WOOD-THERMOPLASTIC COMPOSITES MANUFACTURED USING BEETLE-KILLED SPRUCE FROM ALASKA'S KENAI PENINSULA

By

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To the Faculty of Washington State University:

The members of the Committee appointed to examine the thesis of NELS ROYAL PETERSON find it satisfactory and recommend that it be accepted.

Chair

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WOOD-PLASTIC COMPOSITES MANUFACTURED USING BEETLE-KILLED SPRUCE FROM ALASKA'S KENAI PENINSULA

Abstract

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Over the last 20 years the spruce bark-beetle outbreak has infested an estimated 0.6 million ha (1.5 million acres) of spruce trees within Alaska's Kenai Peninsula. Of these acres 95% mortality is common and accounts for an estimated timber lose of 2 billion board feet. As the bark-beetle infestation continues to spread and trees already dead continue to deteriorate, traditional wood products no longer become a viable option. New uses and products are needed to make logging viable and one possible product is wood-thermoplastic composites (WPC) that use wood flour as a feedstock and are less sensitive to the integrity of the solid wood source. The objective of this project was to characterize the mechanical properties of the solid wood source based on the three deterioration levels; Live, Partially Deteriorated, and Highly Deteriorated and to evaluate the feasibility of manufacturing WPC using wood flour from the three deterioration levels. Based on small clear specimen testing; site location, tree height location and deterioration level was evaluated and statistically differed. All but deterioration level was combined and used to develop a mixture model for manufacturing of WPC. Seventeen runs were developed using varying proportions of wood flour from the three deterioration levels and compared against a run of pine wood flour, which served as the control for this study. WPC deck boards were tested in static bending, moisture effect, high temperature effect, low temperature effect, and freeze-thaw effect and compared against one another. In all cases the seventeen runs exceeded the MOE and MOR values of the pine control run, averaging 14.8% and 9.1% higher respectively. Mechanical and physical properties of the WPC manufactured from beetle-killed spruce were slightly better than properties of WPC composed of pine wood flour.

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CHAPTER ONE

INTRODUCTION

Introduction

The spruce bark-beetle (*Dendroctonus rufipennis* (Kirby)) outbreak has infested millions of acres across southern Alaska, with the Kenai Peninsula being at the center of the infestation. Since the bark-beetle infestation began more than 20 years ago, an estimated 0.6 million ha (1.5 million acres) of White spruce (*Picea glauca* (Moench) Voss) trees have been affected, with mortality rates up to 95% among those affected acres. The timber lose from this infestation has been estimated to exceed 2 billion board feet (Kenai Peninsula Spruce Bark Beetle Task Force, 1998).

As the bark-beetle infestation continues to spread and trees already dead continue to deteriorate, traditional wood products no longer become a viable option. Rather, alternative solutions need to be examined as this problem is cyclical, with historical records showing bark-beetle outbreaks occurring every 20 to 60 years (Holsten 1990, Berg 2003). Wood-thermoplastic composites (WPC) are one potential alternative, as WPC use wood flour as a feedstock and are less sensitive to the integrity of the solid wood source. WPC have gained in popularity recently and today represent a rapidly gaining market share, primarily as a substitute for wood decking (Wolcott and Englund 1999, Clemons 2002). In 2004, wood plastic composites represented 19% of the 4 billion decking market ($790 million), but this market share is anticipated to reach 42% by 2010, accounting for $2.6 billion of the $6.3 billion market. (Hunter 2006)

Objectives

The goal of this study was to characterize the mechanical properties of the beetle-killed spruce, considering deterioration and to evaluate WPC manufactured from beetle-killed spruce, regardless of the deterioration present in the wood and compare to a typical industry standard. In order to attain this goal the following objectives were established:

1. Determine the mechanical properties of spruce from the Kenai Peninsula based on small clear specimen testing. Two classes of spruce were examined for this study: live trees and partially deteriorated.
2. Evaluate the feasibility of manufacturing WPC using beetle-killed spruce from the Kenai Peninsula.
3. Test the mechanical properties of the WPC test specimens and look at the influence of varying proportions of wood flour from the different deterioration levels.

Background

As the infestation intensified through the 1990's and peaked in 1996, studies were conducted to evaluate how deterioration affected the dead trees and if severe how soon harvest was needed to maintain value within the trees for traditional wood products. Three such studies were conducted, one on the lumber and veneer recovery of standing dead timber (Lowell and Willits 1998, Lowell 2001) and another on pulp and paper quality (Scott et. al. 1996) to determine the effect that deterioration caused on each respectful timber product. In these three cases, logs were classified into four deterioration levels, based on visual observation in the field, ranging from live or recently dead trees (case 1) to trees of high deterioration (case 4). In the case of lumber and veneer recovery, log value for solid wood products decreased dramatically following the death of the tree (Lowell and Willits 1998, Lowell 2001), but did not statically differ between the deteriorated cases of 2, 3, and 4. In the study of lumber recovery it was found that one percent of existing defect translated into a 0.3 percent decrease in cubic recovery and a loss of $1.92 per hundred cubic feet of gross log volume (Lowell and Willits 1998). This was closely replicated in the study on veneer recovery, where one percent of existing defect translated into 0.4 percent decrease in cubic recovery and a loss of $1.96 per hundred cubic feet of gross log volume (Lowell 2001). In both of these studies it was recommended that only two deterioration levels be considered for future studies: one for live and infested trees and a second for dead trees. However, in the case of pulp and paper quality, the log value remained relatively neutral through the four deterioration cases (Scott et. al. 1996). This could be attributed to the reduction of solid wood into a fiber, and thus a dispersion of deterioration throughout the final product.

Another factor is the two types of fungal decay generally associated with wood. White-rot fungi are preferential degraders of cellulose, hemicelluloses and lignin components of wood (Blanchette et al. 1990, Zabel and Morrell 1992). Brown-rot generally to degrade polysaccharide components of wood and are responsible for extensive depolymerization of cellulose early in the decay process (Blanchette 2000). WPC products may provide an opportunity for use of beetle-killed spruce, much the same way that the pulp and paper products can.

In a study by Willits and Sampson (1988), it was found that fire-killed White spruce lost significant value between live trees and dead trees because of secondary weathering effects, mainly checking. A loss of 25 to 34 percent in dollars per hundred cubic feet was reported and it was recommended that immediate harvest of fire-killed timber be performed to maintain value for traditional lumber products. This recommendation was also encouraged by Schulz (2003) who studied downed woody debris within the Kenai Peninsula, finding that dead trees became dry, brittle and were more susceptible to breakage during harvest, further reducing salvageable material.

Structure of Thesis

The following thesis is broken into four chapters including this introduction and the final chapter of conclusions. Chapter two addresses small clear specimen tests performed on beetle-killed spruce as per ASTM 143-94 (2000). Chapter three discusses wood-plastic composites (WPC) manufacture and testing based on ASTM D7032-05. Chapter four summarizes the findings of this thesis and provides recommendations based on these findings. A paper co-authored by the chair, Vikram Yadama and Nels R. Peterson is provided in the Appendix A. This paper discusses the chemical and physical properties of highly deteriorated beetle-killed wood flour and WPC production from corresponding wood flour. Mechanical properties of the WPC were tested and are also discussed in this paper. Along with this co-authored paper additional figures, raw data and statistical analysis are provided in the appendices.

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CHAPTER TWO

CLEAR SPECIMEN TESTING OF BEETLE-KILLED SPRUCE FROM ALASKA'S KENAI PENINSULA

Introduction

Beetle-killed spruce, irrespective of degree of deterioration, could potentially be used as a feedstock for wood-plastic composite (WPC) production. Generally, WPC formulation consists of over 50% wood flour, thus its characteristics play a critical role in determining the WPC processing parameters as well as final product properties. As wood quality in a tree influences the preparation and quality of the wood flour, it would help to understand the integrity of the wood source in its original form. This study examines small clear specimens according to ASTM D143-94 (2000) of white spruce from Kenai Peninsula to quantify the effects of deterioration and weathering on solid wood properties. Characterization of live, partially deteriorated, and highly deteriorated trees was based on associated studies by Lowell and Willits (1998), Lowell (2001), and Scott et al. (1996). In these previous studies a four-level visual classification was established based on tree appearance using the following guidelines:

- Class 1 – Live trees or recently attacked (infested) trees that still have green needles
- Class 2 – Fading or brown-red needles, fine twigs remaining, crown may have a reddish cast.
- Class 3 – No fine twigs remaining, tree takes on a gray coloration, 90% or more of the bark is remaining and tight.

- Class 4 – Less than 90% of bark is remaining, bark is sloughing, may be obvious weather checks, gray, weathered coloration.

In these studies Class 1 represented a live or recently infested tree and Class 4 represented a highly deteriorated tree, with Class 2 and Class 3 ranging in between. In Lowell and Willits (1998) and Lowell (2001) it was recommended that only two deterioration levels be considered for future studies: one for live and infested trees and a second for dead trees. In this study three deteriorated levels were investigated; live, partially deteriorated, and highly deteriorated.

As for small clear specimen testing, only live and partially deteriorated trees were evaluated, as the highly deteriorated logs could not cut into clear specimens because of the amount of decay present, prohibiting any further preparation of small clear test specimens according to ASTM D143-94 (2000). This study is part of a project whose goal is to characterize the wood flour generated from beetle-killed spruce trees and investigate its influence on wood-plastic composite's physical and mechanical properties. Similar to results found by Scott et al. (1996) on minimal influence of deterioration on quality of pulp and paper produced, the hypothesis of this study is that deterioration, although could significantly affect the solid wood properties, will have minimum impact on properties when used as wood flour for wood-plastic composite production. This specific paper deals with influence of deterioration level on properties of small clear solid wood specimens, which has never been characterized.

Objectives

The objective of this study was to correlate the clear specimen properties from beetle-killed spruce from Kenai Peninsula to tree deterioration level and location along the tree height. Following tasks were accomplished to meet this objective:

1. Investigate physical (density) and mechanical (flexure and compression parallel to grain) properties of small clear specimens according to ASTM D143-94 (2000) standards.

2. Analyze the effect of deterioration level and specimen location with respect to tree height on small clear specimen properties.

Background

Previous research (Lowell 2001, Lowell and Willits 1998, Lowell 1993) focused on establishing a timeline of harvest in order to maintain valuable traditional timber products from the deteriorated trees. They also looked at how deterioration affected the final timber products, both mechanically and financially. No research has looked at quantifying how the deterioration affects the physical and mechanical properties of the wood within the infected tree. Mechanical properties of sound quality White spruce (*Picea glauca* (Moench)) as listed in the *Wood Handbook* (1999) is given Table 2.1.

Table 2.1. Mechanical properties of White spruce (*Picea glauca* (Moench)) as listed in the *Wood Handbook* (1999).

White Spruce	Moisture Content	Specific Gravity	MOE[1] (GPa)	MOR[2] (MPa)	Compression Parallel to Grain (MPa)
	Green	0.37	7.4	39	17.7
	12%	0.40	9.2	68	37.7

[1] MOE = Modulus of Elasticity
[2] MOR = Modulus of Rupture

Methods and Materials

Sample Preparation

Two classes of spruce trees were evaluated for this study: live trees (control) and partially deteriorated trees. As stated above, the highly deteriorated trees were not fit for clear specimen preparation because of substantial deterioration incurred since infestation. Test trees were obtained from two sites, National Forest Service land and State Forest land (referred to from here on as Site 1 and Site 2). Five live and five partially deteriorated trees (based on visual characterization described by Lowell and Willits (1998)), were sampled from each of the two sites. Each tree had three 2.4 m (8 ft) bolts cut from it: one at the butt end, one from below the 101.6mm (4 in) top, and the third equidistant between those two. Logs were then transported to a sawmilling facility on the Kenai Peninsula for further processing, where each 2.4 m (8 ft) bolt was divided into two 1.2 m (4 ft) bolts. One bolt was sawn into rough clear specimen samples and the other was chipped and hammermilled into wood flour for WPC manufacturing. Each bolt was labeled according to the tree and location within the tree: 1-5 Forest Service (Live), 31-35 State Forest (Live), 11-15 Forest Service (Partially Deteriorated), 22-26 State Forest (Partially Deteriorated), and A-F representing the butt through the top of each tree, with (A) being the butt end and (F) being the top of the tree (Figure 2.1).

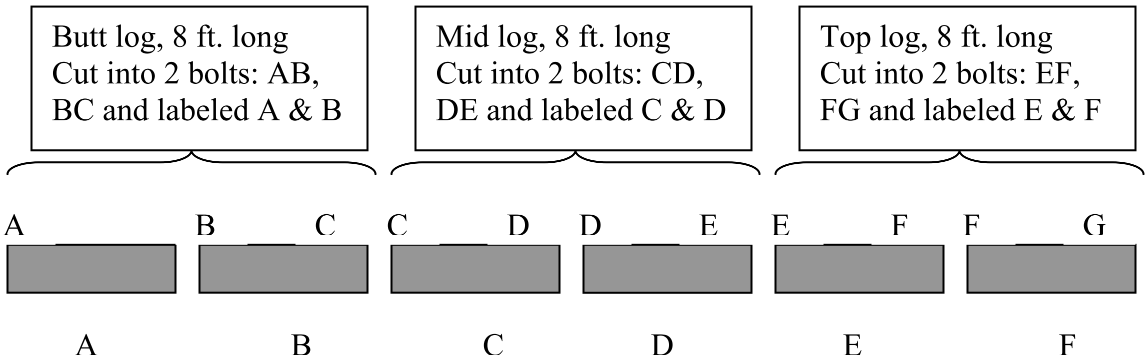

Figure 2.1. Preparation of clear specimens within tree.

The 1.2192 m (4 ft) bolts were cut into rough small clear specimens as shown in Figure 2.2 based on ASTM D143-94 standards (2000). Primary methods provide for specimens of 5.08cm x 5.08cm (2"x2"), whereas secondary methods provide 2.54cm x 2.54cm (1"x1"). Primary has the advantage of taking into account more growth rings and thus less influence of earlywood and latewood is seen (ASTM D 143-94 2000). Secondary methods are becoming more common with the increasing incidence of smaller second growth tress being used in wood products. Specimens were planed to the required final dimensions as per the standard. The resulting sizes were:

- Static Bending: 5.08cm x 5.08cm x 76.2cm (2"x 2"x 30") primary and 2.54cm x 2.54cm x 40.64cm (1"x 1"x 16") secondary
- Compression parallel to grain: 5.08cm x 5.08cm x 20.32cm (2"x 2"x 8") primary and 2.54cm x 2.54cm x 10.16cm (1"x 1"x 4") secondary
- Density: 5.08cm x 5.08cm x5.08cm (2"x 2"x 2") primary and 2.54cm x 2.54cm x 2.54cm (1"x 1"x 1") secondary

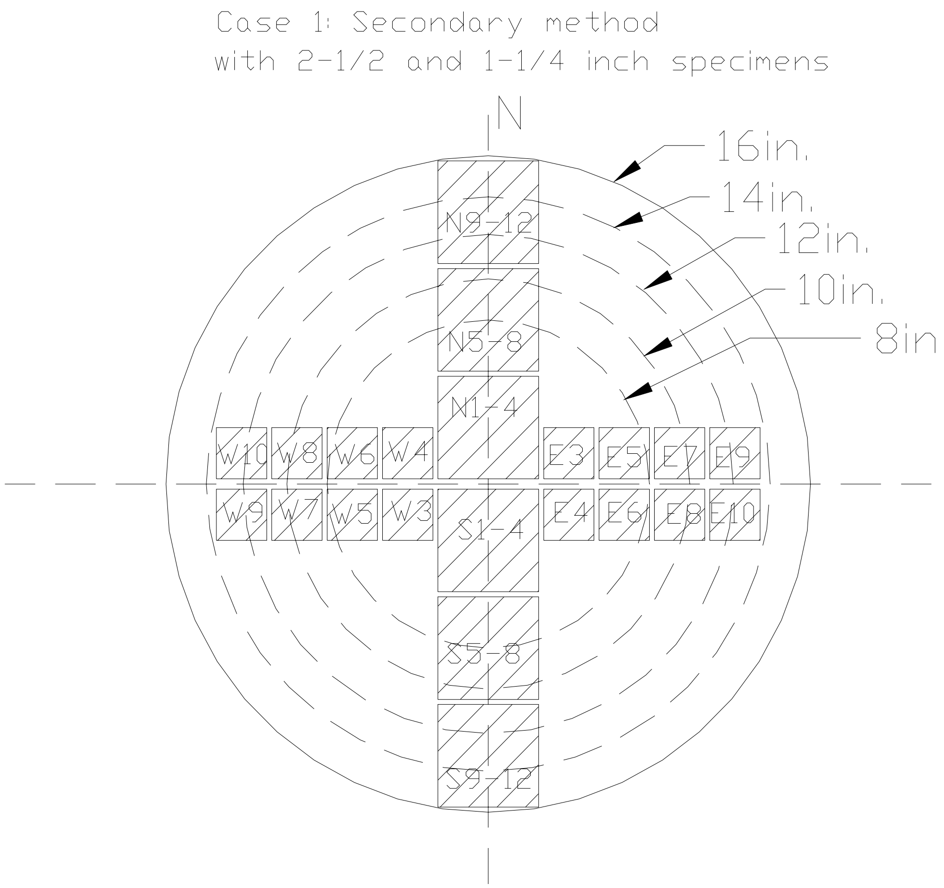

Figure 2.2. ASTM D143-94 (2000) cutting procedure for small clear specimen testing.

All specimens were conditioned at 18.3°C (65°F) temperature and 60% relative humidity to equilibrate to approximately 12% moisture content before being tested in flexure and compression.

Testing

Testing for all clear specimens was performed following ASTM D143-94 (2000) standards. According to ASTM D143-94 (2000), two sizes of clear specimens are viable for accurate results, primary and secondary. Primary size has the advantage over secondary because it takes into account a larger number of growth rings and is thus less susceptible to earlywood and latewood differences within the clear test specimen (ASTM

2000). Secondary size takes into account the use of smaller second growth trees that are becoming increasing popular within the wood products industry (ASTM 2000). In many cases it may be difficult or impossible to obtain primary size samples from the sample logs, as is the case in this study. In this study both primary and secondary size specimens were prepared and tested. This was to assure that results were consistent and comparable, regardless of the original tree size or condition. The mechanical properties analyzed for this study were: Modulus of Elasticity (MOE), Modulus of Rupture (MOR), Young's modulus in compression and ultimate compression strength.

Results and Discussion

Primary and secondary samples were tested and analyzed, but due to diameter of the logs being smaller fewer primary samples were produced and tested. Analysis of the results indicated that primary and secondary specimens both yielded similar values and trends in density variations as well as flexure and compression properties. Only secondary data will be presented in this chapter, as it more appropriately represents the nature of small-diameter trees from which the clear specimens originated. The data of the secondary samples also had lower coefficient of variation (COV), largely due to the greater sample size. Density was also statistically measured as a covariant throughout the tests and showed that it had no affect on the values of the properties, with an average P-value of 0.5499.

Appendix A summarizes data collected regarding the trees sampled to obtain test specimens from both the sites (includes information on tree diameter and approximate number of growth rings). Significant mechanical differences existed between the two sites from which small clear specimens were taken and this could be due to the differences in the growing climates of site one and site two. This is evident by looking at the average age of the trees from site one and site two, which averaged 91 and 149 years old respectively, but retained similar butt and top diameters (Appendix A). Results of the three tests performed on the clear test specimens are shown in Tables 2.2, 2.3, and 2.4.

Table 2.2. Summary of static bending properties.

Tree Location	Number of Samples	Avg. MOE[1]	COV[3]	Avg. MOR[2]	COV[3]	Strain at Break	COV[3]
		GPa (psi)	(%)	MPa (psi)	(%)		(%)
Site One (Live)	41	6.75 (978,760)	24.4	36.3 (5,270)	20.3	0.008	22.5
Butt	25	6.84 (992,100)	24.7	36.0 (5,220)	22.9	0.008	23.6
Middle	15	6.55 (949,300)	25.5	35.9 (5,210)	14.5	0.008	21.3
Top	1	7.47 (1,083,400)	-	49.3 (7,150)	-	0.010	-
Site Two (Live)	76	9.33 (1,353,550)	19.3	44.6 (6,470)	12.0	0.008	20.4
Butt	24	8.63 (1,251,900)	21.3	43.9 (6,370)	15.5	0.008	19.1
Middle	28	9.35 (1,356,100)	18.6	44.8 (6,500)	12.1	0.008	21.3
Top	24	10.01 (1,452,100)	16.2	45.0 (6,530)	7.7	0.007	20.7
Site One (Partially Deteriorated)	56	6.26 (908,420)	25.2	35.7 (5,180)	20.8	0.008	20.4
Butt	34	5.68 (824,400)	23.9	34.5 (5,010)	22.6	0.009	21.0
Middle	18	7.17 (1,039,500)	22.3	38.7 (5,610)	16.4	0.008	18.1
Top	4	7.13 (1,033,400)	15.3	32.1 (4,660)	17.0	0.007	22.1
Site Two (Partially Deteriorated)	79	8.48 (1,229,530)	23.5	41.7 (6,050)	18.3	0.008	21.8
Butt	35	7.99 (1,158,600)	28.8	40.5 (5,870)	21.8	0.008	28.8
Middle	23	8.60 (1,248,000)	20.2	41.2 (5,980)	17.7	0.008	17.1
Top	21	9.15 (1,327,500)	16.4	44.0 (6,380)	12.1	0.007	19.5

[1] MOE = Modulus of Elasticity
[2] MOR = Modulus of Rupture
[3] COV = Coefficient of Variation

Compared to the published data provided in the Table 2.1, the MOE and MOR values for Site One were not significantly different (0.74% lower), but Site Two properties were 27% greater. Between live and partially deteriorated an average decrease of 4.46% was observed for Site One and 7.81% for Site Two. The COV also increased with deterioration for both sites, varying from 0.65% for Site One to 5.25% for Site Two.

Table 2.3. Summary of compression parallel to grain mechanical properties.

Tree Location	Number of Samples	Avg. Young's Modulus[1]	Coefficient of Variation	Avg. UCS[2]	Coefficient of Variation
		GPa (psi)	(%)	MPa (psi)	(%)
Site One (Live)	45	7.33 (1,063,720)	36.1	28.5 (4,140)	19.4
Butt	27	6.94 (1,006,600)	34.7	28.3 (4,100)	21.5
Middle	17	8.13 (1,179,200)	35.5	28.7 (4,160)	17.1
Top	1	4.35 (631,000)	-	31.6 (4,580)	-
Site Two (Live)	93	11.47 (1,663,780)	36.4	35.4 (5,140)	15.6
Butt	32	9.45 (1,370,600)	22.3	34.7 (5,030)	18.6
Middle	33	9.95 (1,443,100)	26.1	34.9 (5,060)	15.9
Top	28	10.16 (1,473,600)	22.8	36.9 (5,350)	10.9
Site One (Partially Deteriorated)	61	6.66 (966,540)	50.4	27.9 (4,050)	21.0
Butt	38	5.34 (774,500)	43.2	25.2 (3,650)	20.5
Middle	19	7.97 (1,156,000)	33.6	32.6 (4,720)	12.9
Top	4	7.91 (1,147,200)	5.8	31.5 (4,560)	5.6
Site Two (Partially Deteriorated)	96	11.21 (1,626,480)	47.5	33.6 (4,870)	17.2
Butt	44	8.26 (1,198,000)	37.0	32.7 (4,740)	18.9
Middle	27	9.59 (1,391,000)	28.6	33.4 (4,840)	14.0
Top	25	10.14 (1,470,700)	28.9	35.4 (5,130)	16.6

[1] Avg. Young's Modulus = Young's Modulus in compression parallel to grain
[2] UCS = Ultimate Compressive Strength

Generally the top portion of the tree exhibited the highest values for MOE and MOR. Comparing properties of small clear specimens from live trees to those published in the Wood Handbook, compression parallel to grain strength was 15.3% lower for 12% moisture content. As for the effects of deterioration, compression parallel to grain Young's modulus decreased by an average of 9.11% for Site One and 2.27% for Site Two; ultimate compressive strength also decreased with deterioration for both Site One and Site Two by an average of 2.11% and 5.08% respectively. Practically speaking these differences may not be significantly different; however they result in lowering of lumber quality which devalues the wood to the extent that it is not economically viable to harvest these trees for lumber or veneer production.

Density results showed that Site Two varied little with deterioration, only a decrease of 0.75%, whereas Site One exhibited a decrease of 4.38%. A summary of density variation within the sample trees from both sites is given in Appendix B. Mechanical properties were also statistically analyzed, considering density as a covariate. Results indicated that density effects were not significant (P-value = 0.5499), therefore in further analysis density was not taken into consideration as a covariate.

The influence of three factors, namely site location, deterioration level, and tree height, on clear specimen properties and their variation was examined and analyzed statistically using Systat 11® (Systat 11® 2002).

Site Location

Site was examined to determine if trees from different regions of the Kenai Peninsula exhibited different mechanical properties due to differences in growing conditions. In comparing the two sites, (Site One, Site Two) the three tree height locations (Butt, Middle, Top) were combined but the deterioration levels (Live and Partially Deteriorated) were kept separate. Statistically the two locations yielded significantly different properties (P-value < 0.001), as evident in the four comparison graphs in Figure 2.3. Based on the average of the five mechanical properties, Site Two had 18.8% higher values than Site One, as shown in Figure 2.3. Density showed the lowest percent difference between the two sites with only a 4.3% difference. Site One showed no statistically significant difference between live and partially deteriorated specimens as for modulus of elasticity, modulus of rupture and ultimate compression strength (P-value = 0.147, 0.704, 0.562 respectively). Only density showed a statistically similar value in Site Two between live and partially deteriorated (P-value = 0.592). All of the comparison graphs show that Site One exhibited lower mechanical properties than Site Two, which can be attributed to differences in localized growing conditions. Although the two sites statistically differed, the likelihood that the trees from any two sites would be segregated upon receiving at a composite processing facility, such as WPC extrusion plant, is low and thus it is more practical to assume that the wood feedstock would be composed of trees from all locations within the Kenai Peninsula. Thus, for further analyses of effects of tree height and deterioration level on properties, the two sites were combined.

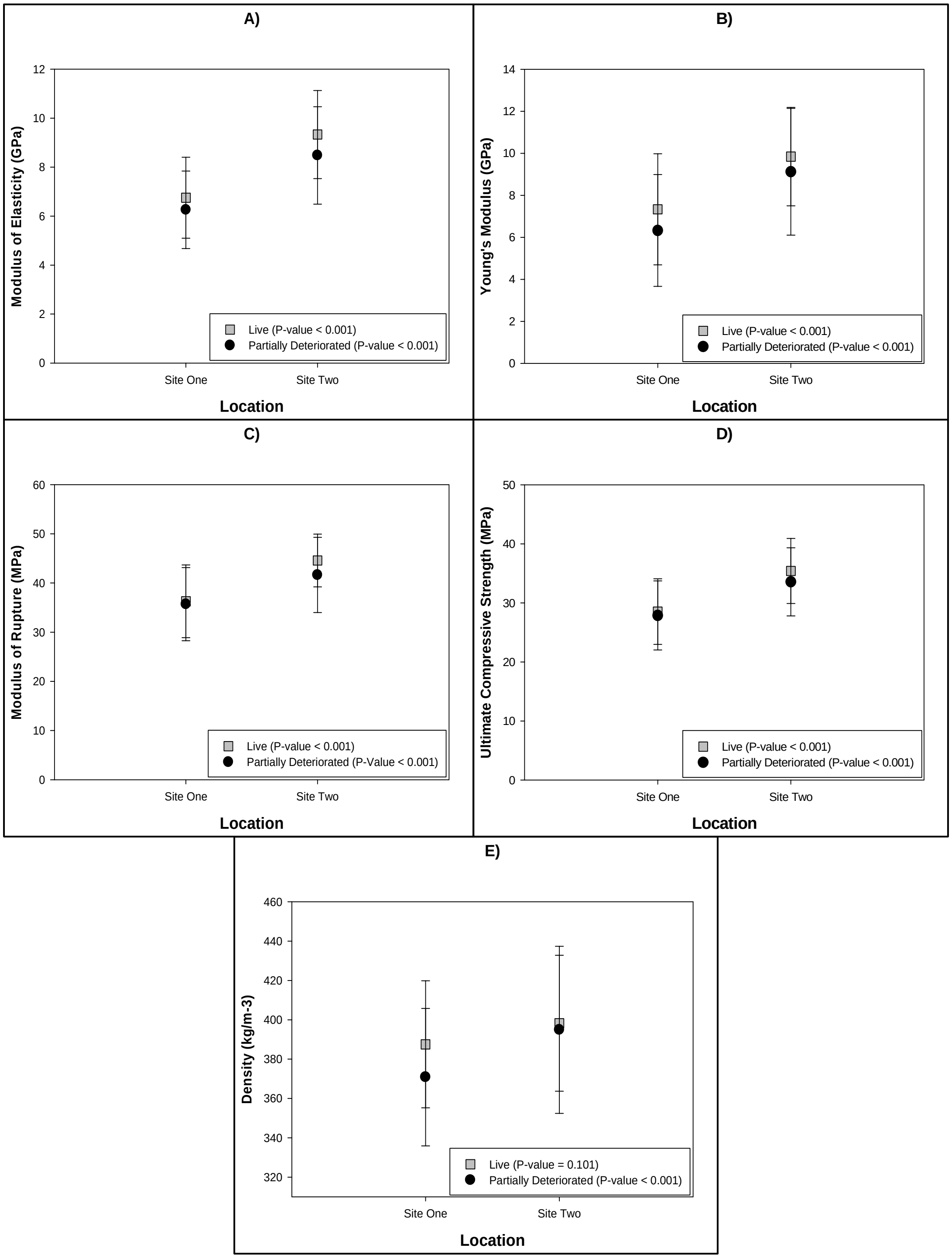

Figure 2.3. Comparison of Site One vs. Site Two: A) Modulus of Elasticity, B) Compression Modulus, C) Modulus of Rupture, D) Ultimate Compression Strength, E) Density, indicating that the two sites are statistically different. (Error Bars are Standard Deviation)

Log Location

Log location within a tree was examined to see if mechanical differences existed between samples taken at the three locations within a tree; butt, middle, and top. In all cases except density the values increased from butt to top for both live and partially deteriorated. The top logs yielded properties that were statistically different (P-value<0.001) and were on average 17.9% greater than the bottom logs based on MOE, MOR, Young's Modulus, UCS, and Density. However, density of the top logs was on the average lower by 4.5% compared to the bottom logs; this trend indicates that the wood in the bottom logs has probably deteriorated more than the top ones. The mid logs did not yield significant differences in properties between live and partially deteriorated (average P-value ~0.515). Densities of both live and partially deteriorated wood specimens were statistically similar (P-value = 0.384 and 0.411), but density also had the largest standard deviation. Overall the differences in properties of butt, middle, and top logs were generally statistically significant as seen in Figure 2.4, with the exception of density. Even though the three log locations yielded properties that were statistically significant, they were combined for further statistical analysis because of the wide scale of standard deviation that overlaps in all cases. Another reason for combining is that it would not be practical to sort logs by tree height similar to different regions as the logs are received to process in a typical wood-plastic composite facility. The deterioration levels were kept separate for further analysis.

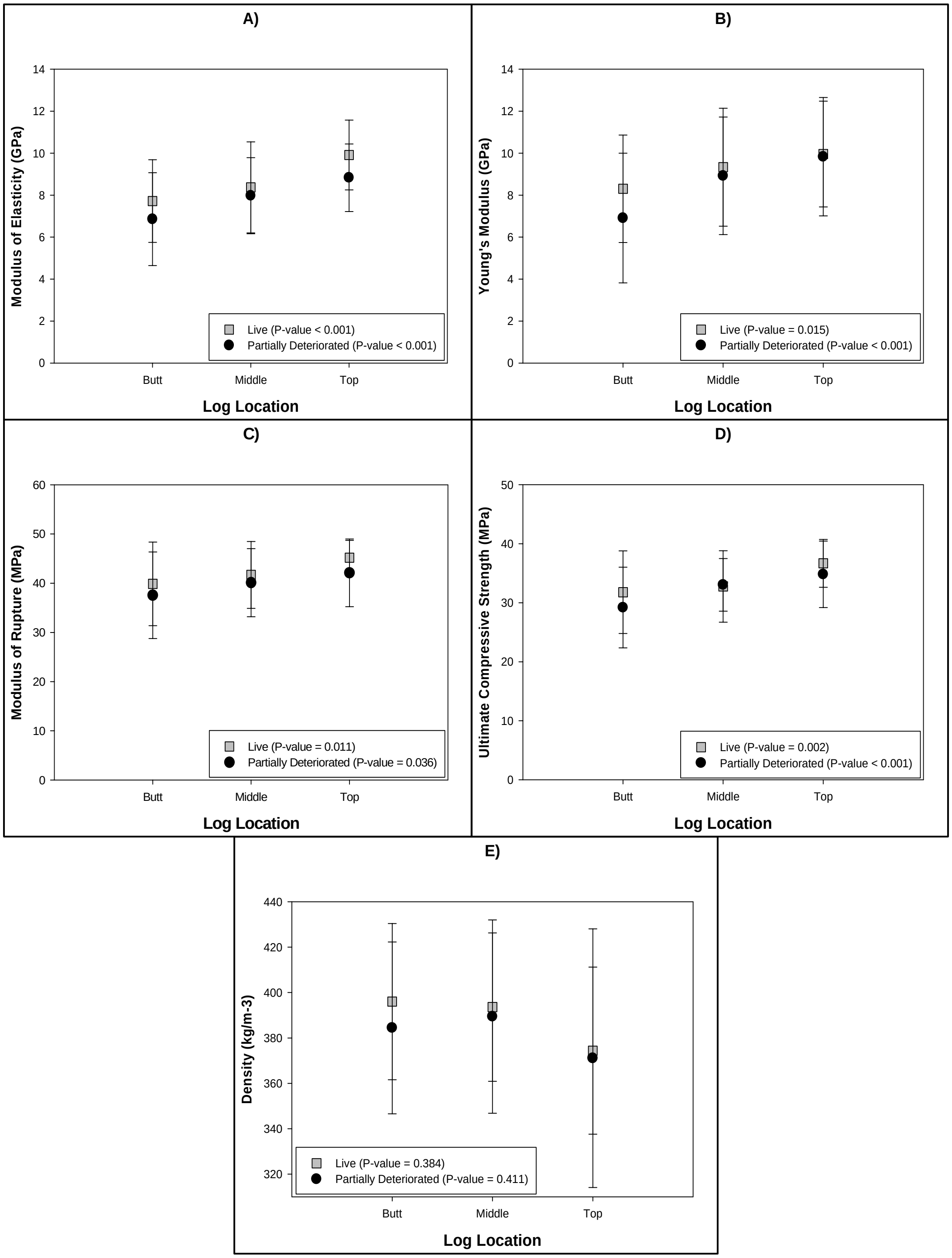

Figure 2.4. Comparison of log location within tree: A) Flexure Modulus, B) Compression Modulus, C) Flexure Modulus of Rupture, D) Ultimate Compression Strength, E) Density, showing that statistically there is a difference depending on tree height. (Error Bars are Standard Deviation)

Deterioration Level

The comparison graphs of the specimens for all mechanical properties shown in Figure 2.5 indicate similar trends of decreasing values from Live to Partially Deteriorated. Live small clear specimens averaged 7.0% higher values for the five mechanical properties shown in Figure 2.5 than that of partially deteriorated specimens. Compression modulus showed the largest discrepancy between live and partially deteriorated at 10.86%. Statistically the two deterioration levels differ enough that they were kept separate for WPC production and evaluation of extruded wood-plastic specimens.

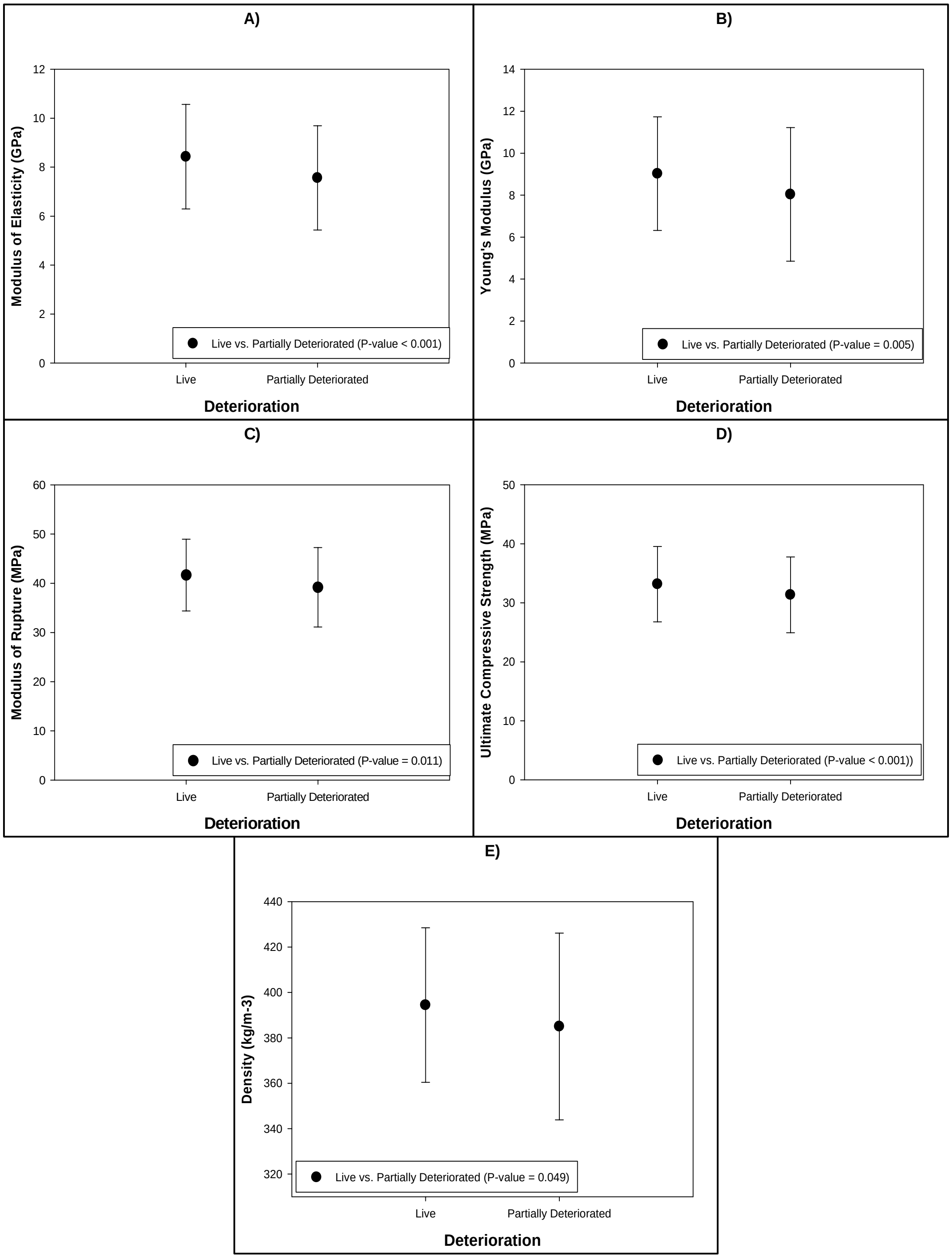

Figure 2.5. Comparison of live vs. partially deteriorated: A) Flexure Modulus, B) Compression Modulus, C) Flexure Modulus of Rupture, D) Ultimate Compression Strength, E) Density, indicate that the two deterioration levels are statistically different. (Error Bars are Standard Deviation)

Summary and Conclusions

The objective of this study was to quantify the mechanical properties of live and partially deteriorated spruce due to bark beetle infestation and assess the deterioration level with the long-term goal of using this wood irrespective of degree of deterioration for production of wood-plastic composites. Statistically the wood differs between the two sites (site one, site two) from which the trees were obtained but considering that trees will not be differentiated at the processing facility irrespective of their origin, the two sites were combined for all future studies. The location of the specimen along the tree height (butt, middle, top) was found to be statistically significant but only averaged a 17.9% difference in mechanical properties from top to bottom. Even after combining the specimens from different sites and tree heights, the two deterioration levels were statistically different for all performed tests, with Live small clear specimens averaging 7.0% higher values than Partially Deteriorated. The two deterioration levels (Live and Partially Deteriorated) were kept separate for further studying their effect on extruded WPC properties.

Having shown that degradation occurs in the wood due to infestation, the trees no longer become viable options for traditional wood products, such as lumber and veneers. WPC are one possible option for these beetle-killed trees as they are more forgiving to the initial quality of the wood feedstock. In the next chapter, a mixture of wood flour from beetle-killed spruce, irrespective of deterioration level will used in WPC production and examined to determine if any final mechanical properties are effected.

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Chapter Three

Wood-Thermoplastic Composites manufactured using Beetle-Killed spruce from Alaska's Kenai Peninsula

Introduction

Over the last 20 years the spruce bark beetle infestation on Alaska's Kenai Peninsula has produced 0.6 million ha (1.5 million acres) of dead and standing spruce (*Picea* spp.) trees. With the knowledge that the infestation is cyclical and occurs every 20 to 60 years (Berg 2003, Holsten 1990), additional uses beyond traditional wood products are needed as damage due to deterioration of these dead trees makes them unfit for production of lumber or veneer. One possible use is wood-plastic composites (WPC), which use wood flour as a feedstock and is less susceptible to initial wood conditions. Value-added options such as wood-plastic composites are needed to reduce the financial burden of harvesting these degraded trees. WPC have gained in popularity recently and today represent a rapidly gaining market share, primarily as a substitute for wood decking and railing (Wolcott and Englund 1999, Clemons 2002).

Objective

The primary objective of this study was to characterize wood flour produced using beetle-killed spruce of varying degrees of deterioration for extrusion of wood-plastic composites, and investigate the feasibility of using it as a feedstock in manufacturing WPC for use in Alaskan environment. More specifically, the tasks of this research include:

1. Characterize the feedstock for particle size distribution, chemical composition, and thermal degradation.

2. Extrude and evaluate WPC deck boards with varying proportions of live, partially deteriorated, and highly deteriorated wood flour based on a simplex mixture model and compare them to a control run extruded with pine wood flour.
3. Establish baseline properties for use as deck boards according to ASTM D7032-05; specifically evaluate flexure, moisture effect, temperature effect, freeze-thaw effect, and water absorption/thickness swell.

Methods and Materials

Based on the previous clear-specimen research within this project, it was determined that neither harvested site nor log location within a tree differed enough to warrant separate classifications; however, effect of degree of deterioration on clear specimen properties was significant. Therefore, in this study three deterioration classes of wood flour were evaluated (Live, Partially Deteriorated, and Highly Deteriorated) in the production of WPC deck boards.

Wood Flour Preparation

Five trees from each deterioration class were visually characterized and removed from three different growing sites on the Kenai Peninsula. Each tree had three 2.4 m (8 ft) bolts cut from it: one at the butt end, one from below the 101.6 mm diameter (4 in) top, and the third equidistant between those two. One-half of each bolt of live and partially deteriorated logs was processed into clear-specimens (see Chapter 2) and the remaining half was processed into wood flour. The highly deteriorated bolts were directly processed into wood flour because the deterioration of the trees prevented preparation of clear specimens.

Bolts were ripped into roughly cut 10.1 cm x 10.1 cm x 121.9cm (4in x 4in x 4ft) cants and processed into chips using a Sumner Iron Works Chipper. Chips were then hammer milled to a 60-mesh particle size, using a Bliss Industries Hammermill with a screen size of 1.17 mm (0.046in). Pine 60 mesh wood flour (American Wood Fiber 6020BB) was obtained commercially to extrude control specimens. Wood flour was characterized for particle size distribution using a Ro-Tap sieve analyzer, for thermal degradation using thermal gravimetric analysis (TGA), and for chemical composition using solvent extraction method and Fourier Transform Infrared (FTIR) spectroscopy. Thermal degradation began around 225°C (437°F) and reached maximum degradation at 361°C (682°F), which related closely with pine thermal degradation. Details of these analyses are presented in a publication included in Appendix C (Yadama et al. 2008). The wood flour was dried to less than 2% moisture content using a cylindrical steam tube dryer and stored in bags until WPC production.

WPC Manufacture

To study the influence of degree of deterioration of spruce wood on WPC properties, a mixture design of experiment based on simplex method was implemented in determining the required design runs of varying proportions of wood flour from the three deterioration levels for extrusion of WPC test specimens. The simplex method is a fractional factorial statistical design and analysis method that is ideally suited to develop response models for products that are a mixture of two or more ingredients (Cornell 1981); it is a required condition that the sum of the individual proportions of the mixture components is always equal to one. The objective of the simplex analysis is to develop an empirical model to estimate WPC properties based on the composition of its constituents. The results of the simplex analysis are shown in (Table 3.1). The wood flour was the only varying component of the WPC formulation as the other constituents of the WPC were held constant for all 17 runs. Selected runs were replicated to obtain a robust prediction model. Run 18 was the control run that was extruded using pine wood flour.

Table 3.1. Simplex mixture model showing design runs of varying proportions of wood flour from the three deterioration levels.

Run	Live (%)	Partially Deteriorated (%)	Highly Deteriorated (%)
1	0.00	100.00	0.00
2	33.33	0.00	66.67
3	100.00	0.00	0.00
4	100.00	0.00	0.00
5	66.67	0.00	33.33
6	66.67	33.33	0.00
7	33.33	33.33	33.33
8	66.67	16.67	16.67
9	16.67	16.67	66.67
10	0.00	0.00	100.00
11	0.00	100.00	0.00
12	0.00	0.00	100.00
13	0.00	33.33	66.67
14	16.67	66.67	16.67
15	33.33	66.67	0.00
16	0.00	66.67	33.33
17	0.00	33.33	66.67
Pine	100.00	0.00	0.00

The results from the simplex analysis provided the schedule of runs for WPC extrusion that were processed in the order shown. The WPC formulation used in all 18 runs consisted of 58% wood flour, 32% high density polyethylene (HDPE) pellets (Equistar Chemical LBO 100 00), 5% Talc (Chemical Distributors Inc.), 2% Zinc Stearate (Chemical Distributors Inc. DLG20), 2% Zinc Borate (Firebrake ZB, US. Borax) and 1% Ethylene Bis-stearamide wax (General Electric Specialty Chemicals). Each run weighed 78.2 kg (172.4 lbs) and was individually mixed in a rotating drum blender for 10 minutes the morning before extrusion took place. All extrusion was performed at Washington State University Wood Materials and Engineering Laboratory (WMEL), using a counter-rotating conical twin-screw extruder (ExtrusionTek Milacron Twin Screw Extruder, TC-86 mm). The rate of production was held constant at 16 rpm or approximately 204.1kg/hr (450 lbs/hr) for all 18 runs. The average temperatures inside the extruder barrel and die were maintained at 171°C (340°F) and 176°C (350°F).

Processing parameters were consistent throughout the 17 runs, with the melt pressure averaging 90.2 MPa (942 psi), torque amps averaging 25 % and screw thrust averaging 16 %. The extruded deck board was rectangular in cross-section, with rounded corners and was approximately 139 mm (5.5in) wide and 25 mm (1 in) thick. Extruded deck boards were cut into 2.4 m (8 ft) boards and later cut into 508 mm (20 in) test specimens according to ASTM D7032-05 (ASTM 2005).

Testing

For all 18 runs, baseline flexure, moisture effect, temperature effect, freeze-thaw effect, and water absorption/thickness swell were tested according to ASTM D7032-05. All specimens were conditioned at 21.1°C (70°F) and 50% relative humidity for at least 48 hours prior to testing and were also tested in the same temperature and relative humidity conditions. Each bending test had six deck boards tested per run except, freeze-thaw, which had five deck boards tested per run.

Baseline flexure specimens were tested after conditioning without any further influences. As for moisture effect specimens, they were submerged in tap water for 24 hours prior to testing. The effects of changes in temperature were determined by conditioning specimens in two different environmental conditions prior to testing in flexure. Six specimens were conditioned at 51.7°C (125°F) for at least 48 hours, and six specimens were conditioned at -28.9°C (-20°F) for at least 48 hours. Freeze-thaw specimens were conditioned according to ASTM D7032-05 Section 4.7. Five specimens were submerged in tap water for 24 hours and then placed in a freezer at -28.9°C (-20°F) for 24 hours. After these two cycles, specimens were placed in a 21.1°C (70°F) and 50% relative humidity room for 24 hours. This process was repeated two more times after which flexure testing was conducted.

Water absorption and thickness swell had three specimens per run and were 15.24 cm (6 in) in length. Water absorption/thickness swell tests were conducted in trays filled with distilled water and measurements were consistently taken at intervals of 2 hrs, 24 hrs, 48 hrs, 168 hrs, 336 hrs, 672 hrs, 1008 hrs and 1334 hrs. The same scale and set of calipers were used for measurements at each time interval throughout the 8 week test. Moisture diffusion coefficient was also calculated to determine the rate of diffusion through the WPC. Water uptake is often described as a non-steady state diffusion (or Fickian behavior) in which a concentration gradient of a molecule diffusing through a medium changes with respect to time and results in a net accumulation in mass (Anderson 2007). Water diffusion coefficients for all runs were calculated using the following formula (Yadav et al. 1999):

$$D_A = \pi \left[\frac{mh}{4M_{sat}} \right]^2 * \left[1 + \left(\frac{h}{L} \right) + \left(\frac{h}{n} \right) \right]^{-2} \qquad (1)$$

Where D_A is the water diffusion coefficient corrected for edge effect; *m,* is the linear portion of the water absorption against square root of time; M_{sat}, is the assumed saturated point after 8 weeks; *h*, the thickness; *L*, the length; and *n* is the width.

The weight and the dimensions of the deck board specimens were measured and recorded prior to testing for analyzing mechanical properties upon completion of testing. All specimens were tested on a bending test fixture (30 kip, Instron Corporation 4400R), with a testing span of 40.64 cm (16 in) and a loading span that was located at the third points, 13.54 cm (5.33 in) from each end support (Figure 3.1). A linear variable differential transformer (LVDT) was used to measure mid-span deflections. A constant cross-head speed of 12.2 mm/min (0.4784in/min) as per ASTM D7032-05 was maintained until specimen failure.

Figure 3.1. Typical flexure test setup for WPC deck boards.

The load-deflection data was recorded at 0.5 second intervals during each test and captured using National Instruments LabVIEW® 8.0 software. Strain was determined using the following equation:

$$r = \left(\frac{4.7 \Delta d}{L^2} \right) \tag{2}$$

where: Δ = the specimen deflection at the mid-span (cm),
d = depth (cm),
L = testing span (cm), and
r = strain (cm/cm).

The Stress was determined with the following equation:

$$\sigma = \left(\frac{PL}{bd^2} \right) \tag{3}$$

where: P = load (kg),
L = testing span (cm),
b = specimen width (cm),
d = specimen depth (cm), and
σ = stress in outer fibers (MPa).

The modulus of elasticity (MOE) was calculated by determining the slope of the least squares fit line representing the stress versus strain data between 10% and 40% of the maximum load. Modulus of rupture (MOR) was calculated from the maximum load achieved or the load at 3% strain, whichever occurred first, per ASTM D7032-05 standards. As variation in density is minimal with WPCs, density was calculated by measuring and weighing all deck board specimens prior to testing and then averaging either six or five specimens, depending on the mechanical test.

Results and Discussion

Wood Flour Particle Size Analysis, Thermal Analysis, and Chemical Composition

Mechanical interlocking is the primary method of interaction between wood fibers and thermoplastic within a WPC deck board (Mahlberg et. al 2001). Therefore, particle size and the corresponding aspect ratio of wood fibers are important properties influencing processing parameters and overall structural qualities of WPC. The resulting particle size distribution for the three deterioration levels and pine are shown below in Figure 3.2 and Table 3.2.

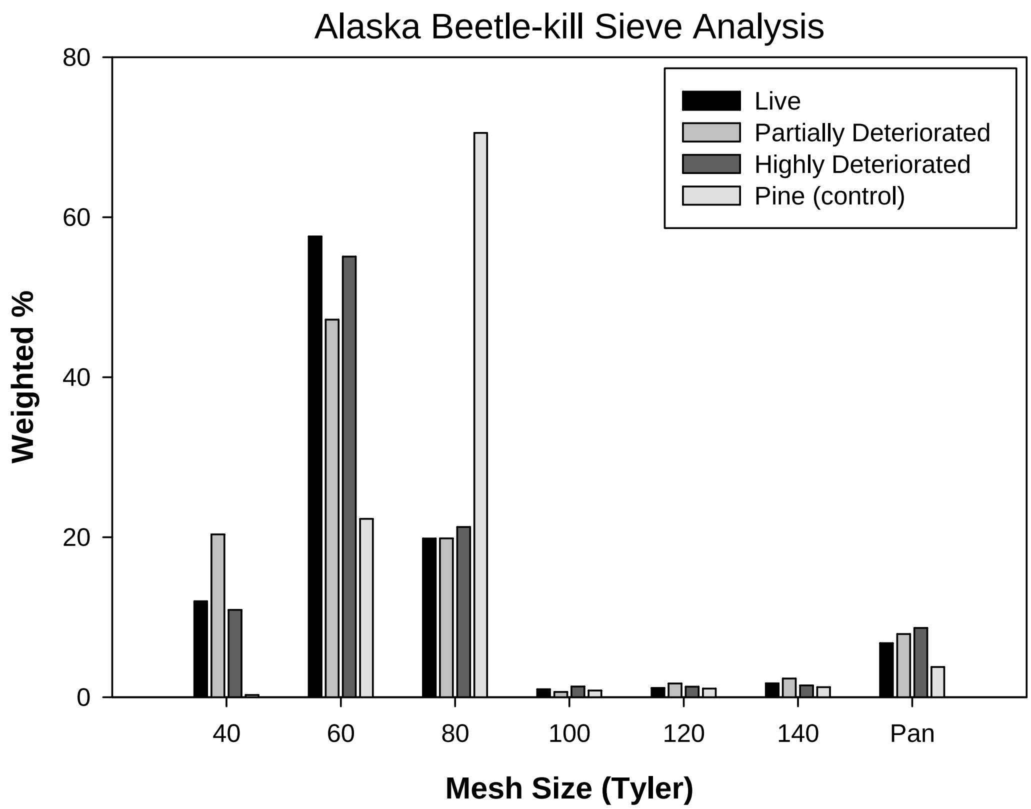


Figure 3.2. Particle size distribution by deterioration level using 1.168mm (0.046") screen.

All three deterioration levels had a much higher percentage retained in the 60-mesh category than pine did and this might be attributed to the way that American Wood Fiber manufactures and distributes their 60-mesh pine (American Wood Fiber 6020BB). The number of fines generally was greatest for Highly Deteriorated compared to the other two deterioration levels including pine. The sieve analysis also indicates a tendency for deteriorated spruce wood to generate higher percentage of finer particles than the live spruce wood; particle size effects the mechanical interaction between the wood and plastic and could influence the packing characteristic of the extruded wood-plastic composite.

Table 3.2. Sieve Analysis for Alaska Beetle-kill spruce using 1.168mm (0.046") screen.

Alaska Beetle-kill Sieve Analysis								
Mesh Size (Tyler)	**Live (%)**	**STDEV[1]**	**Partially Deteriorated (%)**	**STDEV[1]**	**Highly Deteriorated (%)**	**STDEV[1]**	**Pine (%)**	**STDEV[1]**
40	12.0	4.12	20.4	9.02	10.9	4.98	0.3	0.03
60	57.6	7.79	47.2	11.81	55.1	1.45	22.3	9.11
80	19.8	7.96	19.9	4.39	21.3	4.19	70.5	6.25
100	1.0	0.08	0.6	0.14	1.3	0.86	0.8	0.01
120	1.1	0.30	1.7	0.80	1.3	0.41	1.1	0.60
140	1.7	0.13	2.3	0.75	1.5	0.48	1.3	0.65
Pan	6.7	0.32	7.9	0.21	8.6	2.21	3.8	1.79

[1] STDEV = Standard Deviation

In a study by Stark and Berger (1997) it was shown that flexural MOE and MOR appear to increase with increasing particle size (for particles smaller than about 0.25mm). In this study three deterioration levels (Live, Partially Deteriorated, Highly Deteriorated) were used and it was hypothesized that the number of fines (<60-mesh) would increase, moving from Live to Highly Deteriorated and would thus influence the aspect ratio of each respectful wood flour category. Stark and Rowlands (2003) note that the aspect ratio of wood particles increases as the mesh size of wood flour increases. In WPC, stiffness properties improve as the aspect ratio of the wood flour used increases, indicating that having a greater number of fines within the wood flour might result in a less stiff product.

Chemical composition and thermal degradation results are discussed in Yadama et al. (2008) that is provided in Appendix C. In general, the results indicated an evidence of carbohydrate depolymerization although there was no significant change in the chemical composition. As for thermal degradation, wood deterioration level had no significant influence on thermal degradation behavior.

Mechanical Properties

Results of mechanical properties for the five flexure tests performed are presented in Tables 3.3 and 3.4 and figures 3.3 and 3.4. Failure of all flexure testing specimens was commonly due to fracture of the bottom fibers in tension near midpoint between the load points.

Mechanical properties of all runs composed of varied proportions of deterioration levels exceeded that of control pine. Analysis of variance indicated that there were some statistical differences in initial flexure property means of various formulations examined at an ά-level of 0.05. Results of comparison of means tests (Duncan grouping) for MOE and MOR based on initial flexure tests are shown in far right columns of Tables 3.3 and 3.4. The only run that shared no statistically similarities with any other run was the control run using 100% pine wood flour. .

As for moisture effects (i.e., after a water soak of 24 hours), only the run composed of 66.7% Partially Deteriorated and 33.3% Live showed a slight decrease (less than 1%) in MOE and MOR, but all others showed an average increase of 2 to 6% in MOE and MOR values over initial flexure properties. This is also the run that differed drastically from the other runs in the freeze-thaw effect test (decrease of 10.2%), whereas other runs averaged a decrease of -3.5% in MOE. Pine exhibited a greater decrease in MOE (-8.6%) after freeze-thaw effect than any of the beetle-killed spruce runs with the exception of the run listed previously. The high temperature and low temperature effect varied little among the deterioration levels, averaging -28.7% and +27.9% respectively.

Temperature effects were more severe for WPC specimens with pine wood flour as for MOE (-35.8% and +36.0%). Overall pine exhibited comparable MOE properties in the low temperature (-28.9°C) effect, while being substantially lower in the moisture effect, high temperature effect and the freeze-thaw effect. Runs composed of beetle-killed spruce exhibited an average of 14.8% higher MOE values than pine among the five tests conducted. However, the run composed of 100% Highly Deteriorated wood flour still exceeded the MOE values of the control pine for all five properties tested.

Table 3.3. Stiffness properties for wood-plastic composites manufactured from beetle-killed spruce from Alaska's Kenai Peninsula.

Modulus of Elasticity (Coefficient of Variation - %)											
	# of Samples	**Static Bending - GPa**	**Moisture Effect - GPa**	**% Change**	**High Temperature - GPa**	**% Change**	**Low Temperature - GPa**	**% Change**	**Freeze Thaw -GPa**	**% Change**	**Duncan Grouping for Static Bending**
100% Live	12	4.24 (1.50)	4.39 (2.15)	+3.4	3.04 (2.97)	-28.3	5.93 (1.04)	+28.5	4.16 (1.99)	-1.9	A
100% Partially	12	4.28 (1.18)	4.37 (2.28)	+2.1	3.02 (5.26)	-29.4	5.81 (2.27)	+26.3	4.11 (2.72)	-4.0	AB
100% Highly	12	4.05 (1.14)	4.21 (2.94)	+3.8	2.88 (2.53)	-28.9	5.84 (4.20)	+30.7	3.86 (1.99)	-4.7	C
66.7% Live 33.3% Partially	6	4.15 (0.81)	4.31 (0.84)	+3.7	3.04 (2.70)	-26.7	5.56 (2.74)	+25.4	4.13 (0.72)	-0.5	D
66.7% Live 33.3% Highly	6	4.18 (1.06)	4.33 (1.82)	+3.5	2.89 (3.43)	-30.9	5.45 (2.87)	+23.3	4.09 (0.81)	-2.2	DE
66.7% Partially 33.3% Live	6	4.24 (0.47)	4.21 (1.67)	-0.7	2.93 (2.79)	-30.9	5.93 (2.45)	+28.5	3.81 (2.79)	-10.1	AB F
66.7% Partially 33.3% Highly	6	4.21 (1.02)	4.44 (1.35)	+5.2	2.88 (2.34)	-31.6	5.98 (1.45)	+29.6	3.99 (1.22)	-5.2	A EFG
66.7% Highly 33.3% Live	6	4.28 (0.91)	4.38 (2.16)	+2.3	3.01 (2.88)	-29.7	5.93 (0.57)	+27.8	4.23 (1.04)	-1.2	AB H
66.7% Highly 33.3% Partially	12	4.15 (2.74)	4.23 (2.44)	+1.9	2.90 (3.09)	-30.1	5.93 (3.77)	+30.0	3.97 (3.07)	-4.3	DE G I
33.3% Live 33.3% Partially 33.3% Highly	6	4.26 (0.96)	4.37 (0.83)	+2.5	3.07 (3.68)	-27.9	5.54 (3.87)	+23.1	4.06 (1.02)	-4.7	AB F H
66.7% Live 16.7% Partially 16.7% Highly	6	4.12 (3.98)	4.30 (4.01)	+4.2	3.22 (6.15)	-21.8	5.31 (2.36)	+22.4	3.98 (2.59)	-3.4	CDEF I
16.7% Live 66.7% Partially 16.7% Highly	6	4.07 (1.47)	4.18 (1.59)	+2.6	2.85 (2.77)	-30.0	5.91 (1.06)	+31.1	3.96 (1.30)	-2.7	C I
16.7% Live 16.7% Partially 67.7% Highly	6	4.07 (1.00)	4.33 (2.09)	+6.0	2.98 (2.67)	-26.8	6.33 (1.77)	+35.7	4.04 (1.54)	-0.7	C I
100% Pine (Control)	6	3.71 (0.96)	3.82 (1.77)	+2.9	2.38 (3.52)	-35.8	5.80 (5.78)	+36.0	3.39 (2.36)	-8.6	

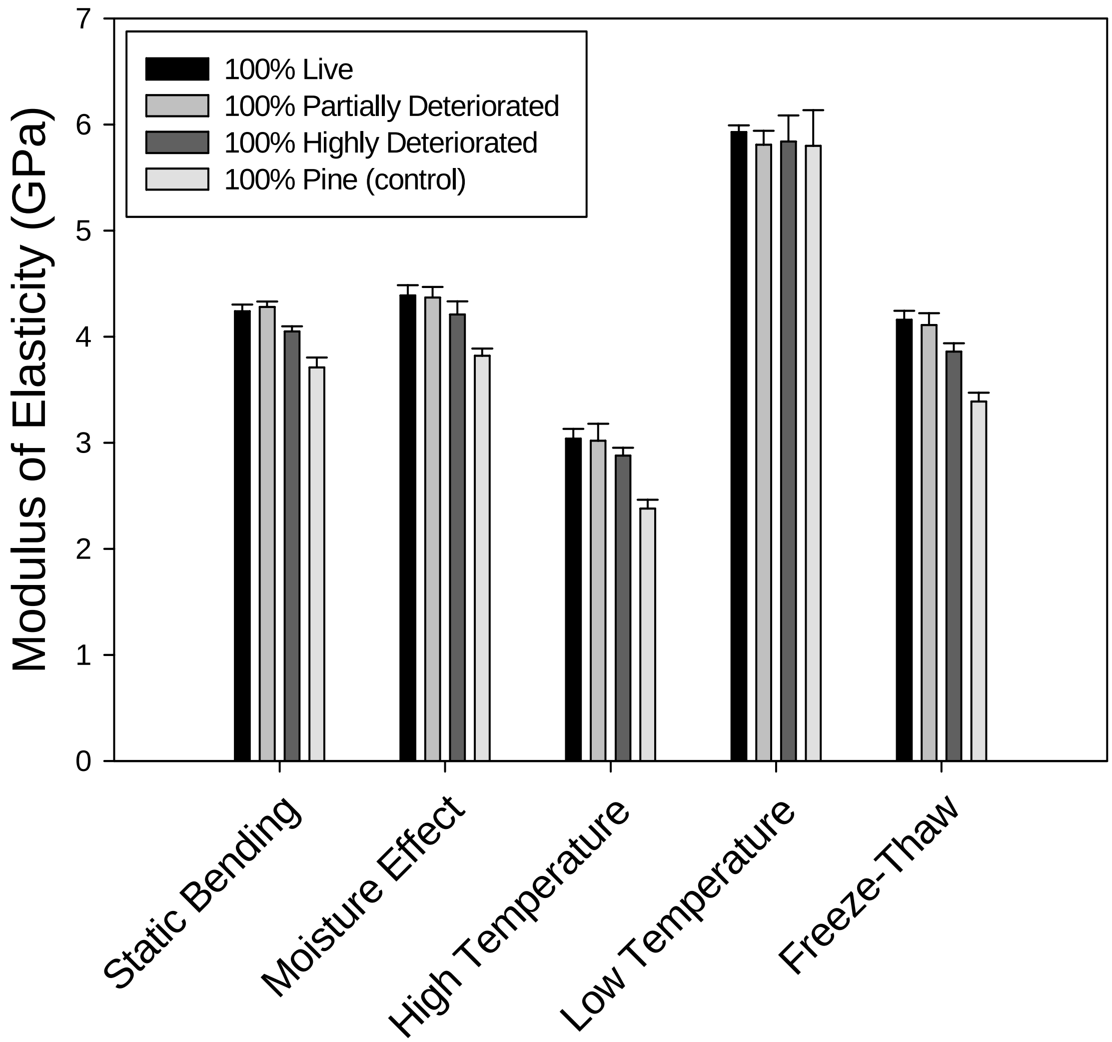


Figure 3.3. Modulus of Elasticity (GPa) for deck boards tested in flexure initially and after moisture, temperature, and freeze-thaw effects.

MOR values showed fewer differences among the runs and were also comparable to the control pine. Runs composed of beetle-killed spruce generally had higher strength values than the control pine in the five tests performed (~9.1% greater). Like the MOE values, the only run to show a decrease in MOR after moisture effect was the run composed of 66.7% Partially Deteriorated and 33.3% Live, which showed a -0.4% decrease. All other runs showed an average increase in MOR of +2.5% after moisture effect. The runs showed similar characteristics after the high and low temperature effect tests, averaging a change of -26.1% and +25.9% respectively. This was also exhibited by the pine control run that showed a decrease of 27.0% after exposure to high temperature and an increase of 26.3% after exposure to low temperature. The run composed of 66.7% Live, 16.7% Partially Deteriorated and 16.7% Highly Deteriorated exhibited the greatest change after freeze-thaw with a decreased in MOR of 3.1%.

Table 3.4. Strength properties for wood-plastic composites manufactured from beetle-killed spruce from Alaska's Kenai Peninsula.

Modulus of Rupture (Coefficient of Variation - %)											
	# of Samples	**Static Bending -** MPa	**Moisture Effect** - MPa	**% Change**	**High Temperature** - MPa	**% Change**	**Low Temperature** - MPa	**% Change**	**Freeze Thaw** -MPa	**% Change**	**Duncan Grouping for Static Bending**
100% Live	12	22.4 (1.86)	22.8 (1.64)	+1.8	16.4 (3.13)	-26.8	30.8 (2.33)	+27.3	22.6 (3.03)	+0.9	A
100% Partially	12	22.5 (1.64)	22.9 (7.30)	+1.7	16.8 (3.53)	-25.3	30.1 (10.77)	+25.2	22.8 (2.76)	+1.3	A
100% Highly	12	22.4 (2.50)	23.3 (3.58)	+3.9	17.2 (2.60)	-23.2	30.3 (3.29)	+26.1	22.3 (2.95)	-0.4	A
66.7% Live 33.3% Partially	6	22.3 (1.19)	22.8 (1.18)	+2.2	16.8 (4.72)	-24.7	31.2 (4.14)	+28.5	22.6 (2.49)	+1.3	A
66.7% Live 33.3% Highly	6	22.5 (1.34)	23.4 (1.75)	+3.8	16.3 (2.71)	-27.6	30.9 (4.99)	+27.2	22.8 (1.74)	+1.3	A
66.7% Partially 33.3% Live	6	22.6 (1.28)	22.5 (1.00)	-0.4	16.8 (3.59)	-25.7	30.7 (3.77)	+26.4	22.3 (1.72)	-1.3	A
66.7% Partially 33.3% Highly	6	22.9 (1.45)	24.1 (2.50)	+5.0	16.9 (2.08)	-26.2	31.2 (3.99)	+26.6	23.3 (2.75)	+1.7	A
66.7% Highly 33.3% Live	6	23.6 (2.39)	24.1 (2.90)	+2.1	16.8 (2.45)	-28.8	32.1 (0.59)	+26.5	23.6 (2.73)	0.0	B
66.7% Highly 33.3% Partially	12	22.5 (1.26)	22.9 (3.07)	+1.7	16.7 (5.56)	-25.8	30.0 (5.31)	+25.0	22.6 (3.72)	+0.4	BC
33.3% Live 33.3% Partially 33.3% Highly	6	22.9 (0.81)	23.3 (1.44)	+1.7	17.0 (3.96)	-25.8	30.5 (3.50)	+24.9	22.5 (0.99)	-1.7	B
66.7% Live 16.7% Partially 16.7% Highly	6	22.6 (4.26)	23.0 (2.10)	+1.7	16.9 (1.49)	-25.2	28.7 (3.21)	+21.3	21.9 (1.96)	-3.1	A C
16.7% Live 66.7% Partially 16.7% Highly	6	22.2 (2.58)	23.0 (2.10)	+3.5	16.3 (4.35)	-26.6	30.0 (1.82)	+26.0	22.4 (0.87)	+0.9	A
16.7% Live 16.7% Partially 67.7% Highly	6	23.0 (0.41)	23.2 (3.62)	+0.9	16.8 (1.76)	-27.0	31.2 (2.90)	+26.3	22.9 (1.11)	-0.4	C
100% Pine (Control)	6	20.8 (3.27)	21.1 (3.05)	+1.4	14.3 (3.10)	-31.3	29.8 (7.00)	+30.2	20.5 (1.63)	-1.4	A

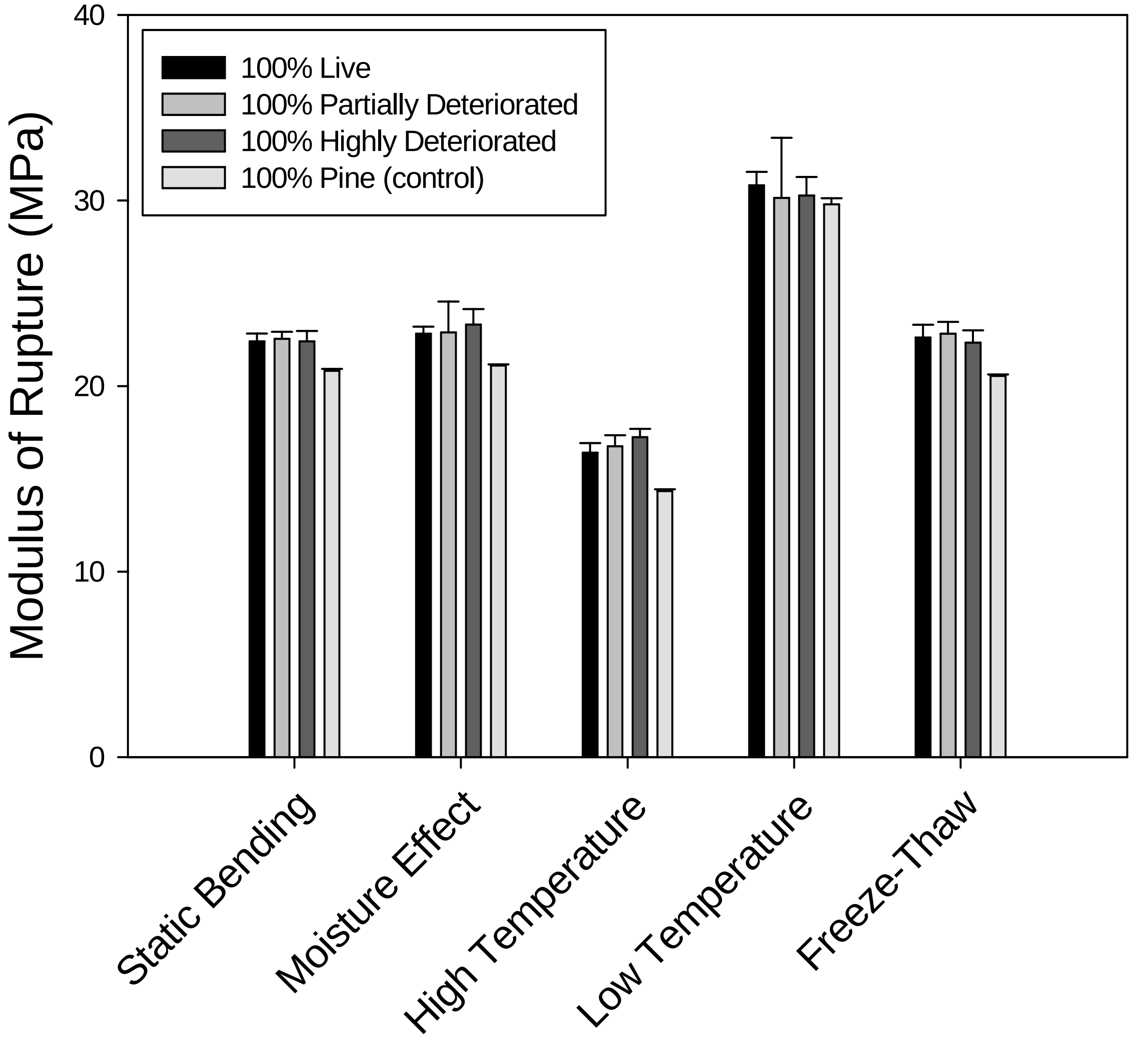


Figure 3.4. Modulus of Rupture (MPa) for deck boards tested by static bending, moisture effect, high temperature, low temperature and freeze-thaw.

Results for flexure testing were also analyzed over the simplex design space to examine the trends in response due to changes in wood flour composition. In all cases, quadratic models were used for the response surfaces.

Each vertex of the triangle represents a pure mixture of, Live, Partially Deteriorated or Highly Deteriorated (A, B, C respectfully) wood flour, accounting for 58% of the total mixture. Points along the lines connecting these vertices are binary and represent a varying mixture of the two components. The center point represents the mixture composed of 1/3rd of each deterioration level. The other points within the triangle are tertiary and represent varying proportions of the three deterioration levels, always adding up to 58% of the total mixture.

Densities (Figure 3.5) varied minimally between the runs, but runs composed of predominately Live and Highly Deteriorated wood flour exhibited higher densities according to the simplex response surface. WPC densities varied only 1.82% between the high and low and were slightly lower than WPC deck boards made from pine which had a density of 1163 kg/m³.

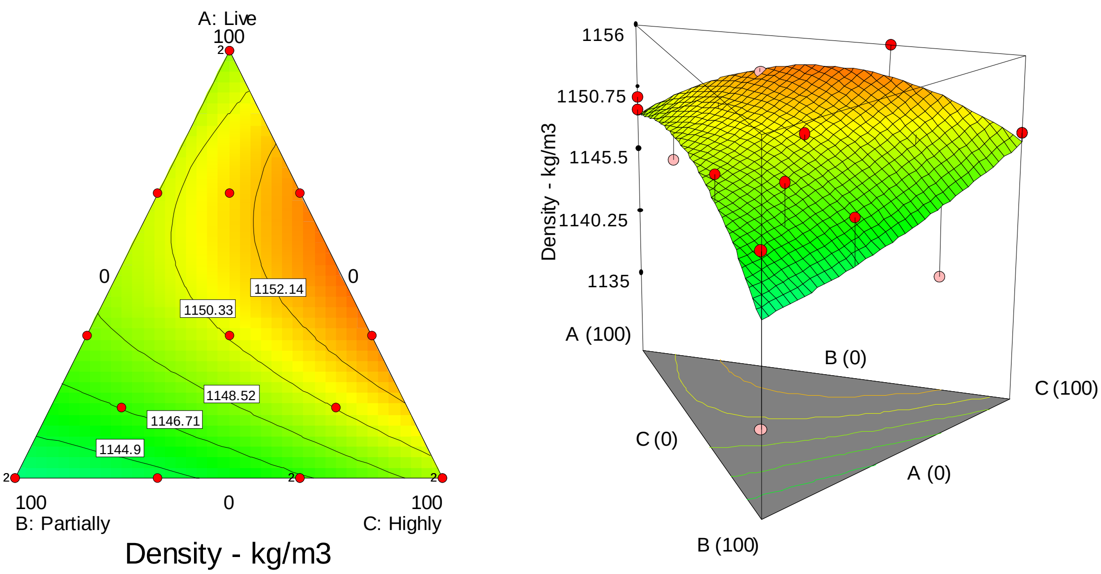


Figure 3.5. 2-D contour map and 3-D response surface of density variations in WPC specimens.

The MOE (Figure 3.6) varied from 3.95 GPa to 4.32 GPa for the formulations tested and the mixture model indicates a decreasing trend as the blend approaches 100% Highly Deteriorated. In general, wood flour from live trees resulted in higher modulus of elasticity, although not significantly higher. As per the analysis, the optimal mixture for MOE in baseline testing is a run composed of 66% Live, 16.67% Partially Deteriorated, and 16.67% Highly Deteriorated wood flour. These results are significant as they indicate that beetle-killed spruce wood flour from Kenai Peninsula, irrespective of degree of deterioration, could be used for production of wood-plastic composites without significantly affecting the modulus of elasticity. Similar trend was noticed for modulus of rupture as well.

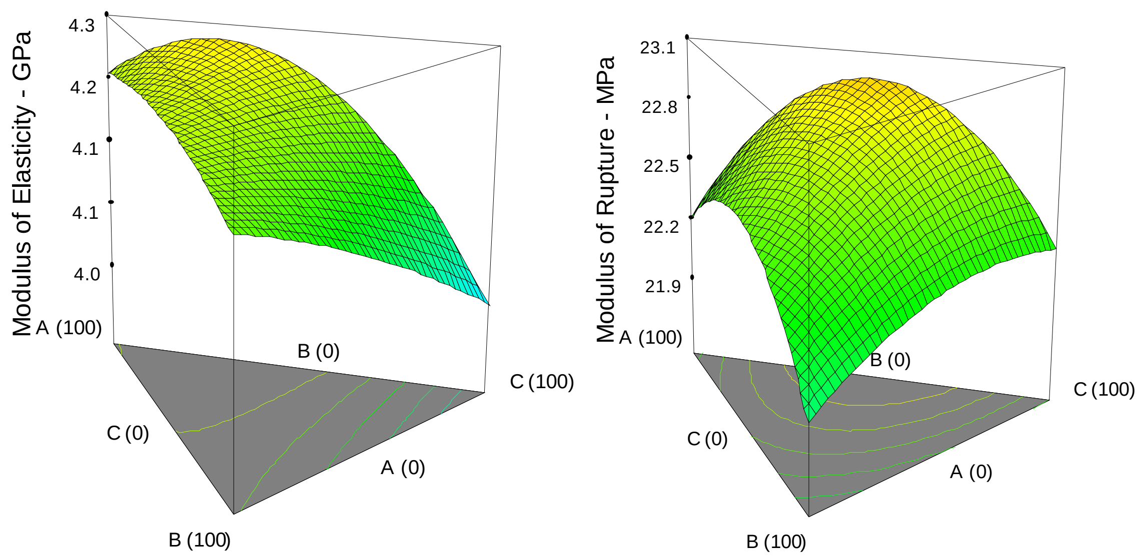


Figure 3.6. 3-D response surface for Modulus of Elasticity (MOE) and Modulus of Rupture (MOR) of baseline flexure specimens. (A – Live, B – Partially Deteriorated, C – Highly Deteriorated)

Higher MOR values were achieved in runs containing Live and Highly Deteriorated wood flour and averaged 22.4 MPa (Figure 3.6). The difference in MOR between the highest value and lowest value was 11.1%. Once again a mixture of Live and Highly Deteriorated wood flour resulted in slightly higher values of MOR.

After the deck boards were soaked in water for 24 hours and tested in bending, runs composed primarily of Live and Highly Deteriorated wood flour showed an increase in MOE compared to baseline flexure tests as shown in Figure 3.7. Runs with Partially Deteriorated wood flour exhibited lower mechanical properties for both MOE and MOR.

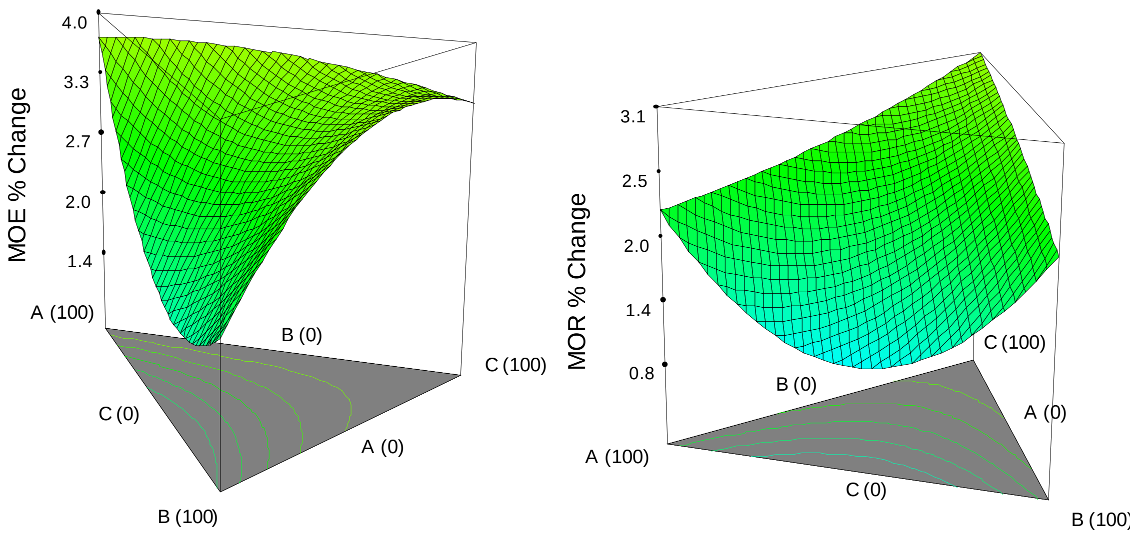


Figure 3.7. 3-D response surface for (%) change in Modulus of Elasticity (MOE) and Modulus of Rupture (MOR) after Moisture Effect compared to Baseline Flexure. (A – Live, B – Partially Deteriorated, C – Highly Deteriorated)

As for high temperature effects on flexure behavior (Figure 3.8), runs consisting of Live wood flour showed the lowest decrease in MOE, while those with Highly Deteriorated wood flour yielded highest MOR values after exposure to high temperature. With increasing proportions of deteriorated wood flour, exposure to high temperature reduced MOE of specimens more than MOR, which probably was a result of particle size distribution differences between live and deteriorated wood flour.

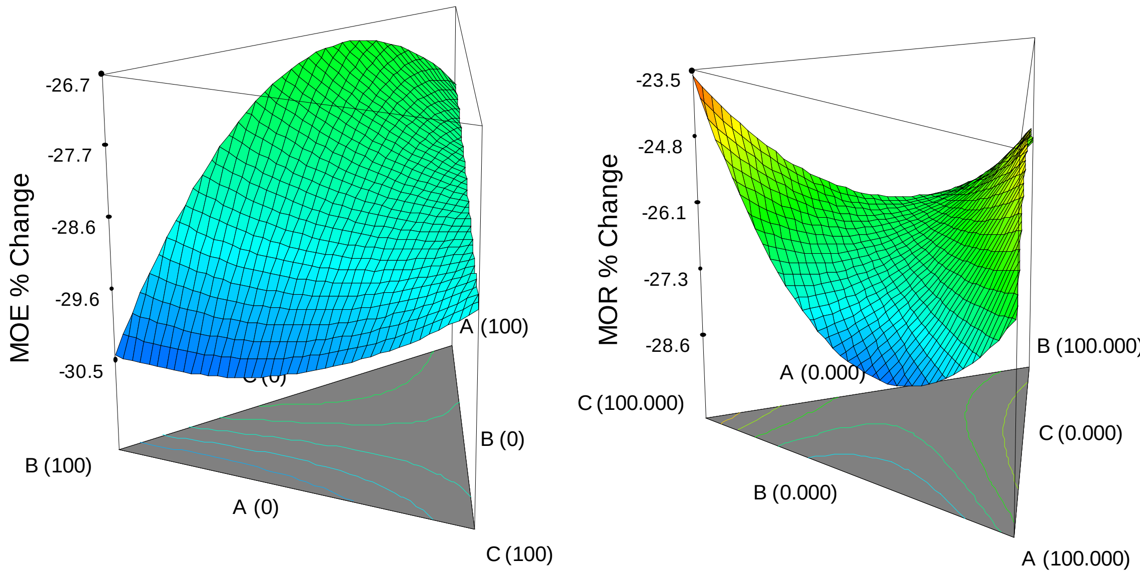


Figure 3.8. 3-D response surface for (%) change in Modulus of Elasticity (MOE) and Modulus of Rupture (MOR) after exposure to high temperature. (A – Live, B – Partially Deteriorated, C – Highly Deteriorated)

Exposure to low temperature increased the MOE and MOR of the deck boards by an average of 27.9% and 25.9% respectively (Figure 3.9). Highly Deteriorated exhibited the greatest increase for MOE, while Live showed the greatest increase for MOR. Once again increase in proportion of deteriorated wood flour resulted in greater change in MOE value and lesser change in MOR value.

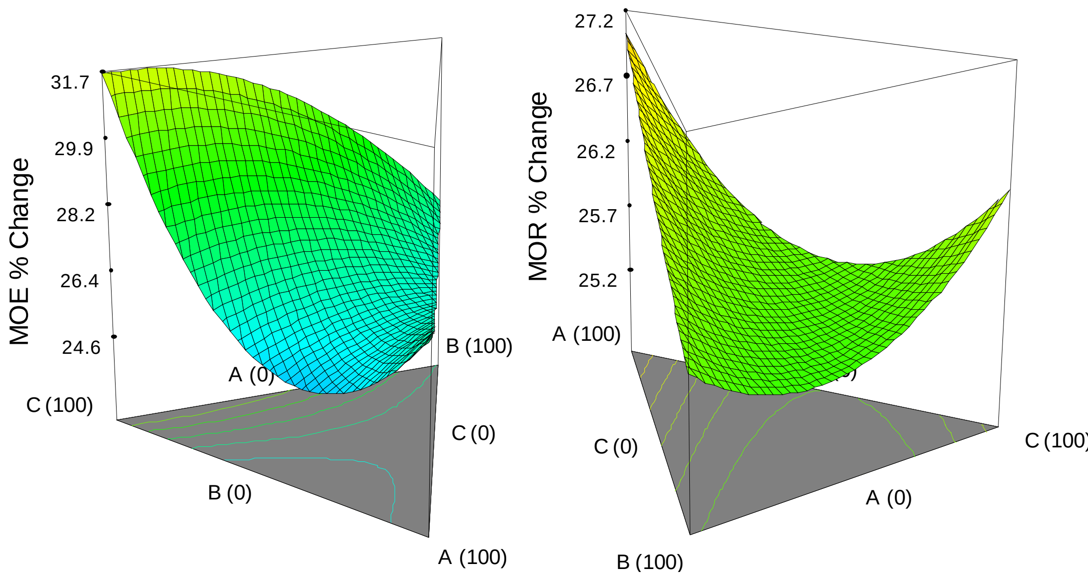


Figure 3.9. 3-D response surface for (%) change in Modulus of Elasticity (MOE) and Modulus of Rupture (MOR) after exposure to low temperature (A – Live, B – Partially Deteriorated, C – Highly Deteriorated).

Deck boards exposed to freeze-thaw effect showed a decrease in MOE and an increase in MOR compared to baseline flexure as percent of deteriorated wood flour was increased, especially in case of partially deteriorated wood flour. Runs largely composed of Live wood flour showed the smallest decrease in MOE among the three deterioration levels.

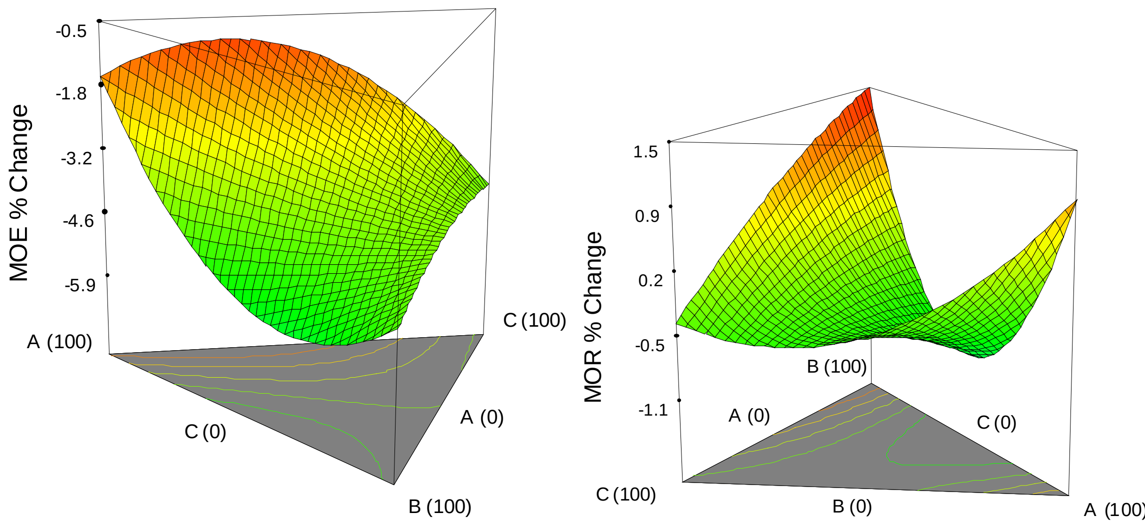
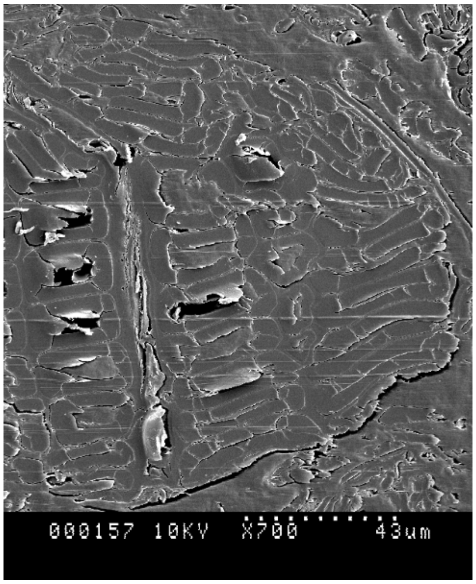
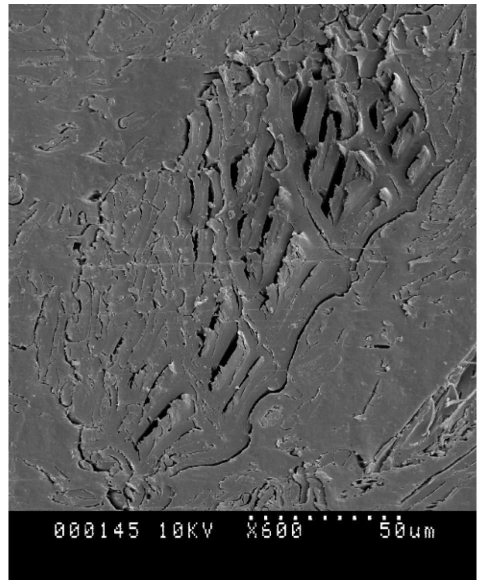


Figure 3.10. 3-D response surface for (%) change in Modulus of Elasticity (MOE) and Modulus of Rupture (MOR) after exposure to Freeze-Thaw cycles (A – Live, B – Partially Deteriorated, C – Highly Deteriorated).

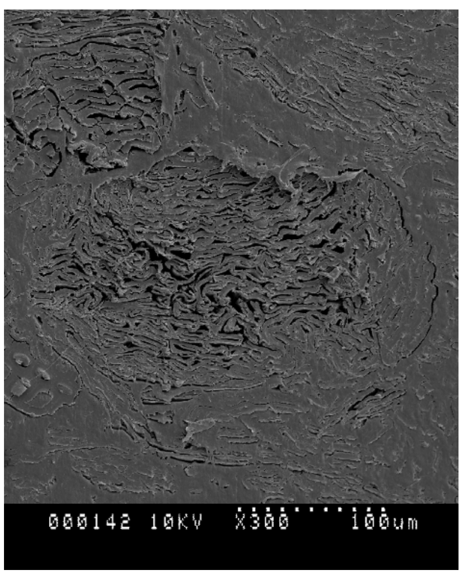
The interaction between the wood fibers and thermoplastic is an important aspect of WPC, as strength and stiffness are largely dependent on mechanical interlocking. SEM micrographs shown in Figure 3.11 were examined to analyze differences in thermoplastic flow within the wood cells lumens and differences in the effects of processing pressures on the wood fibers cell integrity (cell collapse).

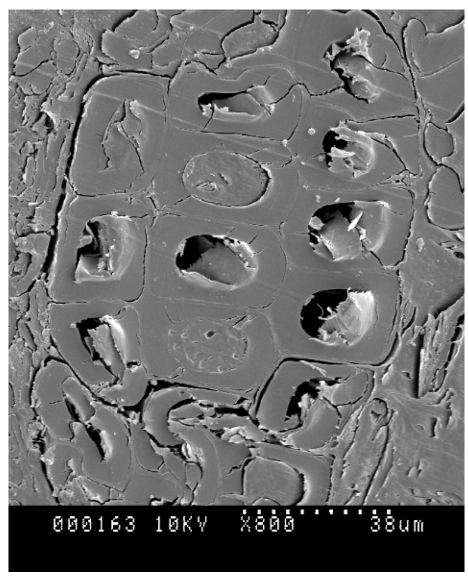
Characterization of WPC Morphology





A) B)





C) D)

Figure 3.11. Micrographs showing A) Live, B) Partially Deteriorated, C) Highly Deteriorated, and D) Control Pine.

The micrographs taken using a Scanning Electron Microscope (SEM) reveal some clues regarding the interaction between wood flour and thermoplastic matrix. As shown in Figure 3.11, the Live wood cells show that most of the fiber lumens are filled with HDPE similar to that of pine fibers. Cell walls of these fibers are intact and show no indication of collapse as a result of extrusion pressures. Fiber lumens of partially deteriorated wood flour were partly filled. However, there is a clear separation between fiber bundles and the thermoplastic matrix in all cases. In contrast, cell walls of fibers of highly deteriorated spruce fibers (Figure 3.11c) collapsed due to barrel pressures during the extrusion process indicating deterioration of their transverse strength due to changes in chemical structure of the cell walls, such as depolymerization of carbohydrates. Collapse of cell lumens prevented flow of HDPE into the fiber lumens of highly deteriorated wood which probably contributed to the drop in mechanical properties although not significantly.

Water Absorption and Thickness Swell

The majority of applications for wood-plastic composites are exterior and thus testing for water absorption and thickness swell is one measure of durability for such products. As mentioned in the testing procedures, readings were taken at intervals (2 hrs, 24 hrs, 48 hrs, 168 hrs, 336 hrs, 672 hrs, 1008 hrs and 1334 hrs) to help establish trends for water uptake and thickness swell. Figures 3.12 and 3.13 show water absorption and thickness swell for specimens after 8 weeks of water submersion.

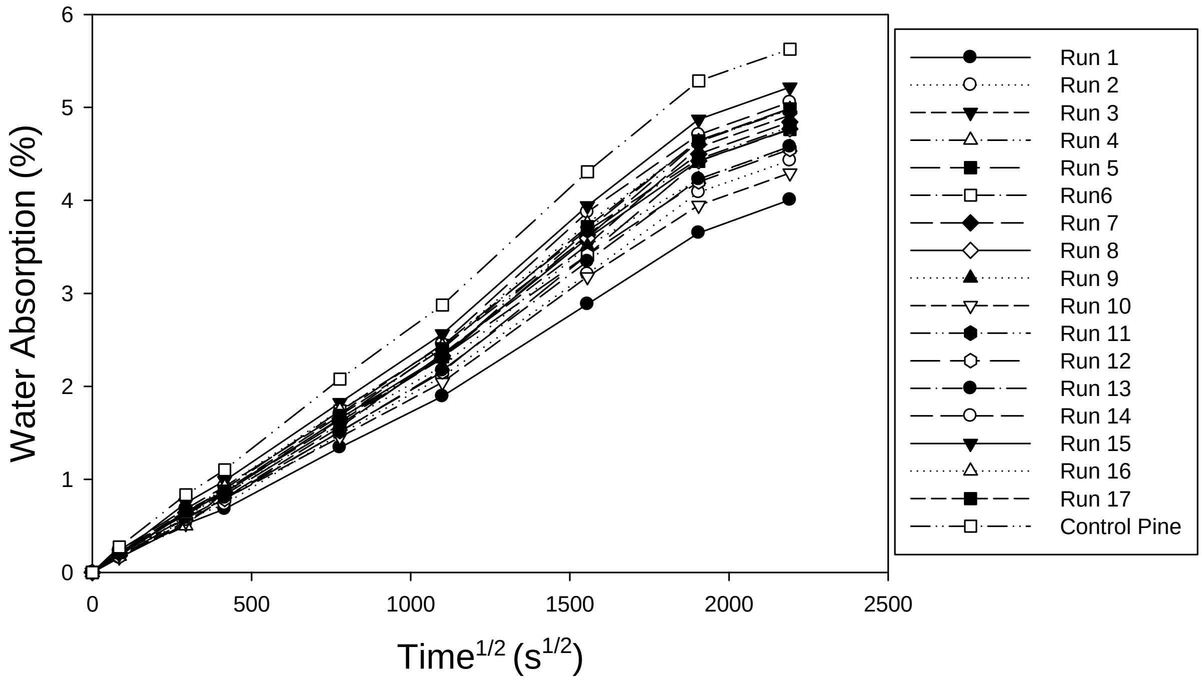


Figure 3.12. Water absorption for all formulations of wood-plastic composite specimens extruded in this study.

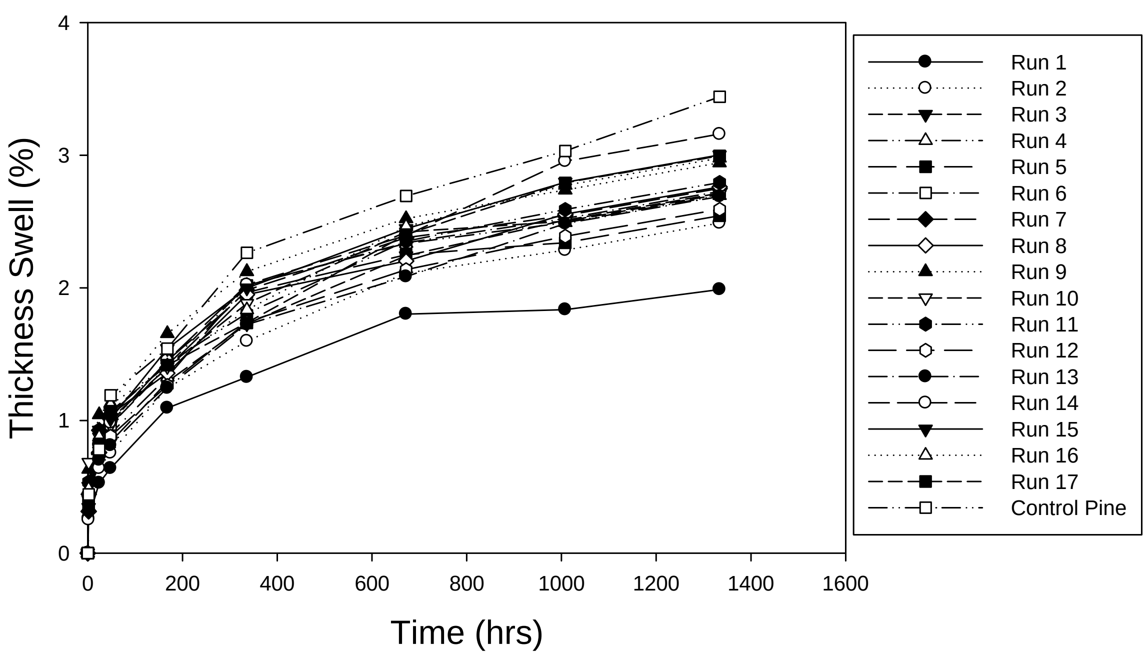


Figure 3.13. Thickness swell for wood-plastic composites manufactured using beetle-killed spruce and pine wood flour.

Water absorption rate was quite rapid between 1000 and 2000 hours, but seem to be slowing down beyond 2000 hours. In thickness swell a plateau started around 672 hours, with only slight increases occurring beyond that point. The WPC composed of 100% Partially Deteriorated wood flour absorbed water and swelled up at much slower rate than other formulations, with a 4.00% and a 1.99% increase respectively after 8 weeks of submersion. Rest of the runs tended to follow the same trend and averaged a 4.79% increase in water absorption and a 2.78% increase in thickness swell after 8 weeks. Besides the rate of absorption and swelling being higher than the other formulations, pine exhibited the greatest water absorption after 8 weeks, with a 5.63% increase; a thickness swell increase of 3.44% was also highest for all WPC specimens tested. A study that has already been published on WPC specimens extruded with highly deteriorated wood flour (Yadama et al. 2008), indicates that much higher percentages of absorption and thickness swell should have been found, as well as higher rates of absorption. We speculate that it was because the specimens in the previous study were planned down to 10.16 cm x 10.16 cm x 0.635 cm (4 in x 4 in x ¼ in) thus removing the polymer rich surface and exposing more wood fibers. The results in this study are lower as the test specimens were not planed and were full size deck boards as per ASTM D7032-05 testing standards. These results indicate the importance of test specimen preparation and interpretation of the results. In both studies the WPC composed of beetle-killed spruce exhibited better water absorption and thickness swell properties than the control pine. Data from the water absorption and thickness swell tests were also analyzed using simplex method to examine the effects of changing proportions of deteriorated spruce wood flour.

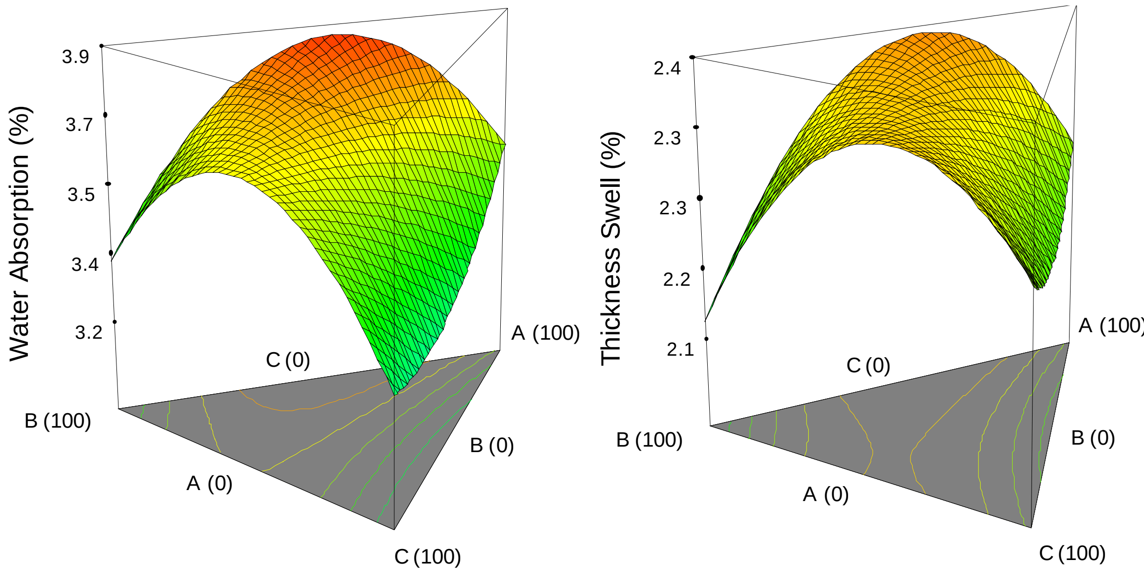


Figure 3.14. Water absorption and Thickness Swell analysis by Stat-Ease® software, for wood-plastic composites manufactured using beetle-killed spruce (A – Live, B – Partially Deteriorated, C – Highly Deteriorated) .

Results indicate that runs composed with a majority of Partially Deteriorated and Live wood flour show less resistance to water absorption and thickness swell.

Diffusion coefficients for each run were calculated and the results are listed in Table 3.5. Even though a saturation point had not been reached after 8 weeks of testing, the diffusion coefficient was calculated based on the assumed water saturation point at the 8 week measurement.

Table 3.5. Water diffusion coefficient for wood-plastic composites manufactured using beetle-killed spruce from Alaska's Kenai Peninsula.

Water Diffusion Coefficient		
	# of Samples	D (mm^2s^{-1})
100% Live	12	2.54E-04
100% Partially	12	2.50E-04
100% Highly	12	2.53E-04
66.7% Live, 33.3% Partially	6	2.50E-04
66.7% Live, 33.3% Highly	6	2.52E-04
66.7% Partially, 33.3% Live	6	2.48E-04
66.7% Partially, 33.3% Highly	6	2.51E-04
66.7% Highly, 33.3% Live	6	2.43E-04
66.7% Highly, 33.3% Partially	12	2.52E-04
33.3% Live, 33.3% Partially, 33.3% Highly	6	2.43E-04
66.7% Live, 16.7% Partially, 16.7% Highly	6	2.51E-04
16.7% Live, 66.7% Partially, 16.7% Highly	6	2.64E-04
16.7% Live, 16.7% Partially, 67.7% Highly	6	2.50E-04
100% Pine (Control)	6	2.49E-04

Trends based on mixture model indicate that as the proportion of partially and highly deteriorated wood flour increased, the water diffusion coefficient decreased as shown in Figure 3.15. This trend could be due to collapse of fiber cell walls during processing, resulting in better encapsulation of HDPE and barrier for moisture movement or due to the chemical changes that occur within Highly Deteriorated wood fiber as discussed in Yadama et al. (2008) (see Appendix B). Live wood flour exhibited the highest water diffusion coefficient based on the analysis using mixture response model.

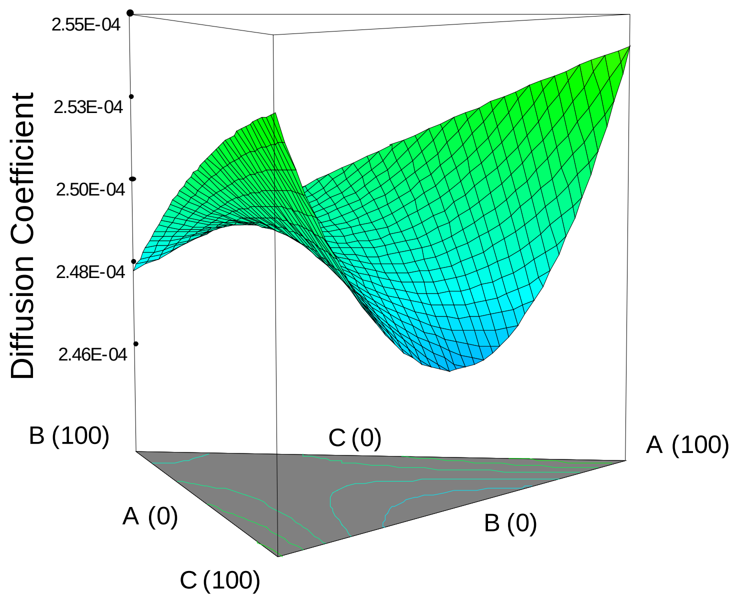


Figure 3.15. 3-D response surface for water diffusion coefficient (mm^2s^{-1}) of wood-plastic composites manufactured using beetle-killed spruce from Alaska's Kenai Peninsula (A – Live, B – Partially Deteriorated, C – Highly Deteriorated).

Summary and Conclusions

The main objective of the study was to examine the feasibility of utilizing beetle-killed spruce of various deterioration levels in the production of wood-plastic composites. Results of this study show that manufacturing WPC with beetle-killed spruce wood flour, irrespective of degree of deterioration, is viable.

Generally, deterioration level had little effect on the mechanical properties of the WPC, showing virtually no decline in MOR values and little decrease in MOE (4.5% decrease from Live to Highly Deteriorated). The largest effect that deterioration had on MOE values was after freeze-thaw, in which a decrease of 4.7% was seen versus a decrease of 1.9% for WPC made from Live wood flour. While temperature effects were significant on flexure properties, as was also seen in the control pine WPC, freeze-thaw cycles and moisture effect had the least effect on flexure behavior for all formulations. In all cases, the mechanical properties of WPC consisting of beetle-killed spruce exceeded the mechanical properties of pine WPC, averaging 14.8% higher. Water absorption and thickness swell rates of beetle-killed WPC were lower than WPC manufactured from pine wood flour, possibly due to greater encapsulation of finer particles by the thermoplastic and collapse of fiber bundles during extrusion process.

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CHAPTER FOUR

PROJECT SUMMARY AND CONCLUSIONS

Summary and Conclusions

The bark beetle infestation has posed numerous problems on Alaska's Kenai Peninsula, one of them being the devaluation of the infested trees for use in traditional wood products. One solution is to use these little to no value trees as a wood feed stock in the manufacture of wood-plastic composites (WPC). The purpose of this study was to characterize the effects of deterioration on the small clear specimens and then look at the feasibility of using varying proportions of beetle-killed spruce in the formulation of WPC and compare to a WPC composed of pine.

The first part of the study tested small clear specimens from both live and partially deteriorated trees and looked at the effects that site location, tree height and deterioration levels has on the mechanical properties of the wood. Statistically the wood differed between the two locations from which samples were taken, but considering that the trees would not be differentiated at a processing facility, the two sites were combined. The location of the specimen within the height of the tree was also found to be statistically different, with the butt of the tree having on average 17.9% lower values than the top. After the specimens were combined from the two sites and along the tree height the two deterioration levels were statistically different. On average the live small clear specimens exhibited 7.0% higher property values than the partially deteriorated small clear specimens.

Using the results from the small clear specimen testing a mixture design was used to extrude 17 runs of WPC comprising of varying proportions of deteriorated wood flour (live, partially deteriorated, and highly deteriorated). These runs were tested in static bending and the following conditioning effects were also examined; moisture effect, high temperature effect, low temperature effect, and freeze-thaw effect. The runs were also testing for water absorption and thickness swell and were compared to a run of pine WPC, which acted as the control.

Deterioration had little effect on the mechanical and physical properties of the WPC, as MOR values tended to show no change and MOE values showed little change, only averaging a 4.5% decline. The two temperature effects were significant on both MOE and MOR values versus static bending, but this was also evident in the pine control. Freeze-Thaw effect and moisture effect had the least effect of the flexure behavior for all formulations. In all cases, the mechanical and physical properties of WPC composed of beetle-killed spruce exceeded that of WPC consisting of pine.

WPC made from beetle-killed spruce exhibited better resistance to water absorption and thickness swell, compared to pine WPC. After 8 weeks of water soak the specimens were far from being saturated with water, but some trends could be concluded. The run composed of 100% partially deteriorated wood flour performed the best of all the runs in water absorption and thickness swell, with a 4.00% and a 1.99% increase respectively. All other runs averaged an increase of 4.79% in water absorption and an increase of 2.78% in thickness swell. WPC composed of pine fared the worst with an increase in water absorption and thickness swell of 5.63% and 3.44%.

This study shows through testing and comparison of properties to a control that manufacturing WPC using beetle-killed spruce is a viable option for the million of acres of beetle-killed spruce within Alaska's Kenai Peninsula.

Recommendations

Based on the results of the WPC manufactured for this study, it is recommended that further experimentation should be conducted:

- Evaluate fastener holding properties and compare to other commercially available WPC deck boards.
- Expose beetle-killed WPC to a regime of UV exposure and fungal growth conditions.
- Examine interfacial bonding of beetle-killed WPC using SEM after moisture effect, temperature effect and freeze-thaw effect.
- Conduct creep and recovery tests on the WPC deck boards and determine if deterioration has any significant affect.
- Investigate water absorption and thickness swell properties further to determine why better qualities are seen in deteriorated spruce as compared to pine.
- Conduct duration of load test on deck boards from all deterioration levels to evaluate affect of deterioration.

Appendix A

Trees sampled to obtain clear test specimens

	Tree Specimen	Butt Diameter (in)	Top Diameter (in)	Age
Site One Live	1	10.0	6.3	95
	2	11.5	5.7	90
	3	16.5	7.1	91
	4	12.0	5.8	83
	5	11.7	5.0	85
Site Two Live	1	11.9	7.1	130
	2	9.7	6.9	130
	3	12.9	8.2	116
	4	16.2	8.0	215 (est)
	5	15.3	9.8	126
Site One Partially Deteriorated	1	14.1	6.9	105
	2	12.9	5.9	80
	3	11.6	4.5	93
	4	22.6	19.1	95
	5	16.6	6.8	88
Site Two Partially Deteriorated	1	12.3	7.1	145
	2	13.9	8.2	170
	3	10.8	6.3	140
	4	14.8	7.3	170
	5	11.7	8.4	146

Appendix B

Summary of density variations within tree height, site location, and deterioration level.

Tree Location	Number of Samples	Avg. Density kg/m3 (pcf)	Coefficient of Variation (%)
Site One (Live)	42	388 (24.2)	8.5
Butt	32	387 (24.2)	9.0
Middle	10	390 (24.3)	5.9
Top	-	-	-
Site Two (Live)	77	398 (24.8)	8.3
Butt	50	402 (25.1)	8.2
Middle	22	395 (24.7)	9.3
Top	5	374 (23.3)	9.9
Site One (Partially Deteriorated)	56	371 (23.2)	9.4
Butt	33	378 (23.6)	8.5
Middle	16	373 (23.3)	9.6
Top	7	331 (20.7)	5.4
Site Two (Partially Deteriorated)	80	395 (24.7)	10.5
Butt	47	389 (24.3)	10.6
Middle	29	398 (24.8)	11.0
Top	4	441 (27.5)	2.2

Appendix C

Paper co-authored on Wood-Thermoplastic Composites Manufactured using Beetle-killed Spruce from Alaska's Kenai Peninsula

WOOD-THERMOPLASTIC COMPOSITES MANUFACTURED USING BEETLE-KILLED SPRUCE FROM ALASKA'S KENAI PENINSULA

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ABSTRACT

The primary objectives of the study were to characterize the critical properties of wood flour produced using highly deteriorated beetle-killed spruce for wood-plastic composite (WPC) production and evaluate important mechanical and physical properties of wood-plastic composite extruded using an industry standard formulation. Chemical composition analysis indicated no significant differences in wood constituents between highly deteriorated and sound wood. Preliminary investigation with Fourier transform infrared spectroscopy (FTIR), however, indicated partial degradation or depolymerization of carbohydrate components in highly deteriorated wood compared to sound wood from green trees; effects of these changes could be seen in cell collapse and poor interaction between thermoplastic matrix and deteriorated wood fiber. Physical and mechanical properties of extruded wood-plastic composites manufactured from highly deteriorated

material were comparable to WPC properties produced using pine wood flour that served as a control material.

INTRODUCTION

In Alaska, the most damaging natural occurrence to the forests south of the Alaska Range, is the spruce bark beetle (SBB), which attacks white (*Picea glauca* (Moench) Voss), sitka (*Picea sitchensis* (Bong) Carriere), and Lutz (*Picea lutzii* Little) spruce [1-4]. Beetle infestation kills the trees and further exposes them to fungal decay and weathering over time, thus damaging the wood. In this paper, the authors examine if this damaged wood can be used in producing wood-plastic composites (WPCs).

Currently valued at nearly $1 billion (USD) annually, wood-plastic composites (WPC) is a growing market with an average annual growth of 25% a year since 1998 [5]. WPCs are primarily used in the auto industry and also the construction industry (specifically as residential decking and moulding). The primary components of a wood-plastic composite are wood in a particulate form and thermoplastic resins. An extruded WPC is comprised of 50 to 70 percent wood flour by weight. Presently, maple and pine are the most common wood flours utilized in WPC extrusion. Polyethylene (PE), polypropylene (PP), and polyvinyl chloride (PVC) are the most commonly used thermoplastics for WPC manufacturing. Besides wood and thermoplastic resins, many other additives are also used in WPC manufacturing to affect processing and performance [6-8]. Profile extrusion is the most common WPC manufacturing process. Performance of WPC depends on the inherent properties of individual components of the formulation, the type of additives, the interactions between the individual components, the processing parameters, and the end use environment. Generally, as the percent of wood filler is increased, there is an increase in the stiffness of the thermoplastic composite. Due to higher percentages of plastic, WPCs absorb less moisture and the rate of absorption is much slower than that of solid wood. The wood filler in WPCs makes them more thermally stable. These attributes make WPCs more dimensionally stable and resistant to fungal decay.

As the beetle infestation continues to spread and trees already dead continue to deteriorate, opportunities to produce primary wood products decline rapidly [9,10]. However, WPCs, which use wood flour as a feedstock, could potentially use wood flour from beetle-damaged/killed trees, with limited concern for the integrity of the solid wood source.

The primary goal of the study was to characterize raw material properties critical for WPC production and evaluate important mechanical and physical properties of wood-plastic composite extruded using an industry standard formulation. A secondary goal was to determine if height location of deteriorated wood within the tree had any significant effect on raw material and WPC properties evaluated. Specific sub-objectives to achieve the goals of this study were to: characterize chemical and thermal analysis of the highly deteriorated wood flour produced from beetle-killed spruce; describe the particle size distribution of nominal 60-mesh wood flour generated from highly deteriorated wood; evaluate the physical and mechanical properties of extruded wood-plastic composite material using highly deteriorated wood; and investigate the effect of location of highly deteriorated wood source by tree height (butt, middle, and top log) on wood flour and WPC properties.

EXPERIMENTAL DETAILS

Three geographic locations containing ten live and five dead, highly deteriorated trees were chosen from three different sites on the Kenai Peninsula, Alaska. Highly deteriorated trees were defined and evaluated based on visual characteristics. Three 2.4 m (8 ft) logs were cut from each tree: one from the base (butt log) of the tree, one from below the 10 cm (4 in) top (top log), and the third equidistant between the two (middle log). Each log was labeled with the tree number and height position within the tree. While keeping the butt, middle, and top logs separate, they were all processed into wood flour to extrude wood-plastic composite test specimens.

Wood flour preparation and analysis

Highly deteriorated logs were chipped using a Sumner Iron Works Chipper, and then hammer milled to a 60-mesh particle size using a Bliss Industries Hammermill with a screen size of 1.1684 mm (0.046 in). Wood flour was characterized for particle size distribution using a Ro-Tap sieve analyzer and then analyzed for thermal decomposition using thermal gravimetric analysis (TGA). Thermal analysis of wood flour was conducted using a Simultaneous Thermal Analyzer (STA) G25-3 from Rheometric Scientific. The pans used in the instrument were 2mm aluminum crucibles and the sample size for each run was <10mg. All wood flour was dried for 24 hours at 103°C (217°F) and then subjected to a two step heating program. The material was equilibrated at 30°C (86°F) for 5 minutes and then heated to 600°C (1112°F) at 10°C/min (50°F/min). All tests were performed in a nitrogen atmosphere with a constant flow rate of 90 ml/min. The weight loss with respect to time was monitored as the temperature was ramped. Thermal analysis results will establish the upper limits of processing temperatures during the extrusion process and will identify any changes in thermal degradation due to insect infestation and weathering over time.

Chemical analysis using wet chemistry process was performed on a sub-sample of wood flour to establish the constituent makeup of each deterioration level. Any differences found in chemical composition could be used as an explanatory variable in final WPC properties. Because of limited funds, only one sample per tree height and deterioration level was randomly selected (six specimens) and analyzed for chemical composition at Integrated Paper Services, Inc. (IPS), Wisconsin, USA. It is noted in past studies that chemical degradation, if not very severe, would not be evident in the constituent makeup (i.e., changes in polysaccharides and lignin percentages as detected through chemical analysis) [11]. Therefore, a preliminary study was also conducted where three samples from each deterioration level were analyzed for chemical composition using FTIR (Fourier Transform Infrared) spectroscopy and compared to the wet chemistry values. FTIR is more sensitive to functional groups and changes in wood chemistry that result from depolymerization of wood constituents during the wood decay process [12-14]. Spectra were collected using a ThermoNicolet Avatar 370 spectrometer from the Thermo Electron Corporation, using an attenuated total reflection (ATR) attachment and mode. The spectra were mathematically ATR corrected using the software program Omnic 7.0. The spectra for each specimen were taken as an average of 64 scans at a resolution of 4cm-1.

Wood plastic composite manufacture and testing

The wood flour was dried to less than 2% moisture content and extruded using the ExtrusionTek Milacron Twin Screw Extruder (TC-86 Extruder) at a rate of 32 rpm or approximately 454 kg/hr (1000 lb/hr) of extruded material. All the wood flour processed through the hammermill with a screen size of 1.1684 mm (0.046 in) was used in extruding WPC specimens. Average temperatures in the extruder barrel and die were maintained at 171 °C (340 ^{o}F) and 177 °C (351 ^{o}F). The WPC formulation consisted of 58% wood flour, 32% high density polyethylene (HDPE) (Petrothene® LB010000; 0.5 g/10min Melt Index; 0.953 g/cc density), 3% Zinc Serrate (lubricant), 5% Talc, and 2% Zinc Borate (biocide). Four batches of wood-plastic composite material were extruded using flour from the highly deteriorated trees; one from each of the three tree height levels (butt, middle, and top) and a fourth batch containing equal components of wood flour from all three tree height levels. A control formulation consisting of 60-mesh pine flour, a primary wood species used in commercial WPC manufacturing, was also extruded.

Six WPC specimens were prepared from each formulation and tested as per ASTM D 7031-04 [15] to determine modulus of elasticity (MOE), modulus of rupture (MOR), strain at break point, and density. Three specimens 10 x 10 x 0.64 cm (4 x 4 x 0.25 in) from each formulation were evaluated for water absorption (WA) and thickness swell (TS) properties as per ASTM D 7031-04 and ASTM D 1037-99 [16] guidelines at intervals of 24 hrs, 48 hrs, 168 hrs, 336 hrs, and 672 hrs. For the remaining three specimens, preparation for WA and TS was modified from that described in ASTM standards to test under more severe conditions. WPC specimens that were 102 mm x 102 mm (4 in x 4 in) in cross-section and 6.35 mm (0.25 in) thick were subjected to WA and TS tests instead of using the full cross section of the as-extruded WPC material. This modified specimen size results in a more severe test than that recommended by ASTM as it could potentially expose more wood fiber to moisture due to disruption of encapsulation during the planing (surfacing) process.

RESULTS AND DISCUSSION

Wood Flour Particle Size Analysis

Viscosity of the formulation during WPC production and interaction between wood flour and the thermoplastic are partly determined by the particle size of the wood flour used. As mechanical interlocking between wood fiber and thermoplastic is the primary method of interaction between the two WPC constituents, particle size also relates to the final structural qualities. Wood particles are used as inexpensive fillers in WPCs and are known to contribute to WPC stiffness. Stark and Rowlands [17] note that the aspect ratio of wood particles increases as mesh size used in wood flour preparation increases. WPC stiffness improves significantly as aspect ratio of wood particles increases. Besides WPC stiffness, wood flour mesh size also affects the impact energy with larger particles leading to higher stress concentrations. Since wood flour was generated starting with highly deteriorated spruce in this study, it was of interest to understand the array of particle sizes produced when a 60-mesh screen was used during hammer milling. It is hypothesized that compared to wood flour from live spruce wood, highly deteriorated spruce wood may lead to different distribution in particle size as breakage of fiber could be influenced by deterioration of fibers. Figure 1 shows the particle size distribution by log position for highly deteriorated wood and of wood flour generated from live spruce trees. Close observation indicates a greater percent of finer particles (~40%) in case of wood flour from highly deteriorated spruce than that of wood flour from live spruce (~25%). All the wood flour that passed through 40- and 60-mesh is considered fine in this case. Wood degradation due to weathering and decay fungi and moisture loss are probable reasons for this generation of higher percentage of finer particles.

Thermogravimetric Analysis

Thermal decomposition tests were administered on wood flour from the three log positions (bottom, middle, top) using Thermogravimetric Analysis (TGA) to determine an optimum processing temperature in the production of the WPC. In all three log locations, the temperature at which thermal decomposition began to occur was around 225°C (437°F) and maximum thermal decomposition occurred at 361°C (682°F) (Figure 2). This was well above the processing temperatures used in the extruder during production, which was around 171°C (340°F) in both the barrel and screw chambers. No trends in thermal decomposition behavior were detected due to log location.

Chemical Analysis

Chemical analysis was performed to determine if decay fungi had altered the proportions of cellulose, hemicellulose and lignin in the beetle-killed wood and whether these proportions varied with height position in the tree. Table 1 presents the percentage of chemical constituents in the highly deteriorated wood and the analysis of wood from live trees. Only minor changes in proportions of chemical constituents were observed. Brown-rot fungi are known to attack softwoods where they mainly decompose the polysaccharides and cause only a small loss in the lignin [11]. Often, in the initial stages of decay, polysaccharides are depolymerized to low-molecular-weight and soluble molecules; thus, chemical analysis would not necessarily indicate a change in the chemical composition. Previous work on Kraft pulp yield, which can be sensitive to changes in chemical composition of carbohydrates in particular, showed no significant difference when comparing yields from live trees and beetle-killed trees that had been dead for 50 years [18].

Fourier transform infrared (FTIR) spectroscopy was used on wood flour from both highly deteriorated and live logs to distinguish if any chemical changes occurred as a result of deterioration. The FTIR spectra for live and highly deteriorated beetle-killed spruce are shown in Figure 3. Within the fingerprint region (1800 cm^{-1} to 600 cm^{-1}), several peaks that indicate changes in wood constituents resulting from the decay process are identified [13,14, 19-22]. Further examination of the FTIR spectra (Figures 4a, 4b, and 4c) indicate that the band peaks at 793 cm^{-1}, 898 cm^{-1}, 1318 cm^{-1}, 1368 cm^{-1}, 1422 cm^{-1}, and 1733 cm^{-1}, assigned to the carbohydrates such as cellulose ring vibrations, became weaker with deterioration of beetle-killed spruce wood. The disappearance of these bands is evident of qualitative changes in lignin and carbohydrate components in highly deteriorated wood. In the functional group region (4000 cm^{-1} to 1800 cm^{-1}) there are two prominent peaks shown, first being a strong hydrogen bonded O-H stretching absorption at 3350 cm^{-1} (Figure 3) and second at 2895 cm^{-1} (Figure 4d) indicating C-H stretching absorption. Bands that exhibit visible variations between live and highly deteriorated wood are 3350 cm^{-1} and 2895 cm^{-1}. While white rot fungi degrade cellulose, hemicellulose, and lignin components of wood, brown rot fungi are preferential degraders of the carbohydrate components of wood and are responsible for extensive depolymerization of cellulose early in the decay process. Initial analysis using FTIR spectroscopy confirms qualitative changes in carbohydrate components in highly deteriorated beetle-killed spruce wood.

Mechanical and Physical Properties

The mechanical properties of the extruded WPC test samples indicated that when compared to the pine controls, the MOE, MOR, strain at failure, and density of WPC from beetle-killed material were all close in value (Table 2). Very little variation in density was observed among all the specimens irrespective of the type of wood flour used (coefficient of variation was less than 0.5%). Comparison of means tests at α-level of 0.05 indicated no significant difference among the means of three tree height levels for density, MOE, and MOR, and strain at failure. However, MOE and MOR of WPC produced using highly deteriorated spruce wood flour were significantly lower from corresponding properties of WPC produced using pine wood flour irrespective of tree height level. Although these differences are statistically significant, they are not practically significant and could be controlled through formulation design, a flexible manufacturing feature of wood-plastic composite technology. WPC specimens produced using pine flour yielded higher MOE values than those produced from highly deteriorated spruce flour, but lower MOR values.

Scanning electron microscope (SEM) micrographs of a highly deteriorated spruce fiber bundle in a matrix of HDPE are shown in Figures 5(a) and 5(b). A clear separation between wood fiber and thermoplastic matrix is evident showing poor interaction between wood and plastic. These figures also show collapsed wood fibers with very little penetration of HDPE into cell lumens. Figure 5(c) illustrates typical penetration of thermoplastic into cell lumens of pine flour. It is hypothesized that cell wall deterioration as a result of brown rot attack after insect infestation caused wood fibers of highly deteriorated spruce to collapse more readily from processing pressures in the extrusion process. Even with poor interlocking between wood and thermoplastic matrix, WPC specimens produced with highly deteriorated wood exhibited reasonably good mechanical properties when compared to WPCs extruded with pine flour. Greatly increased surface area as a result of higher proportions of fines generated with deteriorated wood could have influenced nucleation of the matrix polymer.

Because the hydrophilic nature of wood and intended use of WPC for exterior applications, water absorption testing can provide an indication of how the material will perform in such a setting. Figure 6 shows water absorption following 4 weeks of water submersion, where readings were taken at intervals (24 hrs, 48 hrs, 1 week, 2 weeks, 4 weeks) to help establish trends for WPC specimens extruded with highly deteriorated spruce and control pine flour. The WPC composed of strictly wood flour from the butt log of the tree exhibited the highest water absorption, with a 17.55% increase in moisture content after 4 weeks. A similar water uptake value (16.96%) was found for WPC specimens extruded with wood flour that is a combination of equal proportions of the three log locations (butt, middle, and top). The WPC manufactured using wood flour from the middle section and the top sections of the tree exhibited increases of water absorption of 15.71% and 15.93% respectively. Generally the greatest increase in water absorption occurred between 2 and 4 weeks. Water absorption of WPC specimens with control pine flour was greater than that of deteriorated spruce wood flour over time, but the two begin to converge at the end of four weeks indicating a slower water absorption rate for highly deteriorated spruce specimens. It is interesting to note that WPC specimens with pine flour began to plateau at around 18% which would be equivalent to 31% if normalized to a specimen composed of entirely pine wood flour (calculated with the assumption that only wood flour in WPC contributes to moisture uptake and thickness swell and not HDPE matrix). This value seems reasonable considering that fiber saturation point for all woods is approximately 30%. Results indicate that wood flour from deteriorated spruce wood tends to absorb water at a slower rate than pine wood flour. Collapse of spruce fiber bundles and greatly increased surface area, as a result of higher proportions of fines generated with deteriorated spruce wood, may have contributed to decreased moisture sorption because of better encapsulation by thermoplastic matrix. If immersed for a longer time period, it is possible that moisture absorption of WPC specimens with deteriorated spruce wood flour would also begin to plateau around fiber saturation point of spruce wood.

Similar trend as in water absorption is also evident with thickness swell data. The WPC made with equal parts wood flour from the three log positions (butt, middle, top) showed the greatest increase in thickness swell, with an 8.48% increase from its initial thickness (Figure 7). The WPCs composed of wood flour from the butt, middle, and top showed a gradual decrease in thickness swell from the butt to the top, each showing an increase of 8.23%, 7.78%, and 7.72% respectively after 4 weeks of water immersion. The overall difference between the butt and the top was 0.51%. Once again, the cell wall of highly deteriorated spruce fibers indicated a significantly lower uptake in moisture compared to control pine flour. Collapse of fiber bundles and better thermoplastic encapsulation of finer deteriorated spruce wood flour could have contributed to this behavior. Past research [23] has shown that moisture absorption of wood flour-filled polypropylene composite decreased with decreasing particle size. Slower rates of water absorption and thickness swell results for shorter exposure durations of WPC specimens with deteriorated spruce wood flour point to a possibility of improving WPC moisture resistance by partial inclusion of highly deteriorated spruce flour in the formulation.

CONCLUSIONS

Results show that manufacture of WPC from highly deteriorated spruce trees is a viable opportunity for utilization of beetle-killed spruce trees. Chemical composition analysis did not show any significant differences in constituent make-up between highly deteriorated and live spruce wood; however, signs of break down of chemical constituents due to decay fungi attack was evident in the FTIR spectra. Wood flour produced from deteriorated spruce wood contained greater proportions of fines than control pine wood flour. Nevertheless, mechanical properties were comparable to WPC properties manufactured from pine wood flour, which served as the control. SEM micrographs show collapse in fiber cells and evidence of poor penetration of HDPE into cell lumens of spruce wood flour attributable to degradation of fiber quality in deteriorated trees. Moisture uptake and thickness swell rates of WPC specimens produced with highly deteriorated spruce flour were significantly lower than those with control pine flour possibly due to better barrier provided by thermoplastic encapsulation of finer particles and collapse of fiber bundles during extrusion process. Further research on interaction between highly deteriorated spruce fiber and thermoplastic matrix is necessary to understand this physical behavior.

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Table 1. Carbohydrate, lignin and extractive content vs. tree position for highly deteriorated and live spruce from Alaska's Kenai Peninsula.

Chemical constituent	Tree Condition					
	Highly deteriorated			Live		
	Log Position					
	Butt	Middle	Top	Butt	Middle	Top
Hemicellulose (%)	21.2	20.6	21.8	18.0	18.5	22.0
Cellulose (%)	42.9	41.5	43.0	37.0	37.6	43.7
Lignin (%)	28.5	28.5	28.7	28.1	27.3	27.0
Extractive (%)	1.46	2.37	2.07	2.07	2.93	2.13

Table 2. Mechanical properties vs. tree position for wood plastic composites manufactured from highly deteriorated spruce from Alaska's Kenai Peninsula.

Position within tree	Avg. Density kg/m3 (pcf)	COV[1] (%)	Avg. MOE[2] GPa (psi)	COV (%)	Avg. MOR[3] MPa (psi)	COV (%)	Strain at Failure (%)
Butt Log	1153 (72)	0.3	4.11 (595,750)	4.0	23.1 (3,350)	1.7	0.99
Middle Log	1153 (72)	0.2	4.13 (599,250)	2.6	23.1 (3,350)	2.4	1.00
Top Log	1153 (72)	0.3	4.13 (598,650)	1.5	23.0 (3,340)	1.7	1.00
All combined	1169 (73)	0.4	4.01 (581,450)	0.9	22.8 (3,300)	0.8	1.01
Pine (control)	1153 (72)	0.2	4.29 (621,800)	0.6	21.9 (3,180)	1.3	1.03

[1] Coefficient of Variation
[2] MOE = Modulus of Elasticity
[3] MOR = Modulus of Rupture

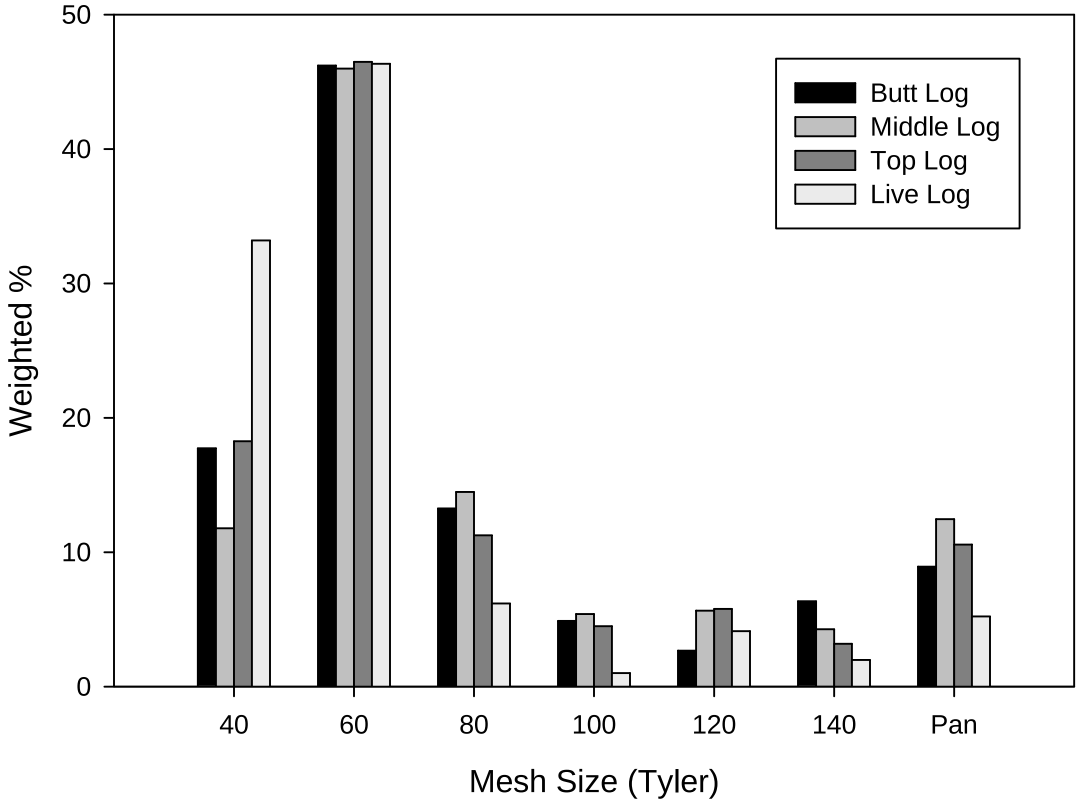


Figure 1. Particle size distribution of spruce wood flour by log position using 1.1684mm (0.046") screen.

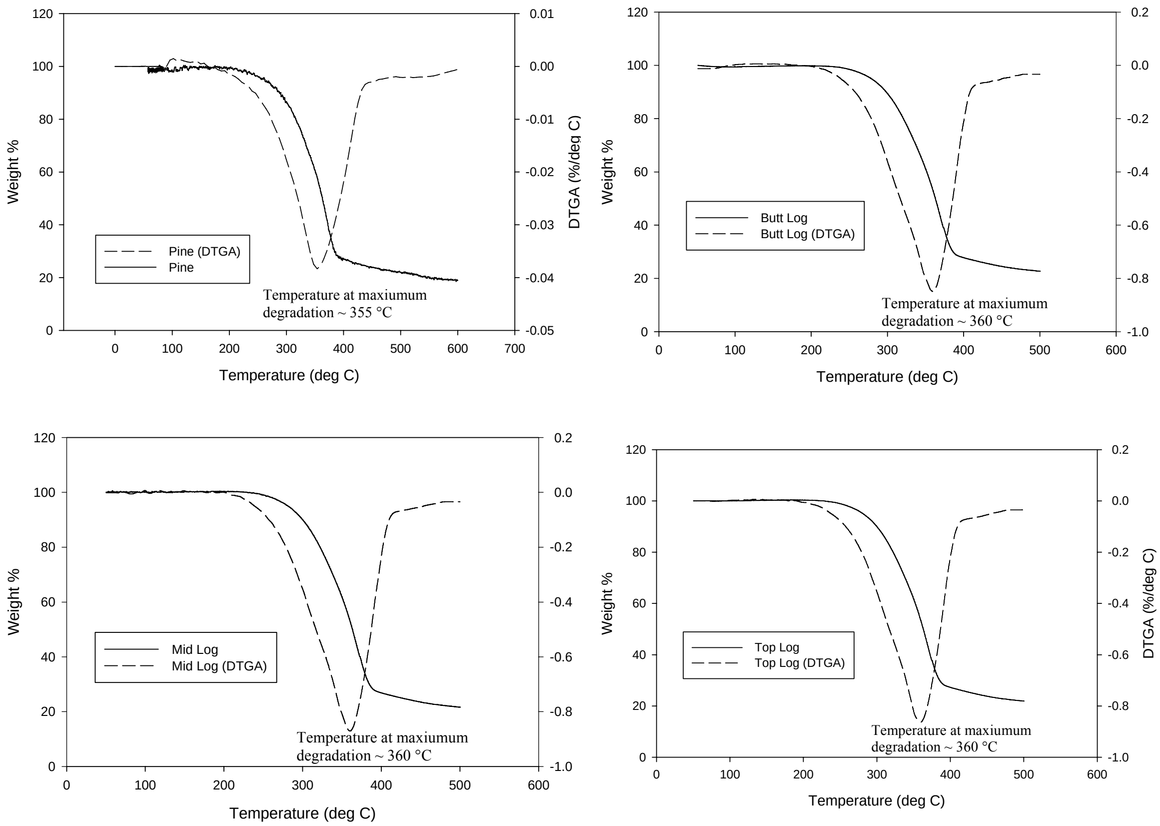


Figure 2. Thermal degradation analysis of pine and highly deteriorated spruce wood flour.

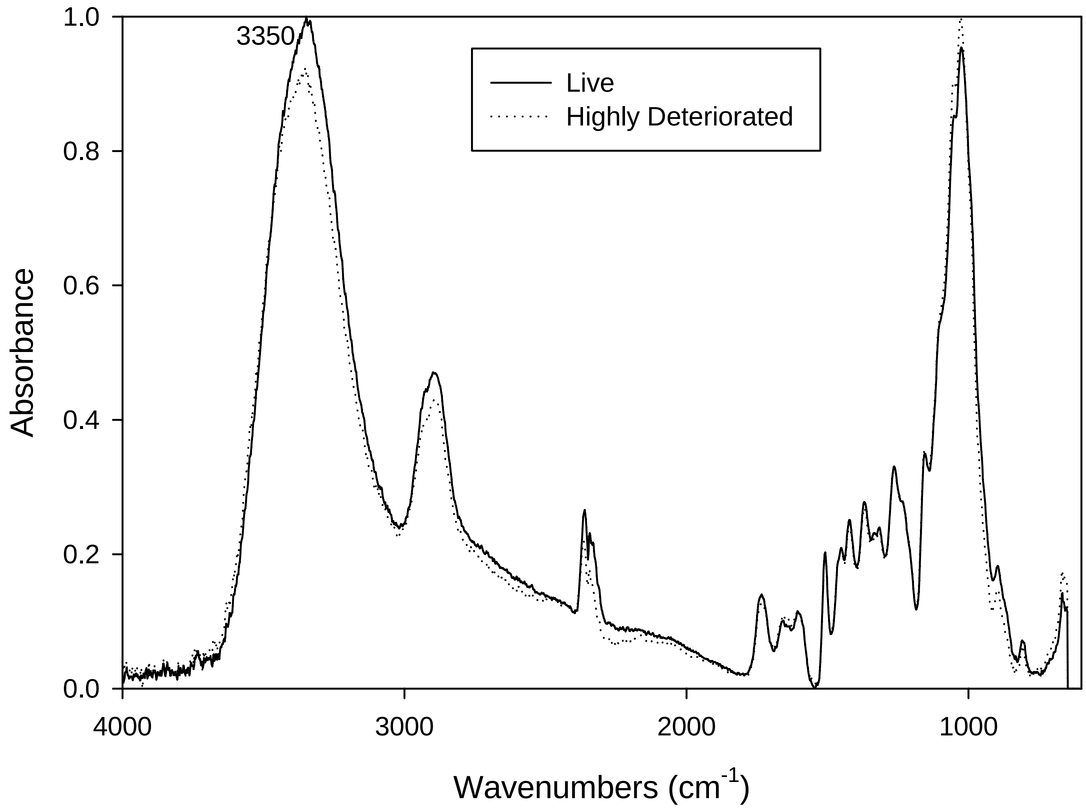


Figure 3. FTIR absorption spectra of live spruce and highly deteriorated beetle-killed spruce.

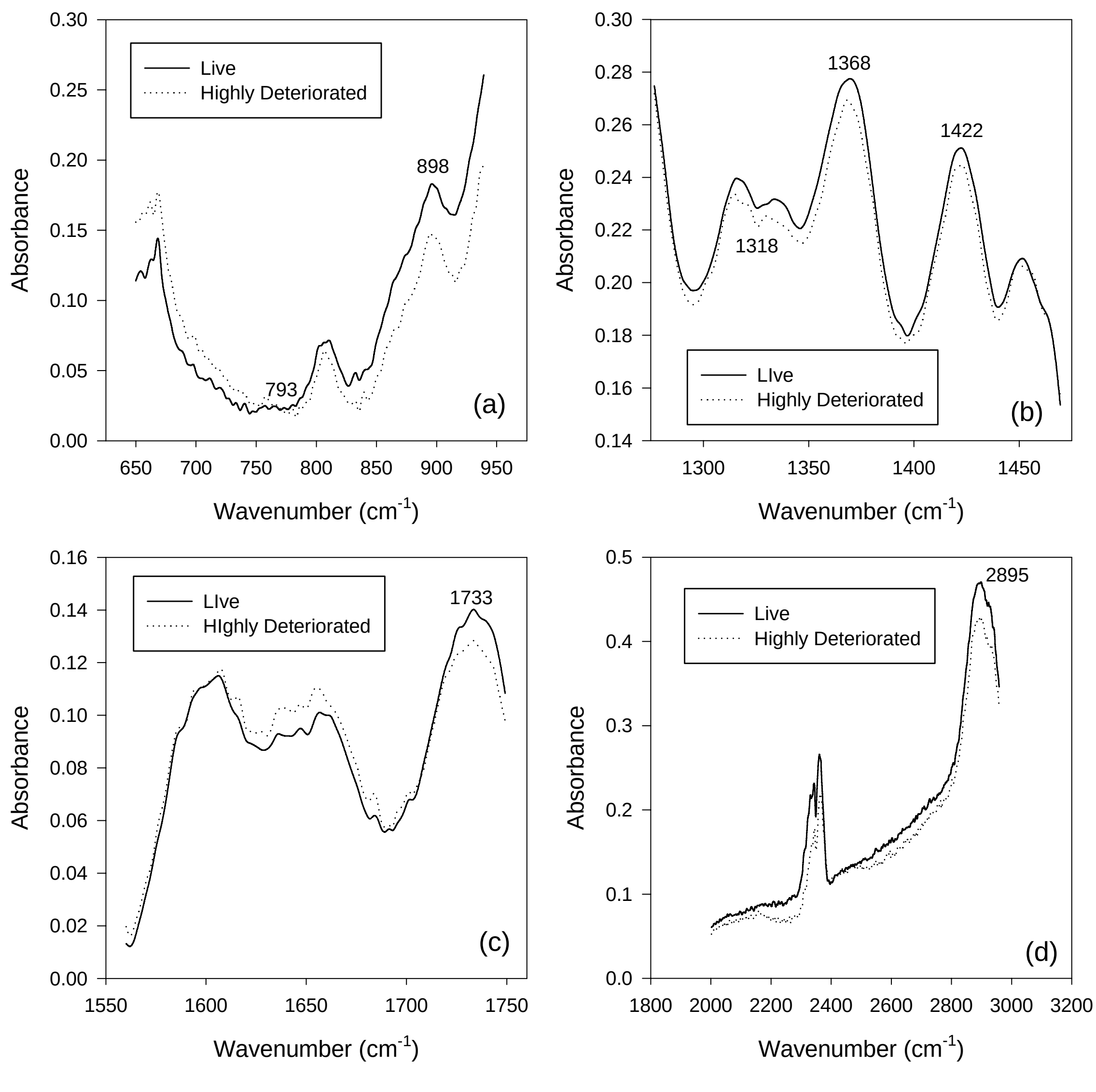
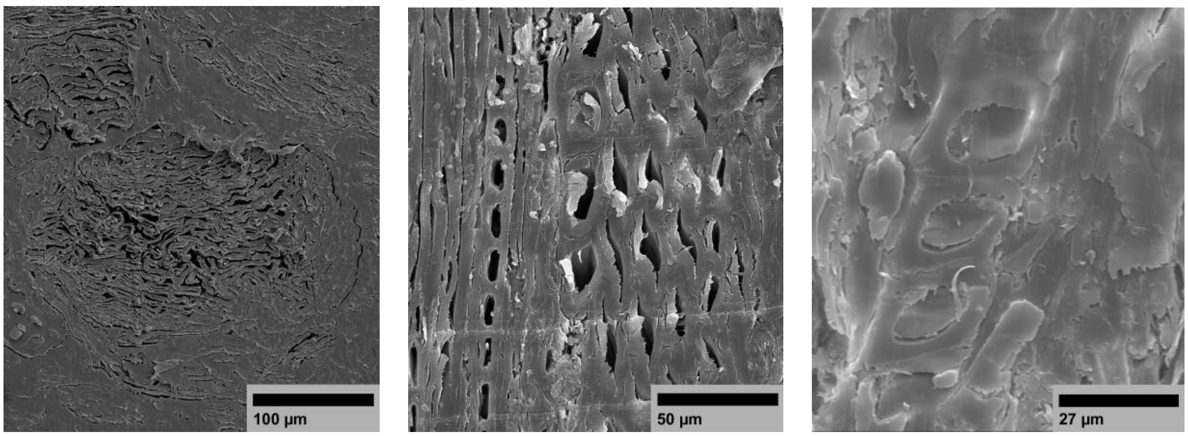


Figure 4. Detail comparison of live and highly deteriorated beetle-killed spruce FTIR absorption spectra within different sub-regions of the spectra presented in Figure 3.



(a) (b) (c)

Figure 5. Micrograph showing (a) cell collapse of deteriorated spruce fiber bundles, (b) poor penetration of HDPE matrix into fiber lumen of deteriorated spruce fiber, and (c) typical penetration of thermoplastic into cell lumens of pine fiber.

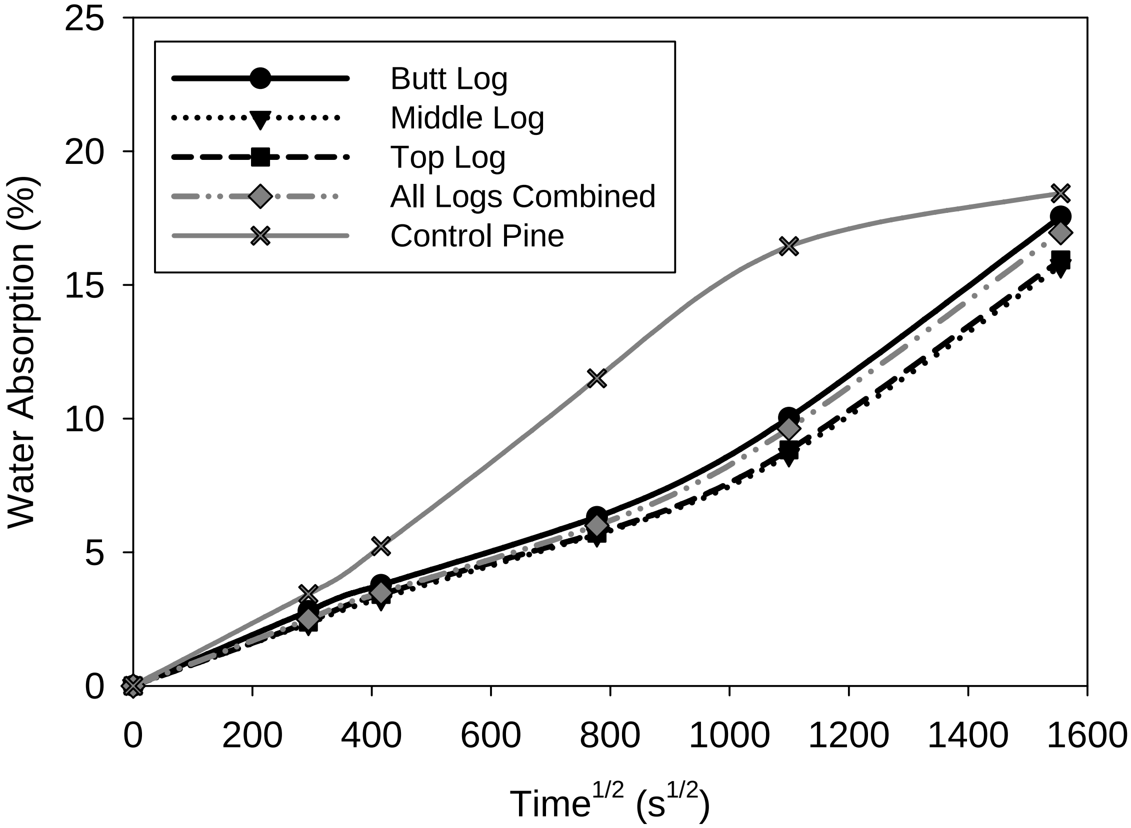


Figure 6. Water absorption, by log position, of wood plastic-composites manufactured from highly deteriorated beetle-killed spruce and pine as a comparison .

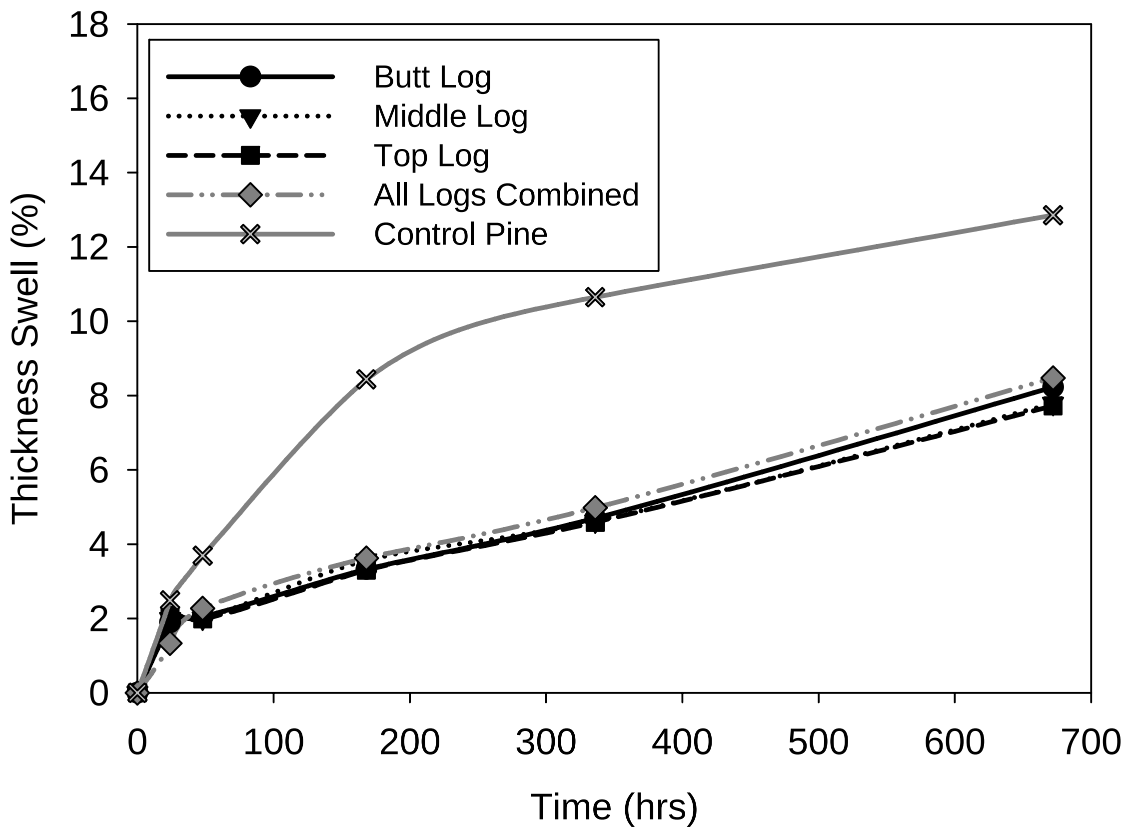


Figure 7. Thickness swell, by log position, of wood plastic-composites manufactured from highly deteriorated beetle-killed spruce and pine as a comparison.