



**Forintek
Canada
Corp.**

Forintek Canada Corp.
Western Division
2665 East Mall
Vancouver, BC
V6T 1W5

**Bondability of Beetle-Killed Lodgepole
Pine for the Manufacture of Wood
Composite Products**

by

Martin W. Feng
Research Scientist
Composites Group

and

Guangbo He
NSERC Post Doc
Composites Group

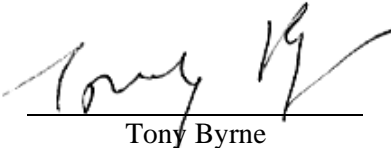
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Martin Feng
Project Leader



Tony Byrne
Reviewed



Jean Cook
Department Manager

Summary

To maximize value recovery from post mountain pine beetle - wood (MPB wood) for the manufacture of wood composite products, it is desirable to use completely MPB wood as OSB, MDF or particleboard furnish. The objective of this study in the first fiscal year was to determine and quantify the chemical properties, bondability and wettability of grey stage MPB wood in order to minimize or reduce the impact of beetle-killed wood on composite panel manufacturing. Investigation of the chemical and physical properties of grey stage MPB wood, such as wood pH and buffer capacity, wettability and bondability was conducted. Green lodgepole pine and aspen were used to compare the test results. Various wood furnish derived from MPB wood and green lodgepole pine have been prepared for the manufacturing testing of OSB, MDF and particleboard panels in the next fiscal year. The test results indicated that some basic chemical and physical properties of lodgepole pine, particularly in the sapwood area, had undergone changes associated with MPB infestation.

Based on the test results so far, the following conclusions are made:

1. The pH values of both the MPB heartwood and sapwood were lower and their acid and base buffer capacities were higher than those of the green lodgepole pine. As a result, the curing rate of pH sensitive adhesives such as UF and MUF may be affected.
2. MPB sapwood showed extremely fast and high water absorption but its thickness swell was lower than those of the MPB heartwood, green pine sapwood and heartwood regardless of water temperatures.
3. Thickness swell of the MPB sapwood almost reached to the maximum in the first two hours of water soaking at 20°C.
4. The water absorption of sapwood was higher but the thickness swell was lower than that of heartwood in both MPB wood and green lodgepole pine. The rates of water absorption and thickness swell of these woods were fast in the first several hours and slowed down thereafter.
5. Both the MPB heartwood and the green pine heartwood behaved very similarly in terms of water absorption rate and percentages. It appeared that the beetle infestation did not significantly affect the water absorption property of the MPB heartwood.
6. Edge thickness swell and center thickness swell of the MPB sapwood behaved very similarly in terms of the rates and percentages, which were quite different from those of the other woods and suggest that the blue stained MPB sapwood had probably undergone profound changes.
7. Higher temperatures led to faster and more water absorption. The water temperature affected the MPB sapwood more than the MPB heartwood.
8. Thickness swell reached to the equilibrium faster at higher temperatures.

9. Water pH had little influence on water absorption but affected thickness swell. The thickness swell of both MPB wood and lodgepole pine decreased under both acidic and alkaline conditions.
10. The bonding strength of MPB and green lodgepole pine with liquid PF, powdered PF and liquid UF were generally comparable to that of aspen at high press temperatures. Both the MPB wood and green pine showed lower bonding strength than aspen at low press temperatures. This may have significant implications on the bonding quality of the core layer of panels.
11. At high temperature (200°C), green pine produced substantially higher MDI bonding strength while MPB wood and aspen gave lower and similar bonding strength. This was also the case at low press temperature (140°C), particularly in longer press time. The MDI bonding strength of MPB wood was close to that of aspen under all these press time and temperature conditions. However, aspen appeared to be less sensitive to low press temperature in terms of bonding with MDI. Therefore, green lodgepole pine may be more suitable as a core furnish material than the MPB wood in the manufacture of OSB, where MDI resin is widely used as a core layer adhesive. Grey stage MPB wood may be more suitable as an OSB face furnish material. This hypothesis will be carefully tested in the 2nd fiscal year of this project.

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1 Objectives

The overall objective of this project is to address the BC government's mountain pine beetle strategy to recover the greatest value from dead timber before it burns or decays. The knowledge developed in this study will assist the B.C. forest industry to extract value from MPB-affected trees. The project will benefit B.C.'s primary (wood composite panel manufacturing) industry and thus contribute to the stability of forest-dependant communities.

The specific objectives in the 1st fiscal year were:

- To determine the chemical characteristics of grey stage MPB wood.
- To evaluate wettability of grey stage MPB wood.
- To evaluate the bondability of grey stage MPB wood with the commonly used wood adhesives.

2 Introduction

The manufacture of wood composite panel products has been raised as a potential use for lodgepole pine that has been killed by the mountain pine beetle. Currently a small amount of beetle-killed lodgepole pine finds its way into wood composite panel products. It would be desirable to be able to handle furnish that is up to 100% beetle-killed pine. Preliminary experimental work at Forintek indicates that the quality of OSB panels derived from 100% MPB wood, whether standing dead for 2 or 20 years, would not be acceptable in the marketplace because they showed greatly reduced water resistance properties and poor dimensional stability. These panels were made using the current aspen panel manufacturing conditions. They were not able to meet the CSA standard for OSB thickness swell after 24 hours water soak and MOR retention after the accelerating aging test. Past work also showed that linear expansion was a problem when particleboard and MDF were produced from dead lodgepole pine (Maloney, 1976).

From a panel product manufacturing point of view, MPB wood can be considered as a new type of wood material due to the following features:

1. Dry and brittle;
2. Blue stain
3. High permeability
4. High resin acid content
5. Lower bondability with water based adhesives

Recent work has indicated that the mountain pine beetle induces 3 times more resin acids production in infested trees than in normal pine trees (Chow, 2004). The effects of these resin acids on wood adhesives and which types of adhesives are most affected is unknown. These are important questions to be answered in order to use the MPB wood effectively for the manufacture of wood composite products. The MPB wood should be considered as a chemically modified wood and thus requires new approaches in order to deal with it. The chemical properties, such as pH, buffer capacity, wood extractive contents and heat stability, are fundamental characteristics of the MPB infested wood. Some of these properties will influence manufacturing conditions and the quality of the resulting products. Past studies on other wood

species have shown that wood pH and acid/base buffer capacity can have profound effects on the performance of wood adhesives, both kinetically and thermodynamically (Wang and Wan, 2004). Heat stability of wood (or thermal degradation) also has great effects on wood – adhesive bonding (Chow and Mukai, 1972).

It is currently possible to produce commercially acceptable composite panel products using a low percentage of MPB wood in the raw material mix. However, in order to increase the value recovery from MPB wood, a critical question for the OSB, MDF and particleboard mills in British Columbia is whether OSB, MDF and particleboard panel products meeting the normal quality standards can be made economically from 100% MPB wood. If not, how high a percentage of MPB wood can be used in the raw material mix? Science-based answers are needed for the composite panel industry in British Columbia.

This report summarises the work done in the first phase of a two phase project.

3 Staff

Martin W. Feng	Project Leader, Adhesive Specialist, Composites Group, Forintek
Robert M. Knudson	Team Leader, Senior Scientist, Composites Group, Forintek
Peter Ens	Senior Technologist, Composites Group, Forintek
Axel W. Andersen	Senior Technologist, Composites Group, Forintek
John Hoffman	Senior Technologist, Composites Group, Forintek
Guangbo He	Industrial Research Fellow, Composites Group, Forintek

4 Materials and Methods

Grey stage MPB logs and green lodgepole pine logs were used for the following tests.

4.1 pH and buffer capacity tests

pH and buffer capacity tests were performed by measuring the corresponding wood solutions, which were prepared using the following procedure:

25 g of wood flour (oven-dry based) was heated in 250 g tap water under reflux for 20 minutes and the cooked mixture was cooled and filtered using Whatman #114 filter paper under aspirator vacuum to obtain an aqueous wood extractive solution. Two replicates were done for each type of wood.

The pH values of the wood extractive solution were determined at room temperature by a pH meter calibrated with buffering solutions at pH values of 4.0 and 7.0.

Base buffer capacity was measured by titrating a 50 ml wood extractive solution with a 0.025 N NaOH aqueous solution to the titration end point of pH 7.0. The base buffer capacity (meq/100g wood) was calculated by the following equation:

$$\text{Base buffer capacity} = \text{Volume of NaOH titrant used for 100 g of wood} \times \text{Normality of NaOH}$$

Acid buffer capacity was measured by titrating a 50 ml wood extractive solution with a 0.025 N H₂SO₄ aqueous solution to reach the titration end point of pH 3.0. The acid buffer capacity was calculated as:

$$\begin{aligned} & \text{Acid buffer capacity (meq/100 g wood)} \\ & = \text{Volume of H}_2\text{SO}_4 \text{ titrant used for 100 g of wood} \times \text{Normality of H}_2\text{SO}_4 \end{aligned}$$

4.2 Water absorption and thickness swell tests

Twenty five blocks of sapwood and heartwood with dimensions 50×50×10 mm were cut from both MPB and green lodgepole pine logs for water absorption tests. The samples were oven-dried at 60°C for 24 hours and then subjected to ambient conditioning in the lab for one week prior to tests. The samples were horizontally submerged in tap water at various temperatures and pH over a 24-hour period. The water absorption and thickness swell were recorded at different soaking times.

At neutral pH, 20, 40 and 60 °C water temperatures were used. At pH 3 and 11, 20°C water temperature was used. Five specimens were tested for each experimental condition.

4.3 Lap shear test

Lap shear tests were conducted using the automated bond evaluation system (ABES). MPB sapwood, green lodgepole pine and aspen were tested. The following resins were used as wood binders in the tests.

Liquid phenolic resin:	LP02 from Hexion Specialty Chemicals, 55% solids content.
Liquid urea-formaldehyde resin:	CP251 from Hexion Specialty Chemicals, 65% solids content.
Powdered phenolic resin:	W800C from Hexion Specialty Chemicals.
Polymeric MDI resin:	ISOSET CX47 from Ashland Chemical.

The bonding strength was evaluated at different hot-pressing temperatures and pressing times. Fifteen replicates were done for each pressing condition.

4.4 Furnish production

Grey stage MPB logs and green lodgepole pine logs were harvested in the Quesnel area by West Fraser Timber and delivered to the Forintek Vancouver laboratory. The MPB logs had little barks and lots of check and the time since death was not known.

Some of these logs were then shipped to Alberta Research Council, debarked and converted to wood chips. The chips were sent to the Forintek's Quebec laboratory for hammer milling into particleboard furnish and refining into MDF fiber, followed by drying.

The remaining logs were conditioned in an outdoor water pool at Forintek for 48 hours and then converted to OSB strands in the pilot flaking facility of Carmanah Design and Manufacturing Inc. The resulting MPB wood strands (mix of sapwood and heartwood) and green pine strands were dried separately in a ventilated oven at 80°C for 18 hours to achieve about 2% moisture content.

5 Results and Discussion

5.1 pH and buffer capacity

Figure 1 shows the average pH values of aqueous solutions obtained from the extractions of MPB wood and green lodgepole pine. S stands for sapwood and H stands for heartwood. Results show that MPB wood, whether sapwood or heartwood, was the more acidic. The pH of the MPB sapwood was 4.45 in comparison to 4.60 for green pine sapwood. The pH of the MPB heartwood was 4.38 compared to 4.95 for green pine heartwood. It is noteworthy that the MPB heartwood, though appeared to be not affected by the blue stain, had undergone some pH changes from that of green pine.

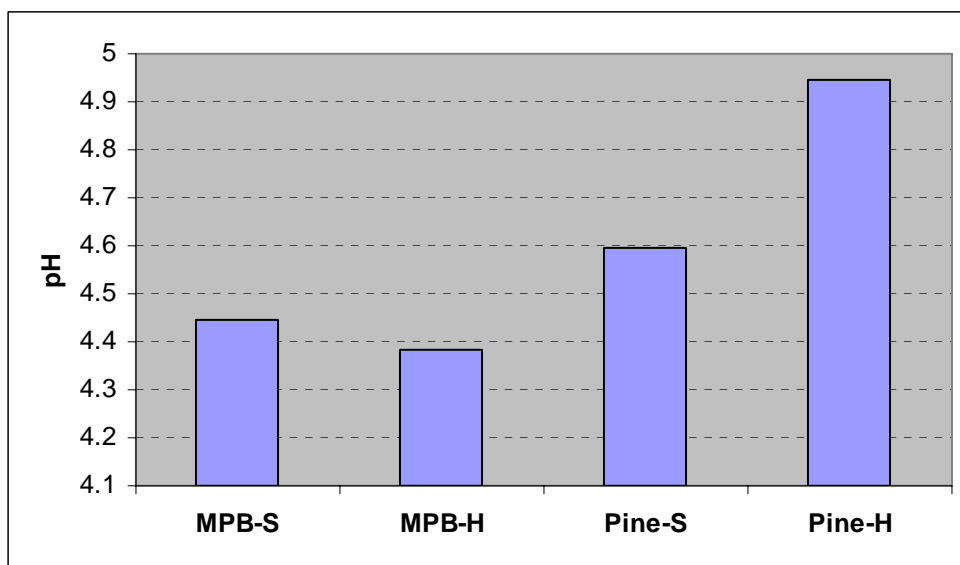


Figure 1 pH values of MPB wood and green lodgepole pine sapwood and heartwoods

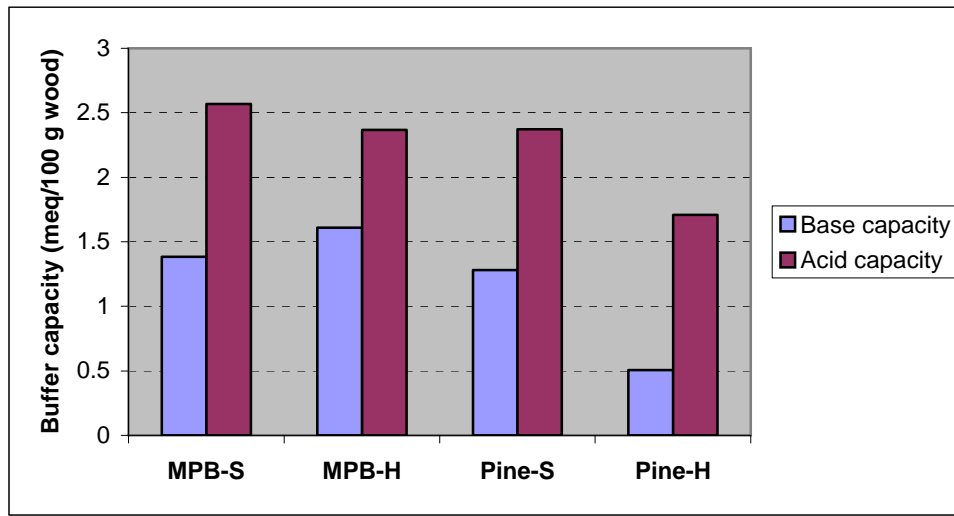


Figure 2 Buffer capacities of MPB wood and lodgepole pine sapwood and heartwoods

The buffer capacity is illustrated in Figure 2. It shows that both acid and base capacities of MPB wood were higher than those of green lodgepole pine if comparing sapwood and heartwood separately. This suggests that MPB wood (both sapwood and heartwood) may contain more extractives that affect pH and buffer capacity. When pH dependent resins (phenolic and amino type resins) are applied to manufacture wood panels from these wood species, the effects of MPB wood on resin curing could be more significant. To counter the higher buffer capacities, adjusting catalyst loading levels could be an option. The extractives in the MPB wood may even react with pMDI (polymeric methylene diphenyl diisocyanate) resins and hence profoundly affect the crosslinking of the resins.

5.2 Water absorption and thickness swell

Figure 3 shows the water absorption of MPB wood and lodgepole pine at 20°C and neutral pH over a 24-hour period. The MPB sapwood showed extremely fast and high water absorption - its moisture content increased from <10% to >80% in 0.5 hour and reached nearly 120% after 24 hours water soaking. Unlike the MPB sapwood, green lodgepole pine sapwood showed water absorption of about 20% after 0.5 hour water soak and approximately 55% after 24 hours, one quarter and one half that of the MPB sapwood respectively. The water absorption of sapwood was higher than that of heartwood in both MPB wood and green lodgepole pine. Interestingly, both MPB and green pine heartwoods had similar water absorption rate and percentages. It appeared that the beetle infestation and the length of time since tree death did not significantly affect the water absorption property of the MPB heartwood. Generally, the rate of water absorption of these woods was fast in the first several hours and slowed down thereafter.

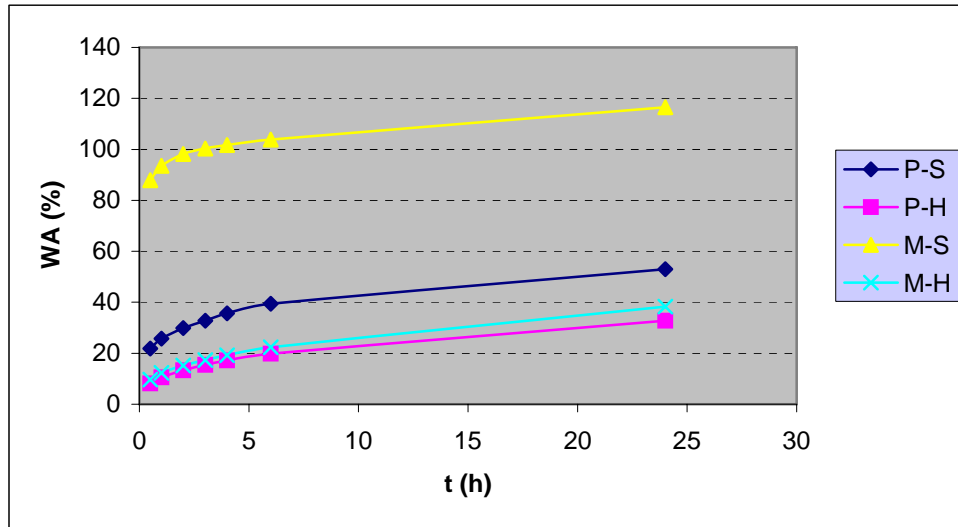


Figure 3 Water absorption at 20 °C and neutral pH

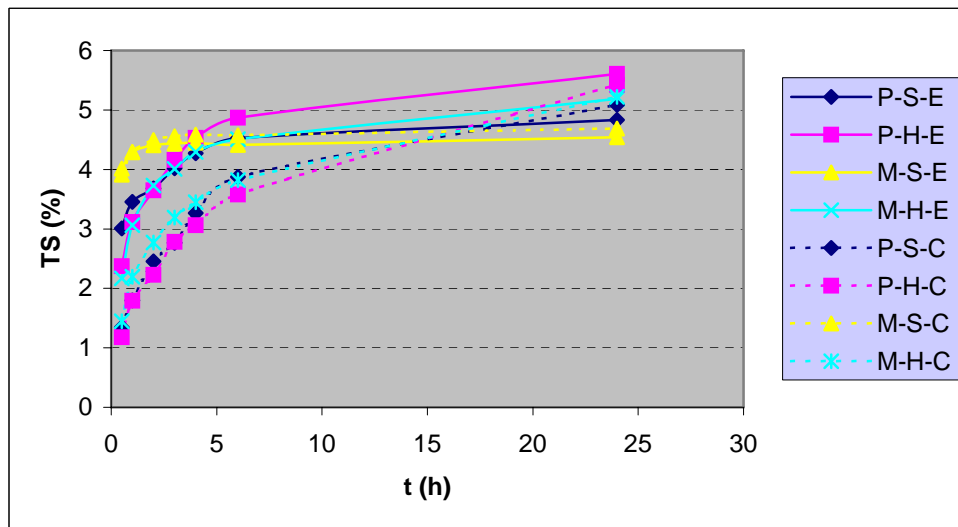


Figure 4 Thickness swell at 20 °C and neutral pH

The edge and center thickness swell (TS) at 20°C and neutral pH is shown in Figure 4. P and M stand for green pine and MPB wood respectively. S stands for sapwood and H stands for heartwood. C stands for centre and E stands for edge. Given the fact that the MPB sapwood showed rapid and high water absorption, it is surprising to see its thickness swell was lower than other types of wood. Furthermore, the thickness swell of the MPB sapwood almost reached to the maximum in the first two hours, and then remained unchanged for the rest of the soaking time. It is also noteworthy that the edge thickness swell

and center thickness swell of the MPB sapwood behaved very similarly in terms of rate and percentage. This is quite different in comparison with the thickness swell of the other woods, which suggests that the blue stained MPB sapwood had probably undergone profound changes.

The 24-hour thickness swell of sapwood was lower than that of heartwood for both MPB wood and green pine, although sapwood absorbed more water. The thickness swell of MPB heartwood appeared to be slightly lower than that of lodgepole pine heartwood. It was observed that the center thickness swell was generally lower than the edge thickness swell at the early stage of water submersion except for that of MPB sapwood, but the differences seemed to diminish over time, perhaps because of approaching equilibrium.

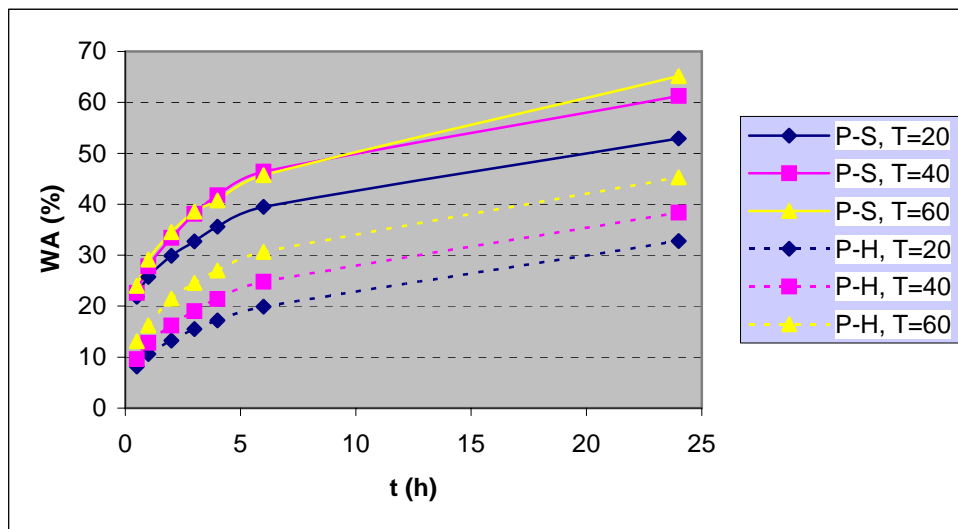


Figure 5 Effect of temperature on water absorption of lodgepole pine

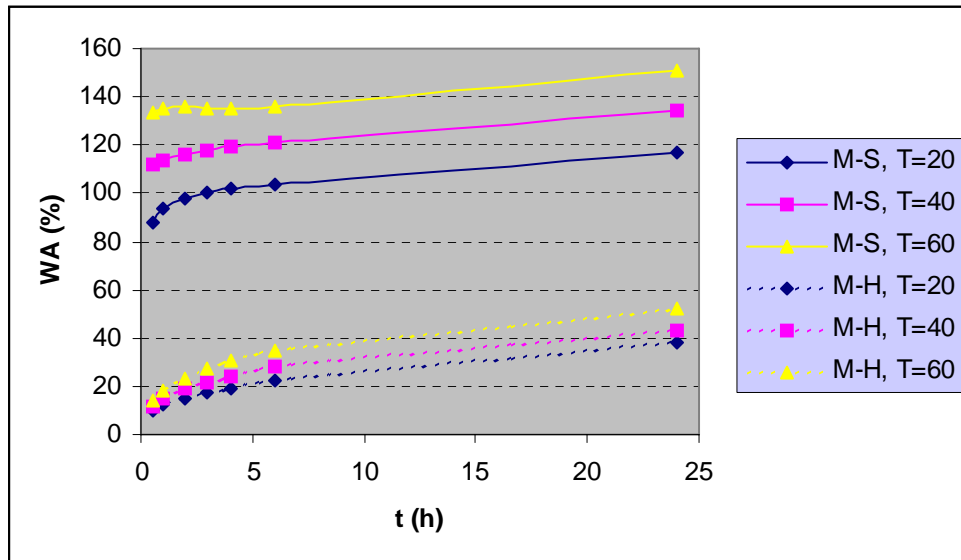


Figure 6 Effect of temperature on water absorption of MPB wood

Water temperature evidently affected water absorption of both green lodgepole pine and MPB wood, as shown in Figures 5 and 6 respectively. Figure 5 showed very similar water absorption patterns of green pine sapwood and heartwood at different temperatures but higher temperatures led to faster and more water absorption. In the case of MPB wood, however, the water temperature affected the MPB sapwood more than the MPB heartwood (see Figure 6). Water absorption rate and quantity increased with higher temperatures. Except for the MPB sapwood, the differential in water absorption due to different temperatures was not big in the first 0.5 hour but increased faster at higher temperatures in the first 5-6 hours. The water absorption curves of MPB sapwood in Figure 6 were parallel to one another for all submersion times.

The effects of temperature on center thickness swell of lodgepole pine and MPB wood are shown in Figures 7 and 8 respectively. The thickness swell reached equilibrium faster at higher temperatures, especially obvious for sapwood, although water absorption was still occurring at these points (see Figures 5 and 6). Most noteworthy is that the MPB sapwood always showed lower thickness swell than the MPB heartwood, green pine sapwood and green pine heartwood regardless of water temperatures.

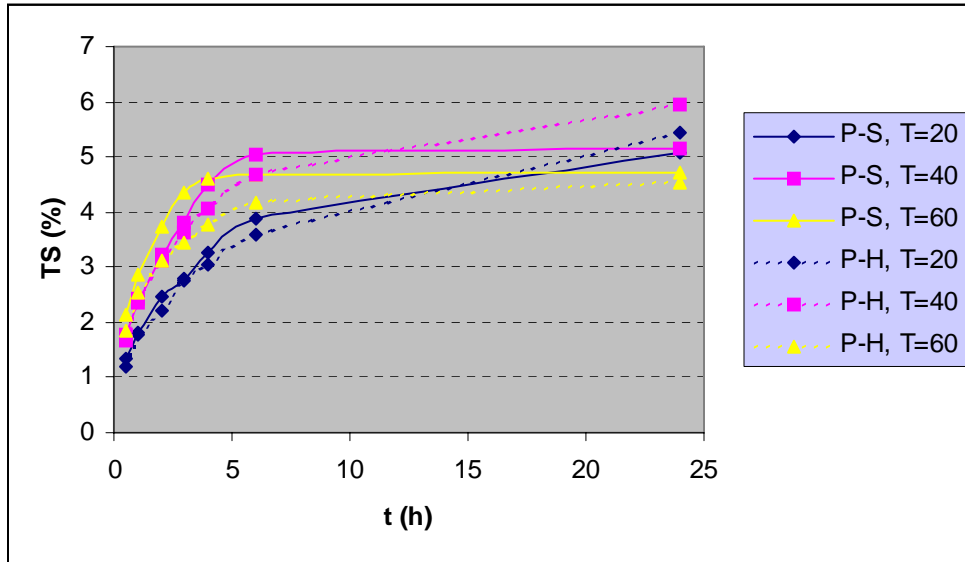


Figure 7 Effect of temperature on center thickness swell of lodgepole pine

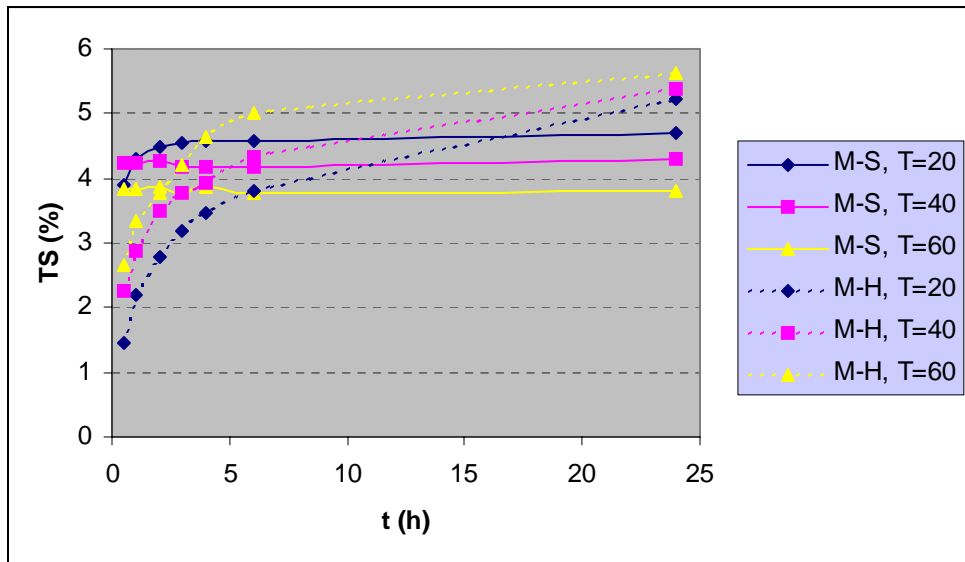


Figure 8 Effect of temperature on center thickness swell of MPB wood

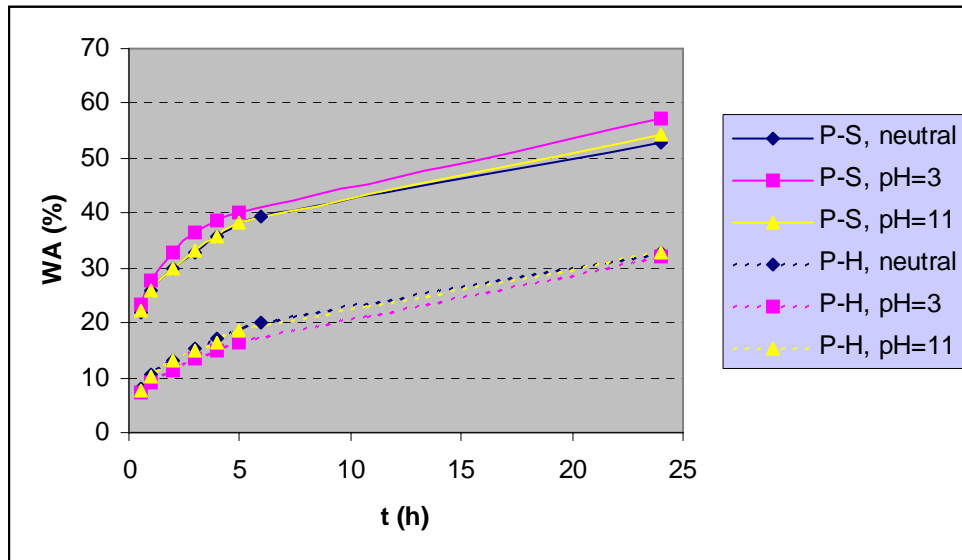


Figure 9 Effect of pH on water absorption of lodgepole pine at 20 °C

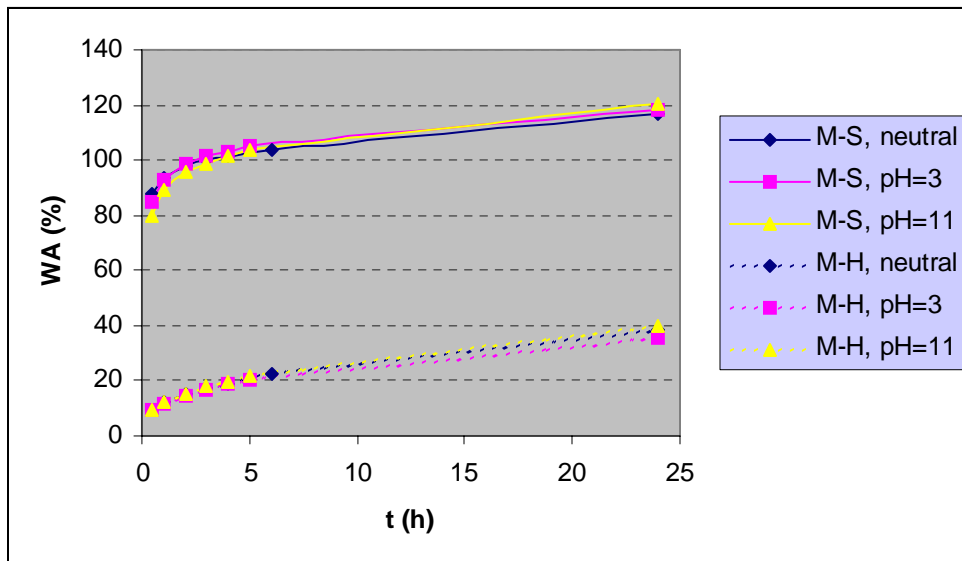


Figure 10 Effect of pH on water absorption of MPB wood at 20 °C

The effects of pH on water absorption of lodgepole pine and MPB wood are illustrated in Figure 9 and Figure 10 respectively. The wood samples were submerged in tap water (near neutral), acidic water (pH 3) and alkaline water (pH 11) and the water absorptions were measured over 24 hours. The results indicated mixed trends for both lodgepole pine and MPB wood. However, the effect of pH on water absorption is insignificant comparing to the temperature effect.

The effects of pH on thickness swell of lodgepole pine and MPB wood are illustrated in Figure 11 and Figure 12 respectively. Both acidic and alkaline water produced somewhat lower thickness swell than that of neutral water in both cases of lodgepole pine and MPB wood.

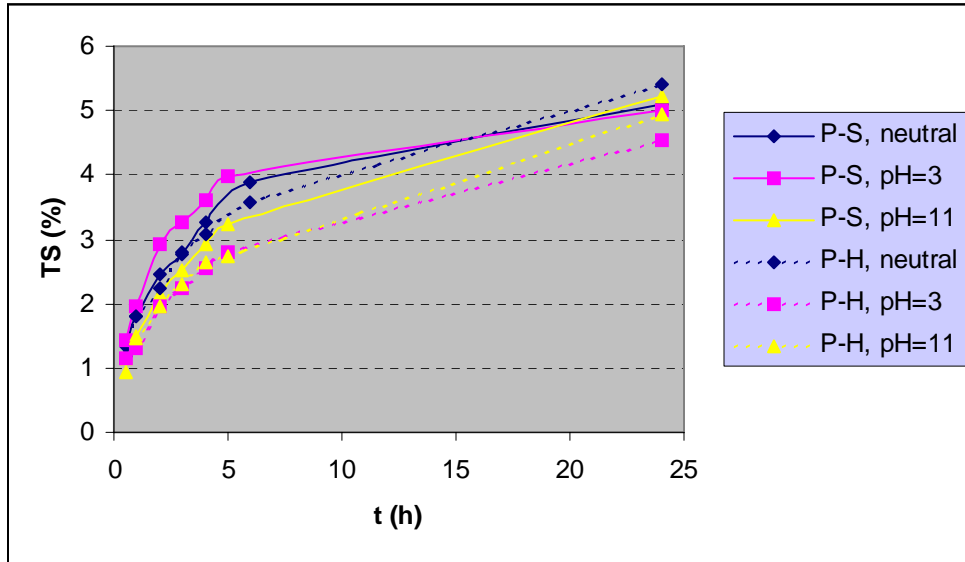


Figure 11 Effect of pH on center thickness swell of lodgepole pine at 20 °C

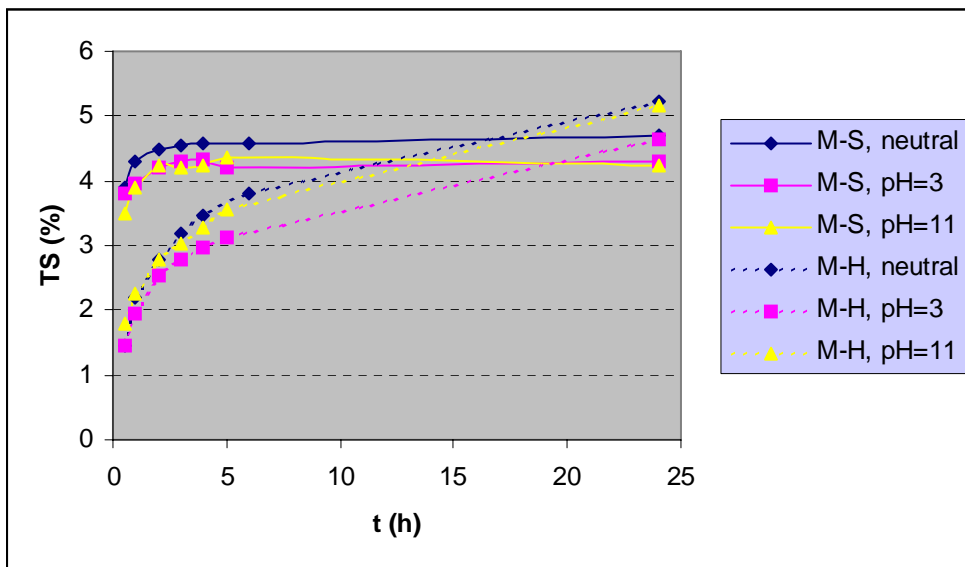


Figure 12 Effect of pH on center thickness swell of MPB wood at 20 °C

5.3 Bondability

The results of average lap shear strength of MPB wood (mostly blue stained), green lodgepole pine and aspen bonded with a liquid phenolic resin are summarized in Table 1. The bonding strength of MPB wood was comparable with that of aspen at the high press temperature of 200°C, while the bonding strength of green lodgepole pine appeared to be slightly lower than those of MPB wood and aspen. When the press temperature was decreased to 140°C, the bonding strength of all these woods decreased, especially in shorter press time (60 seconds) but less so in longer press time (180 seconds). Both the MPB wood and green pine showed lower bonding strength than aspen. This may have significant implications on the bonding quality of the core layer of panels. It is not clear, however, why aspen gave higher phenolic bonding strength.

The results of average lap shear strength of MPB wood (mostly blue stained), green lodgepole pine and aspen bonded with a powdered phenolic resin are summarized in Table 2. The overall findings are similar to those of liquid phenolic resin.

Table 1 *Lap shear strength of wood bonded with liquid phenolic resin*

Temperature & press time	MPB		Green lodgepole pine		Aspen	
	Resin solids (mg/cm ²)	Strength (MPa)	Resin solids (mg/cm ²)	Strength (MPa)	Resin solids (mg/cm ²)	Strength (MPa)
200 °C 60 seconds	0.43	2.25 (0.34)	0.46	2.06 (0.29)	0.37	2.07 (0.40)
200 °C 180 seconds	0.41	1.95 (0.35)	0.43	1.71 (0.34)	0.37	2.23 (0.38)
140 °C 60 seconds	0.43	0.99 (0.45)	0.49	0.79 (0.24)	0.36	1.42 (0.21)
140 °C 180 seconds	0.41	1.66 (0.32)	0.48	1.47 (0.33)	0.37	2.02 (0.31)

* Numbers in parentheses are standard deviations.

Table 2 *Lap shear strength of wood bonded with powdered phenolic resin*

Temperature & press time	MPB		Lodgepole pine		Aspen	
	Resin solids (mg/cm ²)	Strength (MPa)	Resin solids (mg/cm ²)	Strength (MPa)	Resin solids (mg/cm ²)	Strength (MPa)
200°C 60 seconds	0.63	1.96 (0.32)	0.63	2.35 (0.50)	0.62	2.51 (0.47)
200°C 180 seconds	0.65	2.05 (0.31)	0.64	2.14 (0.36)	0.61	2.11 (0.37)
140°C 60 seconds	0.63	0.95 (0.19)	0.65	0.64 (0.18)	0.62	1.17 (0.17)
140°C 180 seconds	0.61	1.49 (0.29)	0.64	1.42 (0.26)	0.60	1.78 (0.33)

* Numbers in parentheses are standard deviations.

Table 3 *Lap shear strength of wood bonded with liquid urea-formaldehyde resin*

Temperature & press time	MPB		Lodgepole pine		Aspen	
	Resin solids (mg/cm ²)	Strength (MPa)	Resin solids (mg/cm ²)	Strength (MPa)	Resin solids (mg/cm ²)	Strength (MPa)
180 °C 60 seconds	0.69	2.13 (0.33)	0.75	1.94 (0.38)	0.76	2.16 (0.24)
180 °C 180 seconds	0.73	2.08 (0.31)	0.73	2.09 (0.32)	0.75	2.52 (0.28)
120 °C 60 seconds	0.74	1.23 (0.24)	0.74	0.94 (0.22)	0.91	1.37 (0.17)
120 °C 180 seconds	0.74	1.64 (0.31)	0.82	1.07 (0.25)	0.74	1.82 (0.31)

* Numbers in parentheses are standard deviations.

The results of average lap shear strength of MPB wood, green lodgepole pine and aspen bonded with a liquid urea-formaldehyde resin are summarized in Table 3. The bonding strength of both MPB wood and green pine appeared to be comparable with that of aspen at the high press temperature of 180°C. When the press temperature was decreased to 120°C, the bonding strength of all woods decreased, especially in the shorter press time (60 seconds) but less dramatically in longer press time (180 seconds). Both the

MPB wood and green pine showed lower bonding strength than aspen at low temperature, particularly in longer press time (180 seconds).

The shear strength of wood bonded with a polymeric MDI resin is shown in Table 4. Unlike the water-based resins (UF and PF), the lap strength was poor in all cases, although the wood strands used in the lap shear tests were conditioned in a humidity chamber to a high moisture content of 22% prior to the bondability tests. These results were not consistent with the observations during wood composite panel manufacturing, which showed equal or better MDI bonding strength than phenolic or urea-formaldehyde resins. The probable cause of the problem is that the moisture on the thin wood strand surface evaporated rapidly upon contact with the hot press, thus giving insufficient moisture to initiate the MDI crosslinking reactions and hence resulting in poor shear strength.

At high temperature (200°C), green pine produced substantially higher MDI bonding strength while MPB wood and aspen gave lower and similar bonding strength. This was also the case at low press temperature (140°C), particularly in longer press time (180 seconds). The MDI bonding strength of MPB wood was close to that of aspen under all these press time and temperature conditions. However, aspen appeared to be less sensitive to low press temperature in terms of bonding with MDI.

Between grey stage MPB wood and green lodgepole pine, the lap shear data suggested that the green pine may be more suitable as a core furnish material than the MPB wood in the manufacture of OSB, where MDI resin is used as the core layer adhesive. In other words, grey stage MPB wood may be more suitable as an OSB face furnish material. This hypothesis will be carefully tested in the 2nd fiscal year of this project.

Table 4 *Lap shear strength of wood bonded with MDI resin*

Temperature & press time	MPB		Lodgepole pine		Aspen	
	Resin solids (mg/cm ²)	Strength (MPa)	Resin solids (mg/cm ²)	Strength (MPa)	Resin solids (mg/cm ²)	Strength (MPa)
200 °C 60 seconds	0.94	0.53 (0.27)	1.14	1.09 (0.49)	0.88	0.48 (0.16)
200 °C 180 seconds	1.15	0.46 (0.28)	0.79	1.17 (0.47)	1.11	0.44 (0.33)
140 °C 60 seconds	0.90	0.27 (0.15)	1.04	0.62 (0.18)	0.89	0.44 (0.42)
140 °C 180 seconds	1.06	0.43 (0.25)	1.16	1.10 (0.38)	1.08	0.35 (0.17)

* Numbers in parentheses are standard deviations.

6 Conclusions

Compared with green lodgepole pine, some chemical and physical properties of the grey stage MPB wood had changed. Based on the test results, the following conclusions are made:

1. The pH values of both the MPB heartwood and sapwood were lower and their acid and base buffer capacities were higher than those of the green lodgepole pine. As a result, the curing rate of pH sensitive adhesives such as UF and MUF may be affected.
2. The MPB sapwood showed extremely fast and high water absorption but its thickness swell was lower than those of the MPB heartwood, green pine sapwood and heartwood regardless of water temperatures.
3. The thickness swell of the MPB sapwood almost reached equilibrium in the first two hours of water soaking at 20°C.
4. The water absorption of sapwood was higher but the thickness swell was lower than that of heartwood in both MPB wood and green lodgepole pine. The rates of water absorption and thickness swell of these woods were fast in the first several hours and slowed down thereafter.
5. Both the MPB heartwood and the green pine heartwood behaved similarly in terms of water absorption rate and percentages. It appeared that the beetle infestation did not significantly affect the water absorption property of the MPB heartwood.
6. Edge thickness swell and center thickness swell of the MPB sapwood behaved very similarly in terms of the rates and percentages, which were quite different from those of the other woods and suggest that the blue stained MPB sapwood had probably undergone profound changes.
7. Higher temperatures led to faster and more water absorption. The water temperature affected the MPB sapwood more than the MPB heartwood.
8. Thickness swell reached equilibrium faster at higher temperatures.
9. Water pH had little influence on water absorption but affected thickness swell. The thickness swell of both MPB wood and lodgepole pine decreased under both acidic and alkaline conditions.
10. The bonding strength of MPB and green lodgepole pine with liquid PF, powdered PF and liquid UF were generally comparable to that of aspen at high press temperatures. Both the MPB wood and green pine showed lower bonding strength than aspen at low press temperatures. This may have significant implications on the bonding quality of the core layer of panels.
11. At high temperature (200°C), green pine produced substantially higher MDI bonding strength while MPB wood and aspen gave lower and similar bonding strength. This was also the case at low press temperature (140°C), particularly in longer press time. The MDI bonding strength of MPB wood was close to that of aspen under all these press time and temperature conditions. However, aspen appeared to be less sensitive to low press temperature in terms of bonding with MDI. Therefore, green

lodgepole pine may be more suitable as a core furnish material than the MPB wood in the manufacture of OSB, where MDI resin is widely used as a core layer adhesive. Grey stage MPB wood may be more suitable as an OSB face furnish material.

7 References

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