids content in comparison to the SPF (Table 4). Likewise, the resin acid content was significantly higher in the MPB pulped black liquor. Relative to the SPF, dehydroabietic acid concentrations were 72 % higher in black liquor from the 3 year attack and 22 % higher from the 5 year attack. This data has implications on a mill's effluent treatment capacity. However, dehydroabietic acid was not detected in any of the pulp samples

3.3 Physical properties of kraft pulps

Table 5 shows that the MPB attacked fibers were less coarse than the SPF. Coarseness of the 3 year and 5 year attacked fibres were lower by 8% and 12% respectively. In addition, the fiber length of the MPB attacked wood was lower than that of the SPF by 7% for the 3 year attacked wood and 3% for the 5 year attacked wood. There was no significant difference in the ease of refining between the SPF and MPB attacked pulps (Fig. 2). However, the MPB attacked pulps had some different physical properties, for example, they form less porous and denser sheets in comparison to the SPF pulp (Figs. 3 and 4).

Table 5. Coarseness and fiber length of SPF and MPB grey attacked fibers

Sample	Coarseness (mg/m)	Fiber length (mm)
SPF	0.233	2.68
3 year attack	0.213	2.49
5 year attack	0.206	2.60

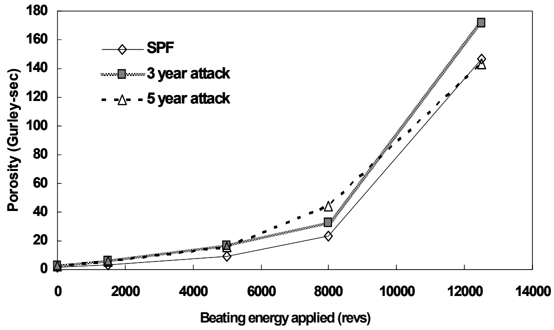

Fig. 3. Porosity versus refining energy.

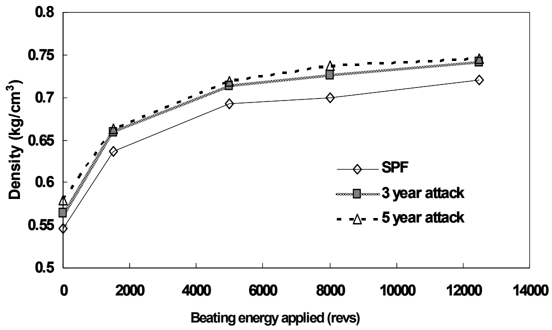

Fig. 4. Density versus refining energy.

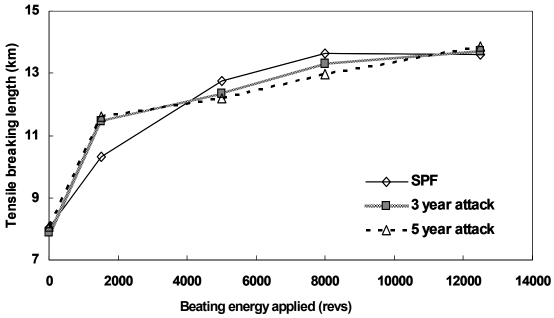

Fig. 5. Tensile breaking length versus refining energy.

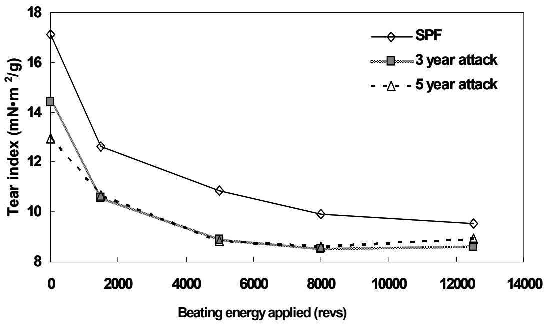

Fig. 6. Tear index versus refining energy.

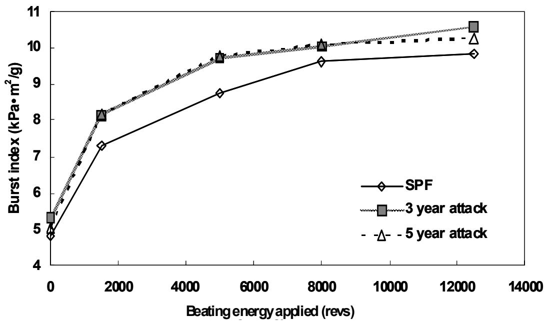

Fig 7. Burst index versus refining energy.

No significant difference in tensile strength was observed between the SPF and MPB attacked pulps (Fig. 5). However, the MPB attacked pulps were shown to have lower tear strength and higher burst in comparison to the SPF pulp (Figs. 6 and 7). This may be due to the MPB attacked pulps having less coarse and denser fibers in comparison to the SPF pulp, which could affect tear and burst strength properties.

3.4 Pulp bleaching

Table 6 summarizes the pulp response to chlorine dioxide. The MPB attacked pulp bleached to higher final brightness that the SPF. Brightness of the 3 year attacked and 5 year attacked MPB pulps were 5 % and 9 % higher, respectively, than that of the SPF. This suggests improved bleaching response with increasing time since beetle attack.

Alkali solubility tests were 5 % and 1 % higher for the bleached 3 year and 5 year attacked pulps respectively in comparison to the SPF, and suggest that there may be an impact on yield. Bleach filtrate results

Table 6. Bleaching response of pulp and filtrate analysis

Pulp		SPF	3 year attack	5 year attack
Kraft pulp	Brightness	22.9	25.4	24.4
	Kappa	36.0	31.0	30.0
D_oE_p	Brightness	44.4	47.9	48.8
	Kappa	10.6	8.2	8.0
	Filtrate COD (mg/L)	3278	5476	5938
	Filtrate dissolved solids (%)	42.0	41.5	43.9
D_1	Brightness	64.1	67.3	69.7
	Kappa	3.5	2.7	2.5
	Alkali solubility (S18)	16.1	16.9	16.3
	Filtrate COD (mg/L)	1748	1472	1579
	Filtrate dissolved solids (%)	0.43	0.43	0.40
Total filtrate COD (mg/L)		5026	6948	7517
Total filtrate dissolved solids (%)		42.4	41.9	44.3

are shown in Table 6. Filtrate COD was significantly higher in the MPB attacked samples as compared to the SPF. Bleach filtrate from the 5 year attacked samples had on average 8 % higher COD than filtrate from the 3 year attacked samples, and the total filtrate dissolved solids were 6 % higher in the 5 year attack than in the 3 year attack.

4. Conclusions

Moisture content of the MPB attacked chips was lower in comparison to the SPF reference, whereas total acetone extractive and dehydroabietic acid content in the MPB attacked chips were higher in the 3 year attack and lower in the 5year attack in comparison to the reference SPF. Lignin and carbohydrate content of the MPB attacked wood samples were comparable to the SPF.

The MPB grey attacked wood appeared to be easier to cook in comparison to the SPF. Kappa, yield, and liquor consumption results indicated that there was little difference between the 3 and 5 year attacked material. The black liquor from the pulping of MPB grey attacked wood had a 6 % higher solids content in comparison to the SPF. Resin acid was significantly higher in the MPB pulped black liquor. Relative to the SPF, dehydroabietic acid concentrations were 72 % higher in the 3 year attack and 22 % higher in the 5 year attack black liquors. This has implications on a mill's effluent treatment capacity.

The MPB attacked fibers were less coarse and had shorter fiber length than the SPF. However, both the MPB attack and SPF fibers refined similarly. In comparison to the SPF, the MPB attacked pulps have slightly lower tear strength, higher burst, and similar tensile strengths. No significant difference was seen in the physical properties between 3 year and 5 year attacked conditions. There was improved bleaching response seen in MPB attacked pulp, but at the cost of slightly lower yield. The MPB attacked bleach filtrates had higher COD and solids than that of the SPF.

Acknowledgements

The authors gratefully acknowledge financial assistance provided by the Province of BC through the Forestry Innovation Investment Program, FII, as well as Canfor R&D for their financial support of this research.

Literature Cited

1. McGrarrity, K. and Hoberg, G.,The beetle challenge: An overview of the mountain pine beetle epidemic and its implications, 2005 http://www.policy. forestry.ubc.ca/issuebriefs/pinebeetle.html.
2. Aukema, B.H., Carroll, A.L., Zhu, J., Raffa, K.F., Sickley, T.A. and Taylor, S.W. ,Landscape level analysis of mountain pine beetle in British Columbia, Canada - Spatiotemporal development and spatial synchrony within the present outbreak - , Ecography 29(3): 427 (2006).
3. Lee, S., Kim, J.J. and Breuil, C., Leptographium longiclavatum sp. nov., a new species associated with the mountain pine beetle, Dendroctonus ponderosae, Mycol. Res. 109(10): 1162 (2005).
4. Martínez-Iñigo, M.J., Immerzeel, P., Gutierrez, A., Del Rio, J.C. and Sierra-Alvarez, R., Biodegradability of extractives in sapwood and heartwood from Scots pine by sapstain and white-rot fungi, Holzforschung, 53(3): 247 (1999).
5. Dorado, J., Claassen, F.W., Van Beek, T.A., Lenon G., Wijnberg, JBPA and Sierra-Alvarez, R., Elimination and detoxification of softwood extractives by white-rot fungi, J. Biotech. 80(3): 231 (2000).
6. Zheng, Y., Ruddick, J.M.R. and Breuil, C., Factors affecting the growth of Ophiostoma-piceae on Lodgepole pine heartwood, Mat. und Org. 29(2): 70 (1995).
7. Solheim, H., Early stages of blue-stain fungus invasion of lodgepole pine sapwood following mountain pine-beetle attack, Can. J. Bot. 73(1): 70 (1995).
8. Parmeter, J.R., Slaughter, G.W., Chen, M. and Wood, D.L., Rate and depth of sapwood occlusion following inoculation of pines with bluestain fungi, Forest Sci. 38(1): 34 (1992).
9. DeAngelis, J.D., Hodges, J.D. and Nebeker, T.E.,

Phenolic metabolities of *Ceratocystis minor* from laboratory cultures and their effects on transpiration in loblolly pine seedlings, Can. J. Bot. 64(1): 151 (1986).

10. Zink, P. and Fengel, D., Studies on the coloring matter of blue-stain fungi, 1. General characterization and the associated compounds, Holzforschung, 42(4): 217 (1998).
11. Gao, Y., Breuil, C. and Chen, T., Utilization of triglycerides, fatty acids and resin acids in lodgepole pine wood by a sapstaining fungus *Ophiostoma piceae*, Mat. Org. 28(2): 105 (1994).
12. Ballard, R.G., Walsh, M.A. and Cole, W.E., Blue-stain fungi in xylem of lodgepole pine: a light-microscope study on extent of hyphal distribution, Can. J. Bot. 60(11): 2334 (1982).
13. Kim, J.J., Allen, E.A., Humble, L.M. and Breuil, C., Ophiostomatoid and basidiomycetous fungi associated with green, red, and grey lodgepole pines after mountain pine beetle (*Dendroctonus ponderosae*) infestation, Can. J. Forest. Res. 35(2): 274 (2005).
14. Harvey, R.D., Rate of increase of blue stained volume in mountain pine-beetle killed lodgepole pine in northeastern Oregon USA,Can. J. Forest Res. 9(3): 323 (1979).
15. Fahey, T.D., Snellgrove, T.A. and Plank, M.E., Changes in product recovery between live and dead Lodgepole pine, 1986 A compendium, USDA, Forest Service, Pacific Northwest Research Station, Portland, OR, pp, 1-25.
16. Woo, K.L., Watson, P. and Mansfield, S.D., The effects of mountain pine beetle attack on lodgepole pine wood morphology and chemistry: Implications for wood and fiber quality, Wood Fiber Sci. 37(1): 112 (2005).
17. Chow, S. and Obermajer, A., Moisture and blue stain distribution in mountain pine beetle infested lodgepole pine trees and industrial implications, Wood Sci. and Tech. 41(1): 3 (2007).