The Effect of Mountain Pine Beetle Killed Wood on Pulp Fibre Quality:
An Exploratory Study

by

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ABSTRACT

Lodgepole pine stands harbour a major commercial significance in British Columbia. The lodgepole pine forests not only make up half of the British Columbia Interior annual harvest, but are also highly utilized and acceptable for lumber and pulp production. Unfortunately, mature lodgepole pine stands have been surmounted by outbreaks of mountain pine beetle, which is the most destructive insect to pine. In addition, mountain pine beetle epidemic outbreaks can last for numerous years depending on climatic factors; damaging vast areas of lodgepole pine forests and making long term planning for timber supply and integrated resource management arduous.

Currently, the loss of lodgepole pine due to mountain pine beetle infestation is substantial, and as an attempt to battle the crisis, abundant volumes have been harvested. However, previous work has concurred that wood from dead trees affect product quality, which is the primary concern for the pulp and paper industry. Currently, there is a lack of information on the mechanism and effects of mountain pine beetle on pulp fibre quality. In order to fully utilize the mountain pine beetle killed resource, it is crucial to understand how the pine beetle impacts fibre quality. As a result, the present investigation evaluates the effects of mountain pine beetle killed wood on wood morphology, chemistry and pulping properties.

Prior to experimental analysis, a dead (infested by mountain pine beetle) and sound standing lodgepole pine tree were harvested from the same site and segregated into sapwood and heartwood at different positions of the tree. Moisture content and density analysis demonstrated that infested sapwood and heartwood exhibited significantly reduced moisture content and lowered density, as tree height increased. The chemical analysis indicated that infested sapwood contained less extractives, lignin and carbohydrates when compared to sound sapwood. These results are likely due to the mountain pine beetle and subsequent blue stain fungi infestation.

Extractive content typically increased with tree height whereas, the carbohydrate content tended to decrease. In general, no clear trend was apparent for the heartwood chemical analysis. Consequently, the permeability analysis demonstrated that infested sapwood was more permeable than sound sapwood; while sound heartwood was more permeable than infested heartwood. These results are likely due to the fact that fungal hyphae were present in infested sapwood and numerous aspirated pits were found in infested heartwood as observed from the microscopic analysis.

Moreover, the chip quality analysis revealed that the infested wood typically generated more fines than sound wood; however, tree height did not appear to affect chip quality. Similarly, the pulp quality analysis demonstrated that the infested wood had a lower kappa number, generated a higher pulp yield and consumed less alkali compared to sound wood. The changes in pulp quality can be attributed to the changes in wood chemistry. Additionally, paper quality analyses revealed that paper from the infested wood had lower burst and tensile indices and higher tear strengths. Paper quality from the infested wood was also more porous, less smooth and less dense. The fibre quality analysis suggest that differences in paper quality made from infested wood may be due to the original fibre attributes rather than the effect of pine beetle or blue stain fungi, in that the results indicated that the infested wood fibres were longer and coarser. Clearly, mountain pine beetle infested wood affects wood quality, however, the differences between sound and infested wood are not so significant that the beetle killed resource cannot be utilized.

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

LIST OF ABBREVIATIONS

°N	degrees North
°C	degress Celsius
°C/min	degrees Celsius per minute
kPa	kilopascal
kg/m^3	kilograms per cubic metre
kg	kilograms
kV	kilovolts
%	percent
μL	microlitre
μm	micrometer
g	grams
g/cm^3	grams per cubic centimeter
g/m^2	grams per square metre
GC	gas chromatography
GDP	gross domestic product
HPLC	high performance liquid chromatography
H_2SO_4	sulphuric acid
hr	hour
L	litre
M	molar
m	metre
m^3	cubic metre
mm	millimetre
mg/L	milligrams per litre
mg/m	milligrams per metre
min	minute
mL	millilitre
mL/min	millitre per minute
mM	millimolar
mN	millinewton
$mN{\cdot}m^2/g$	millinewton square metres per gram
nm	nanometres
N	normal
$N{\cdot}m/g$	Newton metres per gram
NaOH	sodium hydroxide
$Na_2S_2O_3$	sodium thiosulphate
sec	second
sec/100mL	seconds per 100 millilitres
UV	ultraviolet

ACKNOWLEDGEMENTS

CHAPTER 1. INTRODUCTION AND RESEARCH OBJECTIVES

1.1 Global Forestry

In 2000, the total annual global industrial wood fibre production surpassed 1.5 billion cubic metres. It is anticipated that a further 20 to 50% in supply will be required by 2020 in order to meet the demands of the increasing world population, which is expected to reach 7.5 billion by the year 2020 (Natural Resources Canada, 2002). Fibre consumption is projected to increase at an annual rise of 3.2%. Requirement of wood, particularly for pulp and paper consumption, are also projected to double in the same period (Food and Agriculture Organization, 1994). As the world's largest exporter of manufactured forest products (softwood lumber, wood pulp and newsprint) and steward of 10% of the world's forests, Canada will also be subject to pressure as it endeavours to meet the predicted global demand for wood and fibre shortage (Natural Resources Canada, 1999; Council of Forest Industries, 2000).

1.2 Forestry in Canada

Canada's 417.6 million hectares of forest presents a $74-billion industry, and therefore is a prominent contributor to the national economy. Of Canada's forest land, the predominant species are coniferous forests, such that they make up 67% of the forest land (Natural Resources Canada, 2002). Approximately one million hectares or 0.4% of Canada's commercial forests are harvested yearly. In addition, roughly 1.6% of the Canadian forest land is affected by fire, insects and disease each year. For example, in 1999, 6.3 million hectares of forest land in Canada were defoliated by insects (Natural Resources Canada, 2002), of which 2.4 million hectares were affected by bark beetles and insect defoliators located in British Columbia alone (British Columbia Ministry of Forests, 2000).

British Columbia (BC) is a key contributor to the Canadian economy such that it accounts for 46.7% of the total softwood lumber production, 31.5% of the total pulp production, 10.7% of the total paper production, and 80% of the total plywood production in Canada. This substantial industry contributes 20.7% to BC's Gross Domestic Product, and 3% to the total Canadian GDP, making it one of the largest industrial contributors to Canada's economy. BC forests are predominantly coniferous, such that they account for 94.6% of the total tree volume, of that lodgepole pine comprises the largest volume of logs harvested and therefore is a prominent species to the forest industry and the provincial economy (Council of Forest Industries, 2000).

As the focus of this study is on the lodgepole pine species, an introduction to the characteristics of softwood anatomy will be provided. Wood is an anisotropic material in regards to its anatomical, physical and chemical properties. Therefore, a basic knowledge of its structure is of great importance, as these properties dictate its utilization.

1.3 Softwood Anatomy

1.3.1 Macroscopic Structure

Wood (or xylem) is the vascular support system of a tree. The basic structure includes the roots, crown, stem (or bole) and the outer layer of bark. Between the inner bark and sapwood, is a thin layer of tissue called the cambium (Figure 1-1). The sapwood is physiologically active as it contains living cells (parenchyma), which provide storage of starch or fat, and is in continuous communication with the cambium and phloem through sap flow from the crown (Fujita and Harada, 2001). The sapwood provides structural support, serves as a food and nutrient storage reservoir, and provides a means for water conduction from the roots. As sapwood cells age, the majority of the parenchyma cells die, and often change colour. The dead cells form the heartwood portion and heartwood mainly functions as mechanical support. Heartwood is normally darker in colour due to the deposition of resinous organic compounds in

the cell walls and cavities also known as extractives. At the center of the tree is a small core of parenchymatous tissue called the pith (Smook, 1992; Stenius, 2000).

1.3.2 Wood Formation

Wood is produced from cell divisions in the vascular cambium using energy derived from the products of photosynthesis. After cell division in the cambium, each successive cell undergoes enlargement, cell wall thickening and lignification. The cambium actively divides cells between the xylem and phloem. The rate of cell division and the final cell size is regulated by growth regulating hormones called auxins. The light coloured portion of the growth ring, formed in the early part of the growth season, contains earlywood cells, which typically have large cross-sectional areas, thin cell walls and large lumens (the open center of the cell). The darker portion, formed in the latter part of the season, contains latewood cells, which typically have smaller cross-sectional areas, thicker cell walls and narrower lumens compared to earlywood (Figure 1-2). Generally, the earlywood to latewood ratio increases with tree height. There are more latewood cells present near the base of the tree as it is farthest away from the source of auxin supply. The amount of earlywood and latewood content affects the pulp and paper industry, as thin-walled earlywood cells are more flexible than thick-walled latewood fibres. Thus, earlywood cells are more collapsible than the stiff and rigid latewood cells and therefore produce better quality paper (Stenius, 2000; Smook, 1992; Jozsa and Middleton, 1994).

In addition, earlywood cells have many bordered pits on the radial face and this is due to the greater degree of physiological activity during their formation (Figure 1-2). Latewood cells generally have smaller and a lot fewer bordered pits. Pits are recesses in the cell wall between
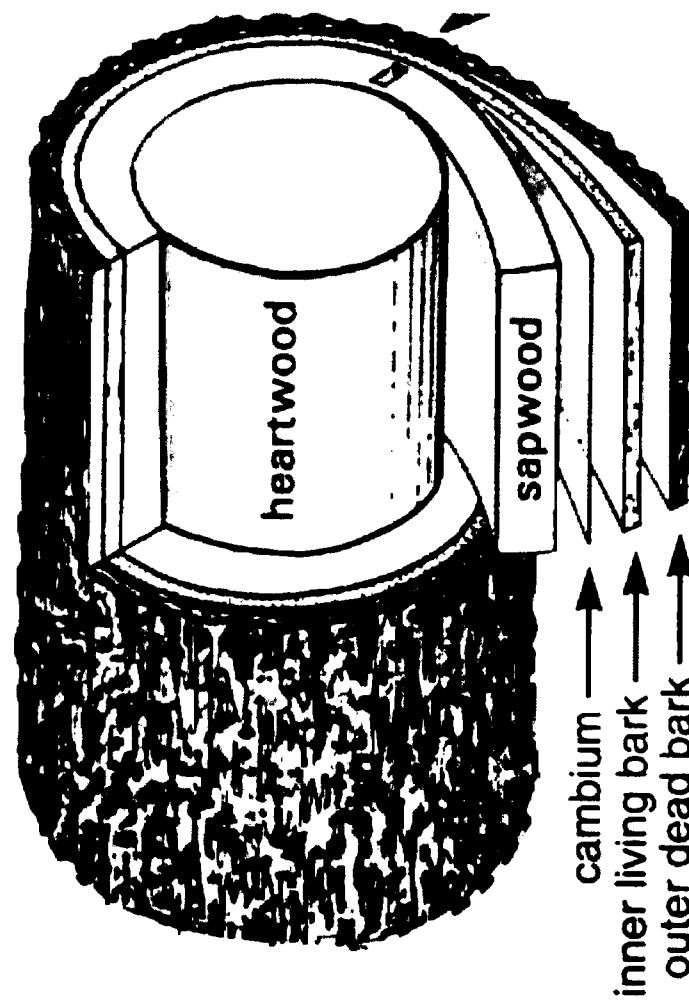
Figure 1-1. Primary tissues types in a tree cross section (Jozsa and Middleton, 1994).
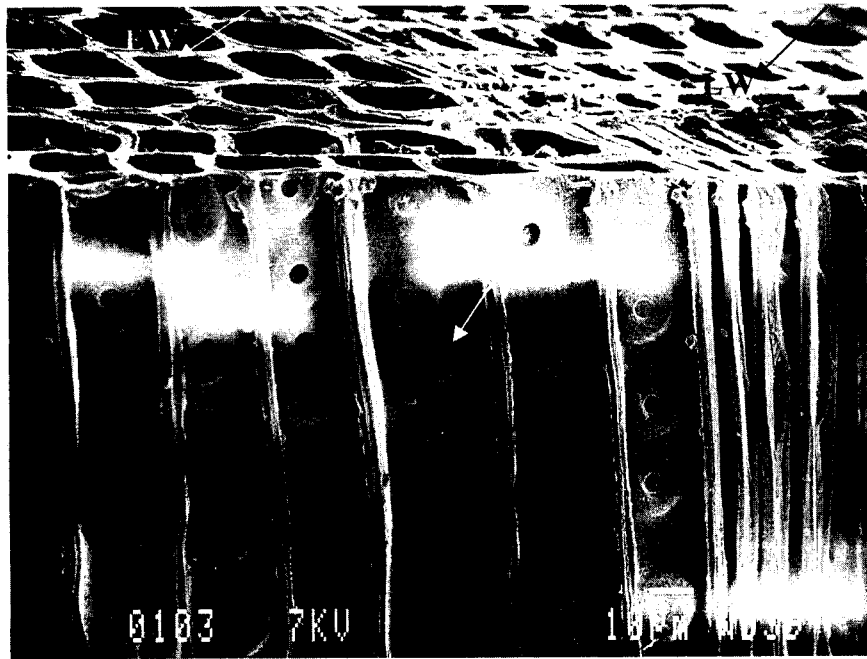
Figure 1-2. Scanning electron micrograph showing the transverse and radial face of lodgepole pine (600 × magnification). EW = earlywood, LW = latewood and BP = bordered pits.

adjacent cells, and are formed during primary cell growth. Pits are responsible for water conduction in the tree (Sjostrom, 1993; Stenius, 2000).

1.3.3 Juvenile Wood

Juvenile wood is intimately associated with the pith of the tree, and is therefore often referred to as pith wood. Juvenile wood displays significantly different wood attributes when compared to mature wood, and thus plays an important role in wood quality characteristics. The formation of juvenile wood varies between species, but often occurs during the first 5-30 years of growth. Juvenile wood affects quality such that it has lower density, shorter fibres, larger microfibril angle, higher earlywood to latewood ratio, slightly lower cellulose content and higher lignin content than mature wood (Stenius, 2000; Sjostrom, 1993). Typically, the lower portion of the stem below the live crown and around the juvenile core is a shell of mature wood whereas in the crown wood is mainly juvenile wood (Figure 1-3).

1.3.4 Cell Types and Cell Wall Organization

Typically, there are two categories of cell types found in coniferous trees: prosenchyma and parenchyma cells. Softwoods are generally composed of longitudinal tracheids (90-95%), which are long tapered prosenchyma cells that provide mechanical support and have a major role in conduction. In contrast, parenchyma cells are generally rectangular in shape and are relatively short and provide storage and reserve for food supplies (Stenius, 2000). In pines, resin canals are also present, which are tube-shaped, intercellular cavities lined with epithelial parenchyma cells that secrete oleoresin (lipophilic gum) into the canal. Pines also have both axial and radial resin canals that form intercommunicating system of ducts in the stem (Bark and Allen, 2000).

Wood is a complex of cellulose, lignin, hemicelluloses, and extractives. The cell wall of a typical fibre is made up of several layers (Figure 1-4). Panshin and Zeeuw (1980) describe the cell wall as an intricate complex of stranded polysaccharides embedded in lignin to form a

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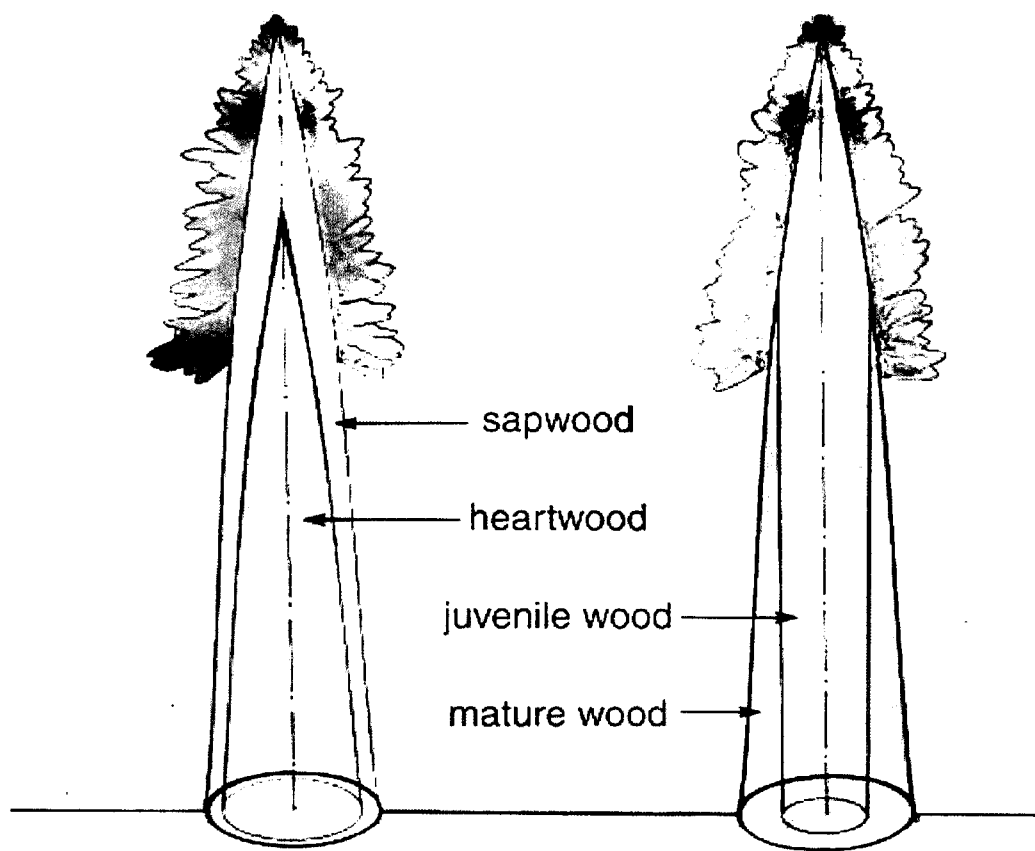
Figure 1-3. Diagrammatic representation of sapwood, heartwood, juvenile wood and mature wood distribution (Jozsa and Middleton, 1994).
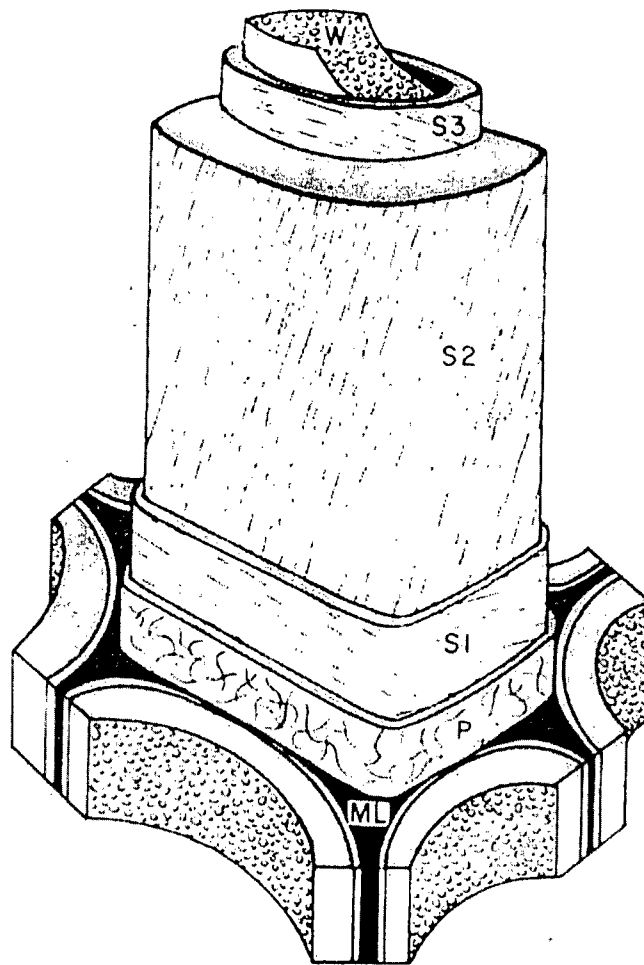
Figure 1-4. Schematic representation of wood cell wall layers illustrating the middle lamella (ML), primary cell wall (P), secondary cell wall layers (S_1, S_2, S_3) and warty layer (W) (Sjostrom, 1993).

rigid cell wall. The cell wall is composed primarly of microfibrils, which are long strands of crystalline cellulose surrounded by short-chain hemicelluloses, and form sheets (lamellae) that lie parallel to the surface wall. The deposition of lignin and extractives, after microfibril formation, binds and encloses the microfibrils into a rigid structure as well as microcapillaries (void spaces) that can form between microfibrils that lack complete lignification. This ultrastructural organization thereby allows water to penetrate into the cell walls. Microfibrils are also arranged in sheets (lamellae) that lie parallel to the surface wall (Panshin and Zeeuw, 1980; Haygreen and Bowyer, 1996; Fujita and Harada, 2001).

The middle lamella separates two contiguous fibres and is composed of pectin and lignin. Each fibre has a primary wall (P) and a three-layered secondary wall with specific microfibril alignments. The primary cell wall, which is composed of microfibrils that are dispersed in an irregular, loosely interwoven pattern, is thin and is generally associated with cellular enlargement. The secondary cell wall makes up the bulk of the cell wall as it forms three distinct layers (S_1, S_2, and S_3). The S_1 and S_3 layers are generally 4-6 lamellae layers thick, whereas the S_2 varies from 30-40 lamellae layers thick in earlywood cells and approximately 150 lamellae layers thick in latewood. The S_2 layer is the thickest of the three layers and is primarily responsible for the overall strength of the cell wall. The warty layer, which is the innermost layer facing the lumen, is comprised mainly of lignin (Smook, 1992; Sjostrom, 1993; Stenius, 2000).

1.4 Lodgepole Pine

1.4.1 Distribution and Importance

Lodgepole pine stands dominate much of western Canada and the United States, covering over 26 million hectares of forest land. As the most widely distributed conifer in western North America, it is a member of the family Pinaceae and of the genus *Pinus*. Lodgepole pines are

recognized as *Pinus contorta* and are comprised of four widely distributed subspecies with the following ranges:

1. *P. contorta* ssp. *contorta*: Pacific Coast
2. *P. contorta* ssp. *murrayana*: Southern Cascades, Sierra Nevada and the Southern Baja Californian Mountains.
3. *P. contorta* ssp. *latifolia*: Rocky Mountains and Intermountain Regions, northern Cascades, Washington.
4. *P. contorta* ssp. *bolanderi*: Mendocino White Plains along Northern California coast (Koch, 1996).

Several characteristics help to differentiate the subspecies from one another including: form, branchiness, longevity of the tree, needle characteristics, cone traits, seed characteristics and dimensions (Wheeler and Critchfield, 1985; Koch, 1996).

Since lodgepole pine is widely distributed and highly variable; it has a latitudinal range that spans 33°, longitudinal range that spans 35° and its elevational limit is approximately 3900m (Figure 1-5) (Wheeler and Critchfield, 1985; Koch, 1996).

The Canadian lodgepole pine resource is three-times greater than that of the United States, with roughly 75% of the total Canadian growing-stock in BC. In Canada, lodgepole pine trees expand over BC, western Alberta, the southern Yukon, and minor volumes are found in the southwest corner of the Northwest Territories. However, most of the merchantable-sized trees are located in BC, which accounts for approximately three-quarters of Canada's total merchantable volume. More specifically, in BC, lodgepole pine represents more than 22% of the total mature standing timber volume, located in three regions: Prince George, Bulkley-Northwest and Cariboo. Although volumetrically, lodgepole pine is only slightly exceeded by the spruces in terms of total volume of mature standing timber, it ranks first among species harvested as well as total volume of log produced in BC. In addition, the stumpage value of lodgepole pine in BC is lower than the other ten most commonly harvested species originally due to its smaller tree
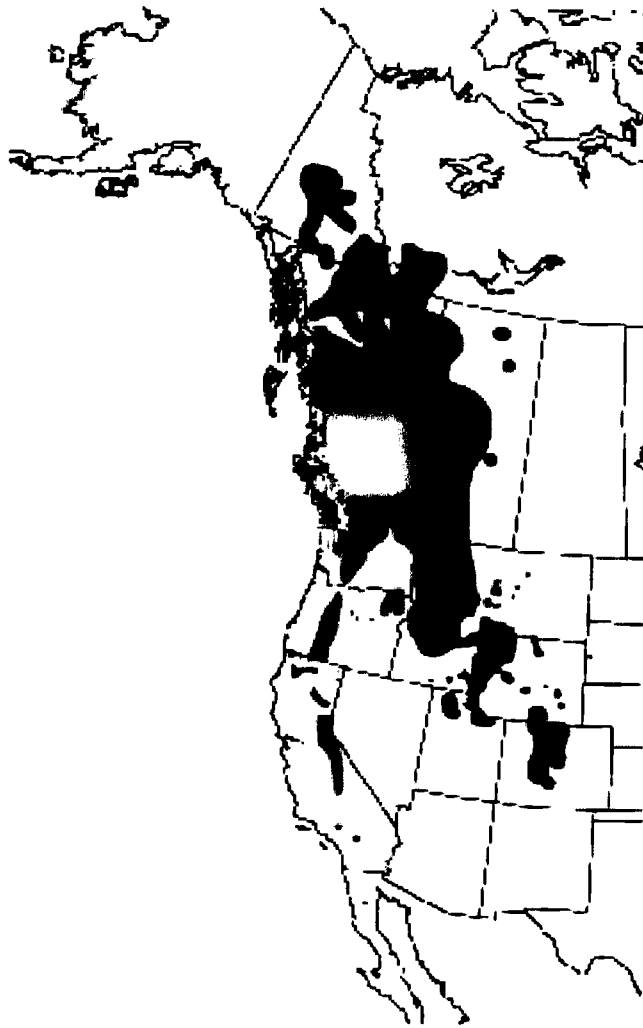
Figure 1-5. Distribution of lodgepole pine in North America (Wheeler and Critchfield, 1985).

and log size, as well as the salvage of beetle-killed wood (Kennedy, 1985; Koch, 1996; Council of Forest Industries, 2000). Thus, lodgepole pine is a key species to the forest industry of BC and more importantly Canada.

1.4.2 Usage of Lodgepole Pine

Lodgepole pine is currently used for lumber, poles, railroad ties, posts, furniture, cabinetry and construction timbers. In Canada, roughly 90% of the lodgepole pine is consumed in lumber production. During sawmilling, approximately 50% of the tree is converted to merchantable lumber; with the residual 50% being in the form of chips (40%) and sawdust (10%). Therefore, lodgepole pine represents a significant primary resource for pulp mills in BC and Alberta. Additionally, some logs are converted directly into veneer for incorporation in Canadian softwood plywood, while smaller amounts are used for oriented strandboard production. Almost all distribution poles roughly 45 feet in height in the Interior of British Columbia and in Alberta are also produced from lodgepole pine. The effective treatment with wood preservatives for lodgepole pine sapwood under standard pressure processes and along with its straight form, makes it a preferred pole species as well as for the construction of log homes. Although lodgepole pine heartwood is more difficult to treat than sapwood, it is a preferred species for railway tie production due to its availability and hardness (Kennedy, 1985; Kim, 1988; Koch, 1996).

In addition, lodgepole pines are used in cement wood composites, laminated structural wood, particleboards, hardboard, medium density fiberboard, thermoformable wood-plastic composites, and fabricated I-joists. It is also a highly acceptable species for pulping such that lodgepole pine kraft pulp gives good yield, burst and tensile strengths, is light in colour, and has moderately long fibers. Furthermore, mechanical pulps also give satisfactory strength (Kim, 1988; Koch, 1996). However, as lodgepole pine is an important species to the industry and is

used to produce a vast amount of products, there are some significant problems associated with this species.

1.4.3 Problems Associated with Lodgepole Pine

Lodgepole pine forests usually contain thousands of trees per acre in pure or semi-pure stands where, most of the trees are either over-mature (>80 years old) or insect-killed. Moreover, a large proportion of the resource is comprised of dense, stagnated stands whereby low growth rates and high mortality are apparent. On average, the lodgepole pine stems are small in diameter, with approximately one-third of the resource consisting of trees with diameter at breast height less than 175mm (Shrimpton and Thomson, 1982; Kim, 1988).

As a result of the accumulation of dead standing trees, wood converters will eventually be faced with utilizing this resource in their wood supply. In 1999, the volume of BC timber losses due to wildfires, insects and diseases totalled 13.5 million cubic metres, with 94.8% of the losses being caused by forest pest infestation. The Prince George forest region suffered the largest loss accounting for 8.1 million cubic metres, of which lodgepole pine was a significant fraction (Council of Forest Industries, 2000). Recent figures suggest that the volume of dead lodgepole pine attributed to mountain pine beetle infestation in BC alone, encompasses 108 million cubic metres, which has been estimated to be worth roughly $9 billion to the forest industry (Penner, 2002). Thus, the volume of dead lodgepole pine trees resulting from the mountain pine beetle has currently become one of the largest epidemic-sized problems in Canadian history. In an attempt to mitigate the mountain pine beetle crisis, the Chief Forester of BC has decided to increase the annual allowable cut (AAC) in order to begin harvesting some of the substantial volumes of timber projected to be killed, before their merchantable value is lost, and to provide for the removal of a large number of dead trees (Pedersen, 2001). As a result, the pulp and paper industry in particular is being forced to utilize more dead-standing wood through

provisions of the forest practice codes and such material can at times comprise a significant volume of chip supply (Natural Resources Canada, 2002).

The problems associated with dead standing lodgepole pines particularly attacked by the mountain pine beetle is that they begin to deteriorate before they are dead through the incursion of blue stain. After the tree dies, there are different levels of deterioration that follow, which include additional incursion of blue stain, moisture content reduction, loss of foliage and bark, onset of stemwood checks, and the development of decay (Koch, 1996). Blue stain fungus, mainly *Ceratocystis* species, is inoculated into the sapwood via bark beetle attack; roughly 50% of the sapwood is stained within one month, and the entire sapwood within 3 months. Blue stain is a major problem because it significantly impacts lumber value (Fahey *et al.*, 1986; Koch, 1996). In addition, Reid (1961) reported that there is a loss in moisture content in the sapwood that occurs in the first year following mortality; while the moisture content of heartwood is lost more slowly. The loss in moisture content, to a point below the fibre saturation point, irreversibly impacts wood quality. Moisture losses can result in stem splitting which affects added-value product recovery in sawmills, chip quality and pulping. Moreover, decline in stemwood specific gravity in standing dead trees also occur (Reid, 1961; Fahey *et al.*, 1986).

1.5 Mountain Pine Beetle

1.5.1 Distribution and Occurrence

The mountain pine beetle, otherwise known as *Dendroctonus ponderosae* Hopkins, is native to North America and is one of the most damaging bark beetles attacking lodgepole pine. It is found in an area spanning from northern Mexico (lattitue 31° N) to northwestern BC (latitude 56° N) and from the Pacific Coast east to the Black Hills of South Dakota. In Canada, the range of the mountain pine beetle extends as far east as the Cypress Hills in Alberta (Figure 1-6). Its habitat ranges from near sea level in BC to roughly 750m near the northern limit, and

14
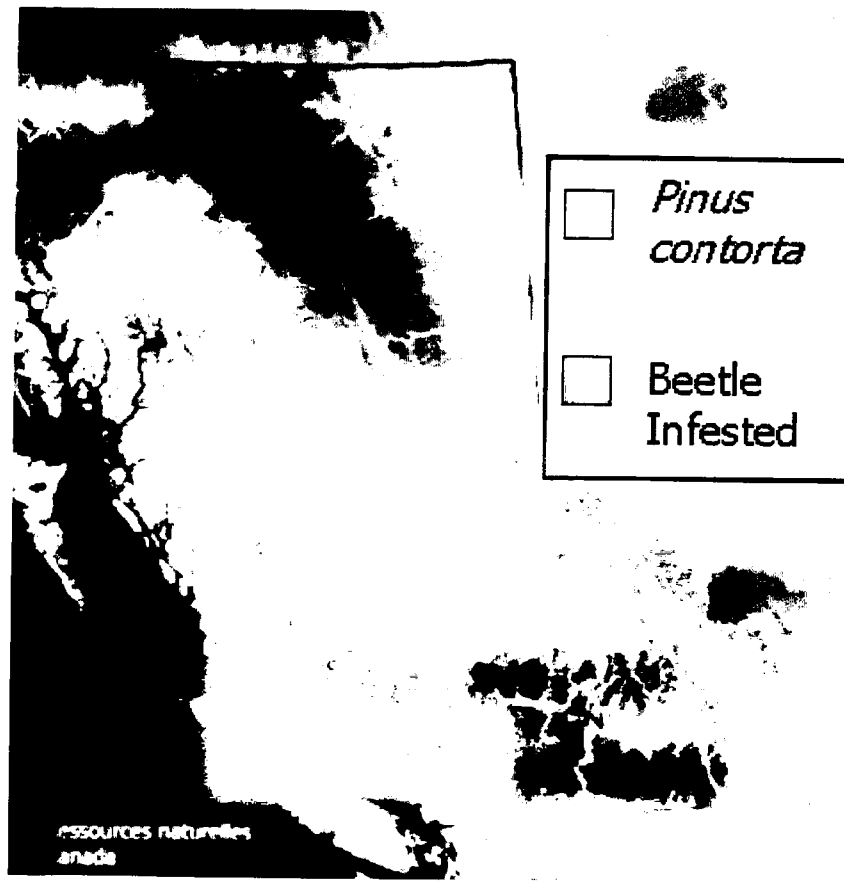
Figure 1-6. Distribution of mountain pine beetle in British Columbia (Natural Resources Canada, 2002).

up to 3650m in the most southerly regions (Safranyik *et al.*, 1999). The primary hosts are lodgepole pine (*Pinus contorta* var *latifolia*), ponderosa pine (*P. ponderosa* Laws.), western white pine (*P. monticola* D.Don.), and sugar pine (*P. lambertiana* Douglas) (Amman *et al.*, 2002). However, the beetle has been known to infest all native pines and several exotic species in its range. In western Canada, lodgepole pine is the beetle's preferential host (Safranyik, 1978; Safranyik *et al.*, 1999).

1.5.2 Life Cycle

The mountain pine beetle overwinters as larvae in trees attacked during July and August, and emerge as adults the following July and August to attack green trees. Throughout all stages of life, mountain pine beetles remain under the bark, except only when emerging as adults to fly and attack other trees in the midsummer (Klein *et al.*, 1978; Mitton and Sturgeon, 1982). It typically has a single generation per year in lodgepole pine (Figure 1-7). In some years, emergence and infestation extend into September. The length of the life cycle is dependent upon temperature conditions. For summers warmer than average, the parent beetles can re-emerge and build a second brood. For cooler summers, which generally happen at high elevations, it may require the brood to take 2 years to mature. Initially, female adults select and infest green trees and further construct vertical egg galleries. Females may perform a second attack and build a second egg gallery during extended warm seasons. Following the initial female attack, she is then soon joined by a male adult. The females build their egg gallery in the bark, and slight disruption is introduced in the adjoining sapwood. The female then lays her pearl-white coloured eggs, which on average are one millimeter in length (Cole and Amman, 1980; Koch, 1996).

The eggs are laid in individual niches scored into the phloem in alternate groups irregularly arranged between the two sides of the gallery, which can reach lengths of 25m

MOUNTAIN PINE BEETLE LIFE CYCLE

MAY — JUNE — JULY — AUGUST

Larva — Pupa — Adult — Egg — Larva

Overwinter as larva in galleries
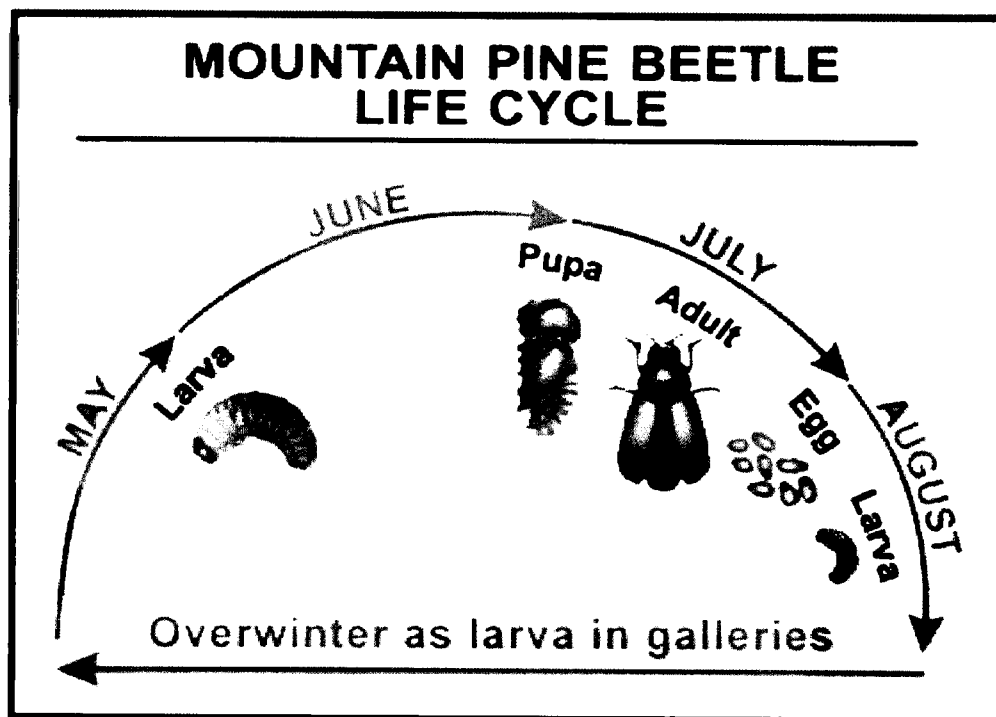
Figure 1-7. One year life cycle of the mountain pine beetle (Amman and Walter, 1983).

(Amman and Walter, 1983; Safranyik, 1985). In the presence of fresh phloem (inner bark), the beetles will continue to lay eggs and construct galleries. The size of the eggs is apparently related to the size of the female. Factors affecting the increase in oviposition include female size, phloem thickness of the host tree, as well as temperature. Furthermore, a greater number of eggs are laid in phloem that is thick rather than thin for example, the average number of eggs laid per centimeter of gallery ranged from 1.4 per cm in phloem 1.5mm thick to 3.8 per cm in phloem 4.6mm thick (Reid, 1962; Amman and Walter, 1983). Both the rate of gallery construction and the number of eggs laid are strongly related to temperature such that some oviposition occurs as low as 1.7°C, and becomes most intense when temperatures exceed 15°C (Amman and Walter, 1983; Koch, 1996).

Roughly within a week, the beetle eggs hatch. The mountain pine beetle has four larval instars. Typically, the larvae are pearl-white in colour, with amber head capsules. They feed on the phloem, while constructing feeding channels that protrude approximately at right angles to the egg galleries. The larvae overwinter and are very cold hardy (Koch, 1996). However, extensive mortality can arise as temperatures occurring during an average winter may interact with the stage of overwintering brood (Safranyik, 1985). Large larvae are less susceptible to winterkill compared to small larvae and eggs (Amman and Walter, 1983) even though the first three larval stages contain proportionately the same amount of glycerol, an alcohol that protects against freezing (Safranyik, 1978; Koch, 1996).

After digging out of the oval cells, the larvae transform into pupae when completely developed. Typically, by July the pupae transform into adults and generally leave the tree in late July to early August to attack new trees. However, the period of beetle migration to new trees varies with elevation, latitude, longitude, and local weather, with some beetles requiring up to two years to complete a generation (Safranyik, 1978; Amman and Walter, 1983; Koch, 1996).

1.5.3 Fungi Associated With Mountain Pine Beetles

Mountain pine beetles, in their mass attack on lodgepole pines, inoculate blue-staining fungi, primarily the *Ceratocystis* species and several species of *Europhium* as well as other mycelial fungi and yeasts, into the infested tree, which ultimately results in eventual tree death. The fungus is introduced into the sapwood of trees under bark beetle attack from pouchlike structures (mycangium) in the head of the beetle (Harvey, 1979; Koch, 1996). As the beetles tunnel into the inner bark region, they apparently inoculate a "fungus complex" into the host. Propagules of the fungus complex develop and grow in the beetle feces within the beetle galleries. Eventually, hyphae grow into the radial parenchyma tissue of the sapwood to which they are initially confined (Whitney, 1971; Ballard *et al.*, 1983).

Hyphae penetrate the primary cell walls or ray parenchyma cells and propagate rapidly and abundantly within, following the path of least resistance. Hyphae also grow lavishly in the portion of the middle lamella of the rays. Eventually, the hyphae penetrate the tracheids by entering the primary cell walls via half-bordered pit pairs. Within the tracheids, fungal hyphae grow in a longitudinal fashion, branching out infrequently. They then pass from tracheid to tracheid via pit pairs, and as a consequence, little resistance to fungal penetration is provided by the cell walls and hyphae readily reach untapped food sources (Ballard *et al.*, 1983).

Growth of blue-stain fungi in sapwood appears mainly to affect the moisture content of host trees. Ballard *et al.* (1982) reported that with the proliferation of blue-stain in the sapwood, the transpiration of trees infested by bark beetle become confined to the inner portions of the sapwood and eventually transpiration ceases altogether. Studies also showed that a high percentage of the bordered pits were aspirated, and Ballard *et al.* (1982) suggest that hyphal penetration of tracheids may be accountable for introducing the frequency of aspirations observed. Another theory to explain how blue-stain fungi affect moisture content was the aspiration of bordered pits in the sapwood was due to encrustation products, which are thought to

be lodged against pit membranes (margo) of bordered pits, and thus slow and eventually stopped the flow of water through the pits (Whitney, 1971; Ballard *et al.*, 1982; Ballard *et al.*, 1983). Additionally, it has been speculated that the destruction of ray parenchyma tissue by blue-stain fungi may be a possible mechanism for transpiration dysfunction in the host tree (Ballard *et al.*, 1983).

1.5.4 Tree Selection

The process by which the mountain pine beetle selects its host differs between two population levels: endemic (low levels) and epidemic. During endemic outbreaks, typically where the population densities of mountain pine beetle are less than one attacked tree per 10 hectares, the beetle usually chooses to infest the lower bole (1 to 2 metres) of trees previously infested by other bark beetles in the spring. Conversely, during an epidemic outbreak, beetles generally attack green or moist phloem at the top of large diameter trees (Moeck and Simmons, 1991; Koch, 1996). In general, mountain pine beetles fly at a level relative to the midbole of lodgepole pine in both thinned and unthinned stands. From beetle catch studies, results showed that the majority of the pine beetles were caught at midbole height. Typically, the average height of initial attack by the mountain pine beetle ranges between 2.0 to 5.0 metres. Succeeding attacks then spread up, down and around the bole (Amman and Walter, 1983). Klein *et al.* (1978) noted that as an outbreak abates there is a decrease in infested height, which is related to lower beetle density since beetles generally like to attack the base first and then proceed to higher environments. The length of the tree that is attacked is related to both the beetle population size and to the tree diameter (Klein *et al.*, 1978).

Moreover, during epidemics, the mountain pine beetles are primarily attracted to large diameter lodgepole pine, regardless of health. In the early years of infestation, beetles can be fairly selective of their hosts; however, selection is hidden when overcome by pheromones,

which bring about mass attack of trees that meet size requirements. When trees are exposed to high beetle populations, it is incapable of withstanding attack. The beetle outbreak will proceed within a stand until nearly all of the large diameter trees are killed. When beetles infest small diameter trees with thin phloem, the population tends to languish, as the host trees dry excessively and rapidly. Therefore, the mountain pine beetle is confined to lodgepole pine stands where optimal temperatures for development are present (Koch, 1996).

The primary element determining brood survival and proliferation in lodgepole pine is related to the amount of phloem present, which is the food source for the developing larvae. Phloem thickness is related to tree diameter within most stands, and affects the behaviour of the female beetle in choosing greater quantities of large diameter trees (Shrimpton and Thomson, 1982). The beetles use both visual and chemical cues to select the trees they infest, including olfactory stimulus from monoterpenes located in the bark. Thus, ensuring brood survival within the thicker phloem found in larger diameter trees (Cole and Amman, 1980; Koch, 1996).

1.5.5 Mode of Attack

The attacking behaviour of the mountain pine beetle is very intricate as it is regulated by numerous pheromones (insect produced) and kairomones (host produced) (Klein *et al.*, 1978). The female beetle releases an aggregating pheromone, trans-verbenol, after penetrating into the bark of the tree, and this pheromone is combined with small amounts of exo-brevicomin, which is typically produced by the male. In combination of pheromones from the beetle and terpenes produced from the host, other beetles are directed to the tree, and the pheromones and terpenes become a signal for mass attack. Terpenes in lodgepole pine are made up mostly of beta-phellandrene, with small amounts of alpha-pinene, beta-pinene, 3-carene, myrcene, and camphene (Shrimpton, 1973; Koch, 1996).

Alpha-pinene and myrcene are the most influential terpenes. Alpha-pinene rapidly induces and augments the biosynthesis of trans-verbenol by the mountain pine beetles. Also, the large quantities of beta-phellandrene in lodgepole pine phloem are an effective kairomone for mountain pine beetles (Koch, 1996; Cole and Amman, 1980).

As beetles establishing their brood galleries occupy a tree, greater concentrations of exo-brevicomin and frontalin from males serve as antiaggregation pheromones. This results in a decreasing concentration of both female-produced trans-verbenol and host volatiles, which reduce the attractiveness of the tree. Moreover, microorganism-controlled conversion of cis and trans-verbenol to verbenone produces an intense antiaggregation signal that ensures overcrowding of the brood does not occur. The other females flying in will settle far away from the source of the antiaggregative pheromones. Thus, the mode of attack is spread along the bole of the tree, and this eventually causes an increase spread of infestation to nearby trees, where the process is repeated (Klein *et al.*, 1978; Cole and Amman, 1980; Koch, 1996).

1.5.6 Tree Vigour

Prior to emergence, as the maturing beetle feeds on the phloem, it obtains fungal spores in its maxillary mycangium. Through the introduction of spores into the living tissues, blue staining fungi inoculate the phloem and particularly the sapwood, where they aid in disrupting the flow of resin and the vascular system (Ballard *et al.*, 1983). Initially the moisture content is reduced substantially in the sapwood, and blue-stain fungi proliferation is evident in the change in colour of the sapwood. In addition to destroying host tissues through the impact of drying stresses, blue stain fungi may also control moisture in the tree during beetle development (Reid *et al.*, 1967). However, blue-stain fungi do not appear to be essential to mountain pine beetle nutrition (Whitney, 1971; Shrimpton and Thomson, 1982; Koch, 1996).

As a tree is mass attacked (infested by numerous beetles within a day or two), pitch flow from holes where beetles have penetrated the bark, is halted thereby causing beetle progeny success. As blue staining fungi is not the only factor affecting the ability of mountain pine beetle to overcome the host tree, exorbitant mechanical wounding causes resin flow to stop within 24 to 48 hours. As the process of resistance requires an immense energetic expenditure, this response may be too great for older trees. Generally, an indication of beetle infestation can be recognized by pitch tubes where beetles have entered the tree and boring dust in bark crevices at the base of the tree. Pitch tube size is related to the quantity and rate of beetle infestations, as well as to the moisture availability to the tree (Shrimpton, 1973).

Indication of beetle-caused mortality is usually observed in the discolored tree foliage starting from the lower crown and traveling to the upper crown. The needles from infested trees also change colour several months to almost a year after beetle attack. The series of colour change to the crown is initially green to yellowish-green, followed by a transformation to red, then changing to a rusty-brown and finally turning grey (Koch, 1996; Amman *et al.*, 2002).

Lodgepole pines also vary in their resistance to mountain pine beetle attack. Some trees show complete resistance to attack, while others only show initial resistance, and some even exhibit little or no resistance. Complete resistance of the host causes failure of beetles and blue stain fungi to complete their life cycle. Typically, tree resistance to attack has been associated with osmotic pressure of cell sap in the phloem and epithelial cells of resin ducts, protein and carbohydrate content of bark tissue, drought conditions, and more recently to resin pressure (resinosis). There are two kinds of resin and these include primary and secondary resin. Primary resin is released when resin ducts are mechanically wounded by initial insect infestation. Secondary resin stems from live parenchyma cells located in the bark and sapwood. A tree's ability to resist insect attack is directly dependent on their ability to produce secondary resin. Most trees in response to wounding undergo primary resinosis, however, not all trees undergo

secondary resinosis. It has been determined that secondary resinosis in the sapwood can progress towards the heartwood. In response to beetle and fungal invasion, trees vary in their ability to produce secondary resinosis, and studies have shown that secondary resinosis is the most important factor contributing to the resistance of lodgepole pine to bark beetles and blue stain fungi. In addition, trees vary in their quantitative resistance to infestation of organisms as a result of genetic, environmental and seasonal factors (Reid, 1962; Reid *et al.*, 1967).

1.5.7 Stand Susceptibility

In order to prevent and control beetle outbreaks, stands have to be identified with the characteristics related to beetle attack. A number of methods have been developed for rating the susceptibility of lodgepole pine stands to beetle infestation, and these are based on mountain pine beetle epidemic characteristics (Amman and Walter, 1983).

The characteristics associated with lodgepole pine susceptibility to beetle attack include: average age more than 80 years old; average diameter at breast height more than 25.4cm in Canada; suitable climate for development based on elevation and latitude, and habitat type (Amman and Walter, 1983). In general, site susceptibility ratings are very predictive when using age, diameter, and elevation to rate the susceptibility of stands over a large area. Other methods of predicting stand susceptibility include the important factors such as weather and climate. Basically, different combinations of variables explain different proportions of variance in stand mortality of lodgepole pine (Berryman, 1976; Koch, 1996; Amman *et al.*, 2002).

The jeopardy of beetle infestation not only depends upon invasion of the stand of interest but also to the surrounding area. Some stands can be at great risks to beetle infestation but may not be in direct peril as a result of no current infestation being present. Thus, the chance of infestation is dependent upon the condition of the stand, that is, the environment (i.e. the yearly temperature); presence of trees of large diameter at low elevation (susceptibility index); number

of beetles in or around the stand (beetle pressure index); and risk of an outbreak actually occurring (risk index) (Koch, 1996; Safranyik *et al.*, 1999).

1.5.8 Control

Years of research into the ecology of the mountain pine beetle have brought about the development of two different strategies for reducing losses: preventive management and direct control. Preventive management is the most satisfactory long-term solution that is related to the influence of the tree and stand conditions to reduce the risk of beetle infestation (Safranyik *et al.*, 1999). Conversely, direct control involves killing or repelling beetles. As direct control tends to treat the cause (the mass of beetles) of the problem, the effects are usually temporary. However, when properly implemented, direct control can reduce the spread and intensity of beetle attack (Koch, 1996; Maclauchlan and Brooks, 2000). Furthermore, direct control may provide an impeding action until stands at risk can be silviculturally treated. In general, strategies that help prevent rather than control beetle infestations are favoured. Moreover, vast outbreaks typically cannot be hindered before trees lose economic value (Amman and Walter, 1983; Koch, 1996).

1.6 Research Objectives

Currently, Canada, and British Columbia more specifically, is faced with a mountain pine beetle epidemic, which has greatly affected the forest industry. At present, the mountain pine beetle attack has caused substantial losses in terms of the total volume of lodgepole pine timber in BC, and it has been estimated that at current rates of spread, the volume of newly attacked ('green-attack') timber in the timber supply area could spread to cover 150 million cubic metres by 2003, and over 200 million cubic metres of 'green-attack' by 2010. At this rate of spread, in 10 years' time, 75% or more of the mature pine in the timber supply area will be infested at some level (Pedersen, 2001; Penner, 2002). In an attempt to fight the mountain pine beetle crisis,

abundant volumes of dead lodgepole pine trees, as well as live lodgepole pine trees are being harvested. The forest industry, in particular the pulp and paper industry, are presently forced to utilize this material in their feedstock.

However, there are many problems associated with the use of dead standing trees, such as substantial decreases in moisture content, incursion of blue-stain, as well as reduced product quality. It is critical to understand the mechanisms of the problems associated with dead wood in order to fully utilize the resource and develop strategies for amelioration. Therefore, the primary objective of this thesis is to undertake a fundamental evaluation on how and why dead wood affects pulp fiber quality.

This thesis will address the effect of mountain pine beetle attack on: 1) moisture content, 2) wood density, 3) wood chemistry, 4) wood permeability and anatomy, and 5) pulp and paper quality.

CHAPTER 2. MATERIALS AND METHODS

2.1 Tree Site Characteristics

Trees were harvested from a previously extensively characterized location (in terms of site characteristics using the biogeoclimatic ecosystem classification method) at the University of British Columbia Alex Fraser Research Forest located in Williams Lake, BC. Ecological assessment of the plot was completed to give an estimate of site quality. A dead (infested with mountain pine beetle) and sound standing lodgepole pine tree were harvested from the same site in the dry warm subzone of the sub-boreal spruce zone. Both the infested and sound standing tree were 12m in length and 440mm in diameter at breast height. The sound lodgepole pine tree was approximately 87 years old and the infested tree was 91 years in age. It was estimated that the infested tree had been infested by mountain pine beetle for approximately 8 months prior to harvest.

2.2 Sample Procurement

Both the sound and infested lodgepole pine tree were bucked and debarked. A biscuit was cut from different positions of the tree (Figure 2-1), starting at breast height (1.5m) and following in four-meter increments (5.5m and 9.5m). Each tree was analyzed for sapwood and heartwood using a sapwood-heartwood indicator for pines determined from the American Wood Preservers Association standard M2 section 5.311. The indicator was a combination of equal volumes of o-anisidine hydrochloride (VWR) and 10% sodium nitrite solution (Sigma). The biscuits were cut in half; one quarter of the biscuit was used for moisture content determination, while the other quarter was used to determine wood density and permeability. The other half of each biscuit was used to investigate wood chemistry; including total extractives (gravimetrically), total lignin (acid soluble and acid insoluble), and carbohydrate content, as well as for microscopic analysis.
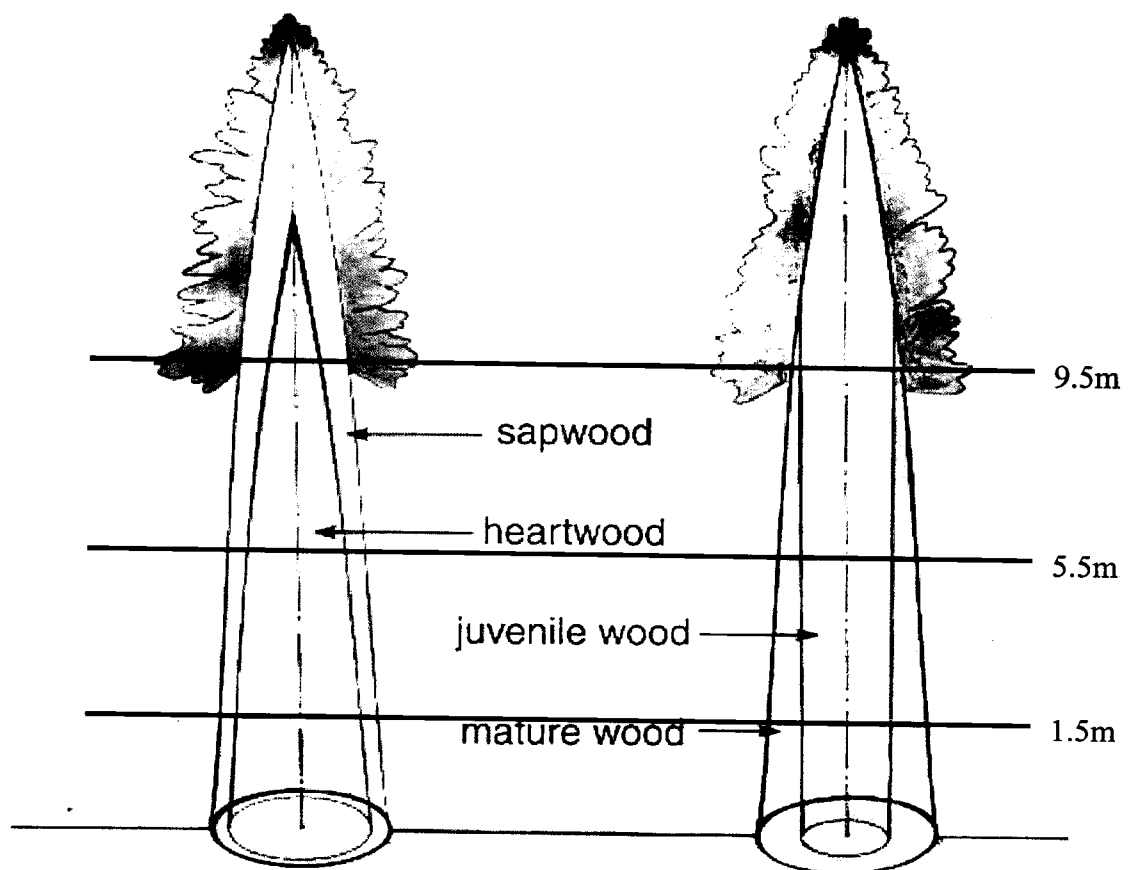
Figure 2-1. Biscuits were cut from the three heights and used to explain tree variations.

The remainder of the tree was cut into bolts at different positions of the tree, starting at the butt section (2m) and followed by two-meter increments (4m, 6m, 8m, 10m and 12m). The bolts were then slabbed on a bandsaw to produce heartwood and sapwood sections. The slabs were then chipped on a CM&E 36 inch 10-knife disc chipper for subsequent kraft pulping experiments.

All experimental analyses were performed on infested and sound sapwood and heartwood at each position of the tree.

2.3 Moisture Content Determination

The moisture content of quartered biscuits, wood chips, ground extractive-free wood, and pulp was determined using the standard TAPPI Test Method T 264 cm-97 for basic density and moisture content of pulp wood.

Approximately 100g of air-dry wood chips, 4g of extractive-free wood, and 50g of air-dry pulp were weighed and dried for 24 hrs at 105°C. The wood chip and pulp samples were weighed immediately after they were removed from the oven. The extractive free-wood samples were placed in a desiccator containing phosphorus pentoxide (Fisher Scientific) to cool and the oven-dry weight was recorded to 0.0001g. The moisture content was determined by the following equation:

(1) Moisture Content (%) $= \dfrac{W_A - W_o}{W_o} \times 100\%$

where:

W_A = Air-dry weight of sample (g)

W_o = Oven-dry weight of sample (g)

2.4 Wood Density

The wood density was measured according to the ASTM Standard Test Methods for Specific Gravity of Wood and Wood-Base Materials (D 2395-93 re-approved 1997) Method B – Volume by Water Immersion, Mode IV.

Basically, wood density evaluation was performed on triplicate samples for the sound and infested tree, between heartwood and sapwood, as well as at different positions along the height of the tree for a total of 36 samples. The wood specimens were cut into cylinders (18mm in diameter and 50mm in length), and conditioned to 12% moisture content. A 50mL graduated cylinder was filled to approximately 30mL and the oven-dry wood specimens were then submerged into the cylinder using a long metal pick. The difference in the initial and final water volume is equal to the volume of the wood sample. Wood density was calculated using the following equation:

(2) Wood density $(\rho_{12\% \, MC}) = \dfrac{W_{12\% \, MC}}{V_{12\% \, MC}}$

where:

$W_{12\% \, MC}$ = mass of specimen at 12% moisture content (g)

$V_{12\% \, MC}$ = volume of specimen at 12% moisture content (cm^3)

2.5 Wood Extractives Content

Wood extractives content was determined according to the standard TAPPI Method T 280 pm-99. Extraction of all wood samples were performed in 5 replicates for a total of 60 samples.

Approximately 4.0g of air-dry wood was ground using a Wiley mill to pass a 0.4mm (40 mesh) screen. The air-dry ground wood samples were weighed and placed into oven-dried glass thimbles. The ground wood specimen and thimble was then oven-dried for 24 hours at 105 °C and the moisture content of the wood determined. The thimbles containing the wood specimens were covered with folded-cone shaped filter paper and a small cotton ball, and weighed prior to extraction. The samples were then Soxhlet extracted with 150mL of reagent grade acetone at 55

ºC for approximately 12 hours. The thimbles were removed and oven-dried for 24 hours at 50ºC, and weighed to determine the difference between extracted and non-extracted wood. The acetone was rotary-evaporated to dryness in a water bath at 40ºC. Extractives were then re-solublized with 5mL of HPLC grade acetone (Fisher Scientific), stored in Kimax culture tubes and concentrated to 1mL by heating at 40ºC under a stream of nitrogen gas. The extracts were filtered and analyzed for composition by gas chromatography. Tetracosane (Sigma), abietic acid (Sigma), betulin (Aldrich), cholesteryl palmitate (Sigma) and tripalmitin (Sigma) were used as internal standards, representative of resin acids, fatty acids, sterols, steryl esters, and triglycerides, respectively. The retention times used to determine the fatty and resin acid content ranged from approximately 3 to 24 minutes, sterols from 24 to 34 minutes, steryl esters from 34 to 42 minutes, and triglycerides from 42 to 48 minutes.

Gas chromatography (GC) analysis was performed on a Hewlett-Packard 5890 Series II Gas Chromatograph equipped with a Hewlett Packard 6890 Series injector and a 10m DB-XLB column (J&W Scientific) using a modification of the method of Fernandez et al. (2001). The chromatographic method involved a 1.0µl injection volume and helium as a carrier gas. The initial injector temperature was 320ºC and the detector temperature was 330ºC. The initial oven temperature was programmed with a three-minute isotherm at 50ºC, followed by a 10ºC per minute ramp to 240ºC where it was held for 3 minutes. The temperature was further ramped to 310ºC at 10ºC per minute and held for 3 minutes. The temperature was then increased to 350ºC at 10ºC per minute and held for 25 minutes.

2.6 Wood Klason Lignin Analysis

Klason analysis was performed in 5 replicates for a total of 60 samples of extractive-free wood. Lignin content was determined using a modified Klason lignin method (TAPPI method T222 om-98).

Approximately 0.2g of extractive-free, oven-dried wood samples were weighed into a reaction flasks and 3mL of cold (4°C), 72% H_2SO_4 (Fisher Scientific) were added and reacted at 20°C. The wood samples and H_2SO_4 were mixed in the reaction flasks every 10 minutes for 2 hours. Acid hydrolysis was terminated with the addition of cold deionized water; whereby the reaction mixture was diluted to a final acid concentration of 4% H_2SO_4 with 112mL of de-ionized water and then transferred into serum bottles. These serum bottles, sealed with septa caps, were then autoclaved (Castle Thermatic 60 Autoclave) at 121°C for 1 hour, cooled and vacuum-filtered through pre-weighed, oven-dried medium coarseness sintered-glass crucibles. The filtrate was added back into the septa bottles and re-filtered to ensure recovery of all solids, and the filtrate retained. The solids were further washed with 100mL of deionized water (4°C) and oven-dried at 105°C for 12 hours. Acid-insoluble lignin was gravimetrically determined by weighing the oven-dried crucibles containing the acid-insoluble lignin.

The filtered klason lignin hydrolysate was then analysed using the TAPPI Useful Method UM-250 to determine the percentage of acid soluble lignin. 100μL of the filtered klason lignin hydrolysates were added into test tubes and further diluted with 900μL of 4% H_2SO_4. The solutions were mixed thoroughly, and measured on a spectrophotometer (Milton Roy Spectronic 1001 Plus) such that the absorbance readings were between 0.2 and 0.7 absorbance units (AU) at 205 nm. 4% H_2SO_4 was used as a reference solution in order to calibrate the spectrophotometer. After the calibration procedure, quartz cuvettes with diluted filtrate solutions were placed into the spectrophotometer and absorbance readings were taken. Using the following expression of Beer's Law, the acid-soluble lignin content was calculated:

(3) Acid-soluble lignin (%) $= \dfrac{B \times V \times 100}{1000 \times W_o}$

Where:

$B = \dfrac{\text{Absorbance (AU)} \times \text{volume of diluted filtrate } (\mu L)}{110 \times \text{volume of original filtrate } (\mu L)}$

V = Total volume of filtrate (mL)

W_o = Oven-dry weight of wood (g)

2.7 Wood Carbohydrate Analysis

Wood carbohydrates content, performed in 5 replicates for a total of 60 samples, was quantified using a Dionex DX-600 High Performance Liquid Chromatography (HPLC) system (Dionex, Sunnyvale, CA USA) controlled by Peaknet 6.10 software. The column (PA1) was equilibrated with 250mM NaOH. The klason lignin hydrolysate was filtered through 0.45mm HV filters (Millipore, MA, USA), and fucose (5mg/mL) was added as an internal standard. After injecting $20\mu l$ of filtered hydrolysate using an AS 50 autosampler (Dionex), the sugars were eluted using Nanopure water at a flow rate of 1.0mL/min. The monosaccharides were monitored using a Dionex E50 electrochemical detector (gold electrode), with parameters set for pulsed amperometric detection of sugars, as recommended by the manufacturer. A post column addition of 200mM NaOH before the detector at a flow stream rate of 0.5mL/min was used to achieve the most sensitive detection. Arabinose, galactose, glucose, xylose and mannose standard curves were used to convert the electrical signals into sugar concentrations. The total carbohydrate content was determined by calculating the total sum of all the monosaccharides present.

2.8 Longitudinal Specific Permeability

Longitudinal specific permeability was determined by using the falling water volume-displacement method (Siau, 1995).

Permeability analysis was performed on triplicate wood samples for a total of 36 samples. Wood specimens (18mm × 50mm cylinders) were taken at random from the heartwood and sapwood areas from the sound and infested tree; as well as along different positions of the tree

starting at breast height followed by four-metre increments. In order to eliminate the influence of moisture content on permeability, the wood samples were conditioned in a conditioning chamber (4.5 cubic foot Climate-Lab) at 23°C for two weeks so that moisture content was equilibrated to approximately 12%. Using a razor blade, surface cuts were made on each end of the wood sample prior to inserting the specimen into the apparatus.

The diameters of displacement tubes used were 10mm and 20mm. Using a vacuum, water was drawn above point 1 in the displacement tube (Figure 2-2). The vacuum was then turned off so that air would flow through the specimen causing the water level in the displacement tube to drop. The time for the water to drop from point 1 to point 2 was measured. Permeability then was determined using the following equation:

$$(4)\ k_g = \frac{V_d\ C\ L\ (P_{atm}-0.074\ \bar{z})}{tA(0.074\ \bar{z})(P_{atm}-0.037\ \bar{z})} \times \frac{0.760m\ Hg}{1.013 \times 10^6\ Pa} \times 1.81 \times 10^{-5}\ Pa\ s$$

$$C = 1 + \frac{V_r\ (0.074\ \Delta z)}{V_d\ (P_{atm}-0.074\ \bar{z})}$$

Where:

k_g = longitudinal specific permeability ($\mu m^3/\mu m$)

$V_d = \pi r^2 \Delta z$ (r = radius of measuring tube (m)) (m^3)

C = correction factor for gas expansion as a result of change in static head and viscosity of water

L = length of wood specimen (m)

P_{atm} = atmospheric pressure (mHg)

$\bar{z}$ = average height of water over surface of reservoir during period of measurement (m)

t = time (s)
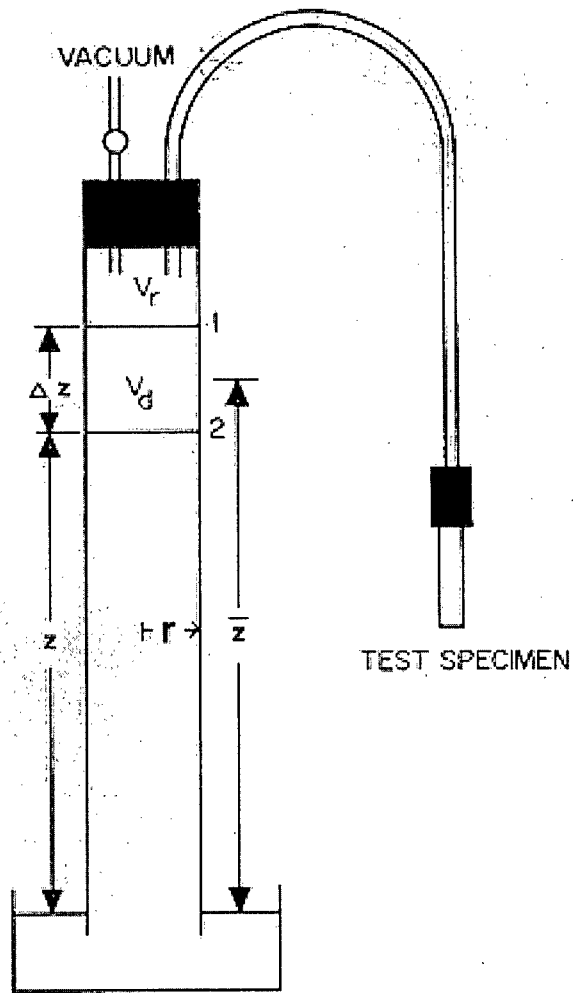
Figure 2-2. Falling water volume-displacement method apparatus (Siau, 1995).

A = cross-sectional area of wood specimen (m²)

Δz = change in height of water during time t (m)

V_r = total volume of apparatus above point 1 (including volume of hoses) (m³)

2.9 Scanning Electron Microscopy Analysis

Wood samples (selected at random from heartwood and sapwood in both the infested and sound tree at three different tree heights and performed in duplicates for a total of 24 samples) were cut into rectangular shaped blocks measuring 10mm × 10mm × 20mm. Tangential, radial and transverse splits were made with uncoated single-edged razor blades for each sample. The wood specimens were then placed into scintillation vials and submerged in acetone for 24 hours. After 24 hours, the wood samples were removed from the vials and dried on bibulous paper and then mounted on Scanning Electron Microscope (SEM) aluminum stubs and sputter coated with 60:40 gold palladium alloy using a Hummer VI Sputtering System. The sample mounts were then observed in a JEOL JSM-840A scanning electron microscope (Japan Electron Optics Laboratory) using 10 kV accelerating voltage.

2.10 Wood Chip Classification

Each tree was cut into bolts at different positions along the tree (2m, 4m, 6m, 8m, 10m and 12m), slabbed on a bandsaw to produce heartwood and sapwood sections, and then chipped in a CAE 36 inch 10 knife disc chipper. Wood chip classification was performed in duplicates for a total of 48 samples. The wood chips were thoroughly mixed for each sample and further screened on a EMP/Wennberg Chip Classifier to obtain accept chips in the thickness range from 2 to 6mm, as well as to remove oversized and fine material. The quality parameter used for evaluating chip screening is called screen removal efficiency. The chip size distribution was measured before and after screening by weight. The chip classifier contained plates perforated

with round holes. The top screen has 45mm wide round holes and the chips retained on this screen are oversized chips. The next screen has 10mm wide round holes and retains overthick chips. The third screen has 6mm wide slots and accept chips are retained on this screen. The fourth screen with 2mm wide slots also retains the accept chips and the pan found on the very bottom collects the fines. The removal efficiency of a size fraction is then calculated by the following equation:

(5) Screen Removal Efficiency % $= \dfrac{B - A}{B} \times 100\%$

Where:

B = weight of fraction before (g)

A = weight of fraction after (g)

2.11 Kraft Pulping

Two series of kraft cooks were conducted using a similar method to Hatton and Gee (1994).

First Series

Kraft pulping was performed on both infested and sound sapwood and heartwood samples at different positions along the tree (2m, 4m, 6m, 8m, 10m and 12m) to give a total of 24 samples. A representative aliquot of accept chips from each of the 24 samples were cooked in small bombs (50g, oven-dry charge) and placed in a 28L Weverk laboratory digester. All 24 samples were cooked at three different H-factors (a numerical value that represents the cooking variables, time and temperature) starting with a H-factor of 900 (time at temp. = 47 min.), then followed with 1200 H-factor (time at temp. = 66 min.) and 1500 H-factor (time at temp. = 85 min.) for a total of 72 samples. Table 1 summarizes the kraft pulp cooking conditions that were used for the first series.

Table 2-1. Kraft pulp cooking conditions for series I.

Wood Air Dry Weight	53-55g
Wood Oven Dry Weight	50g
Liquor Volume	225mL
Effective Alkali	16% (% oven-dry weight of wood)
Sulfidity	25%
Liquor:Wood	4.5:1
Maximum Cooking Temperature	170°C
Time to Temperature	86 minutes

For both series of kraft cooks, the black liquor was collected and the pulp was washed extensively with water, and placed into a pulp disintegrator for 15 minutes. The resulting pulp was filtered and washed until filtered wash water was clear. The pulp was dried over night at 150°C, weighed for total pulp yield, and moisture content was determined for the first series. The pulp samples were then further screened and washed on a CAE 200 mesh Laboratory Flat Screen to remove shives for Kappa number analysis.

Second Series

The second series of kraft pulping was undertaken to produce large quantities of pulps with Kappa numbers of approximately 30 for handsheet and fibre analysis. Only the butt section and crown wood were analyzed to produce a total of 8 samples. A representative aliquot of the accept chips from each of the 8 samples were cooked (900g, oven-dry charge) in a 28L Weverk laboratory digester. The infested sapwood and heartwood chips samples were cooked at a H-factor of 1165 (time at temp. = 62 min.) to produce a Kappa number of 30, whereas the sound sapwood chips were cooked at a H-factor of 1254 (time at temp. = 78 min.) and the sound

heartwood was cooked at a H-factor of 1334 (time at temp. = 84 min.). The cooking conditions are summarized in Table 2.

Table 2-2. Kraft pulp cooking conditions for series II.

Wood Air Dry Weight	965-970g (for sound wood)
	971-990g (for infested wood)
Wood Oven Dry Weight	900g
Liquor Volume	4050mL
Effective Alkali	16% (% oven-dry weight of wood)
Sulfidity	25%
Liquor:Wood	4.5:1
Maximum Cooking Temperature	170°C
Time to Temperature	85 minutes

2.12 Black Liquor Analysis

Black liquor analysis for each sample was performed in duplicates for a total of 48 samples. The residual effective alkali (EA_{res}) of each black liquor sample was determined by barium carbonate precipitation and titration with hydrochloric acid to pH 8.3 (Kesler, 1966).

Initially, 100mL of deionized water was added to a 500mL graduated cylinder. 25mL of black liquor was pipetted into the graduated cylinder followed by the addition of 50mL of 10% barium chloride (Aldrich). Deionized water was then added to the graduated cylinder to make up 500mL of solution. The solution was capped and thoroughly mixed by shaking. The solution was then allowed to sit for sedimentation for 12 hours. After sedimentation, 50mL of the solution was pipetted into a 100mL beaker and placed into the 682 Titroprocessor (Brinkmann) for hydrochloric acid titration to pH 8.3. Readings for residual effective alkali (in % of 100g of

oven-dry weight of wood) were recorded by the 665 Dosimat system. The percentage of effective alkali consumed per 100g oven-dry weight of wood was calculated from the following equation:

(7) EA consumed (%) $= EA_T - EA_{res}$

Where:

$EA_T = 16\%$ (total percentage of effective alkali used prior to kraft pulping)

$EA_{res} = $ residual effective alkali after kraft pulping (%)

2.13 Kappa Number Determination

The residual lignin content of the pulp was determined using a modified standard TAPPI Method for Kappa Number of pulp T236 om-99.

Kappa number determination was performed in triplicate using oven-dried pulp samples for a total of 216 samples. Pulp samples were weighed out to the nearest 0.001g, which would consume approximately 50% of potassium permanganate. Moisture content was also determined for each sample according to the standard TAPPI Method T550. The pulp samples were further disintegrated in 100mL or less of deionized water until free of fibre bundles, and then transferred to a 1L Bomex beaker. All the pulp was removed by rinsing with deionized water (at approximately 25°C) to achieve a total volume of 425mL. The 1L Bomex beakers with pulp solution were placed in a Kappa auto-titrator (Man-Tech Associates Inc. PC Titration Plus with a Mega Beaker Autosampler) controlled by PC Titrate software. The pulp suspension was continuously stirred with automatic stirring rods. 25mL of 4.0N sulfuric acid (Fisher) and 25mL of 0.1N potassium permanganate (Fisher) were added to the pulp suspension simultaneously. At the end of 10 minutes, the reaction was stopped by the addition of 10mL of 1.0N potassium iodide (Fisher) to the pulp mixture. A back titration, with 0.1 N sodium thiosulfate (Fisher), was

then performed to titrate the free iodine. Blank Kappa determination was also performed using the same method as described above, however, omitting the pulp.

In order to account for variations in permanganate consumption varying from 10-70%, the kappa number calculation can be modified, however the amount of pulp used varies for each sample in order to achieve a pulp consumption of sodium thiosulfate equivalent to 50% of that consumed by the blank.

Kappa Number is calculated as follows:

$$(8)\ K = \frac{p \times F}{W} \times (1+0.013(25-t)) \qquad p = \frac{(b-a) \times N}{0.1}$$

Where:

K = kappa number

p = amount of 0.1N permanganate actually consumed by the test specimen (mL)

b = amount of thiosulfate consumed in the blank determination (mL)

a = amount of thiosulfate consumed by the test specimen (mL)

N = normality of the thiosulfate

F = factor for correction to 50% permanganate consumption

W = weight of moisture-free pulp (mg)

t = reaction temperature at 5 min (°C)

2.14 Handsheet Preparation

Handsheets were prepared according to Pulp and Paper Technical Association of Canada (PAPTAC) Standard Testing Method C.4. Five representative samples for each kraft pulp (second series) were beaten in a PFI mill for 0, 3000, 6000, and 12,000 revolutions. Initially, the moisture content of each of the pulp samples was determined and suitable amounts of each pulp were weighed to produce 15 handsheets (60 g/m^2 oven-dry weight) for a total of 560 samples, of

which 5 representative samples were chosen for physical testing, and 3g of pulp were used for freeness determination.

A suitable amount of pulp was weighed (to an accuracy of 0.001g) which was equivalent to approximately 24g of oven-dry fibre. The pulp was disintegrated in 1 L of deionized water in a standard British blender, until the pulp was thoroughly dispersed in slurry. After disintegration, the stock was emptied into a bucket and diluted to 7L with deionized water at 23°C. Freeness was determined at 20°C according to the Pulp and Paper Technical Association of Canada (PAPTAC) Standard Testing Method C.1 using a Canadian Standard Freeness Tester (R. Mitchell Co.). One trial handsheet was made for each sample to determine the volume of pulp slurry required to produce a standard 1.2g oven-dry (60g/m^2 grammage) handsheet. For consistency determination, the stock was stirred and 400mL was measured and added to the deckle of the British hand-sheet machine half filled with distilled water. The deckle was then filled with distilled water to the 7L mark. The pulp solution was dispersed with a perforated stirrer by moving the stirrer down and up 6 times. Water was further drained from the deckle by opening the drain cock of the machine. The pulp sheet was removed from the wire mesh using two standard blotters and oven-dried to a constant weight. The volume of pulp suspension required to produce a 1.2g oven-dry handsheet was then calculated by the following formula:

(9) Volume of Pulp Suspension for 1.2g oven-dry handsheet = $\dfrac{400mL \times 1.2g}{W_T}$

Where:

W_T = weight of oven-dry trial handsheet (g)

After the volume of pulp suspension was determined, 15 handsheets were prepared using the same method described above, and then placed into a British standard press. The handsheets were initially pressed at 345kPa for 5 minutes, and then followed by a second press at 345kPa for

42

2 minutes. The handsheets were removed from the press and then dried in a standard conditioning room with a temperature of 23°C and a relative humidity of 50% for 24 hours. Five handsheets were then chosen for physical analysis.

2.15 Handsheet Properties

Handsheet physical properties were tested according to the PAPTAC Standard Method D.12. Non-destructive analysis for handsheet grammage (PAPTAC D.3), thickness using a Precision Micrometer (PAPTAC D.4), smoothness using Smoothchek (PAPTAC D.29), and porosity using a Genuine Gurley 4340 Automatic Densometer (PAPTAC D.14) were initially measured. The handsheets were then cut according to standard methods and destructive test were measured. Tensile strength using a Model 4202 Universal Testing Instrument (PAPTAC D.34), bursting strength using a Mullen Tester Model C (PAPTAC D.8), tearing strength using an Elmendorf Tearing Tester (PAPTAC D.9), and zero-span breaking strength using a Pulmac Zero Span TroubleShooter (TAPPI T231 cm-96) were examined.

2.16 Fibre Quality Analysis of Wood

A random sample of 50g of oven-dry wood chips from both sapwood and heartwood, infested and sound as well as from the topwood and butt section were selected. A sub-sample of each class of chips was then cut into 5 slivers in approximately 2mm × 2mm × 30mm dimensions. The 5 slivers were placed into test tubes with Franklin solution (1:1 ratio of glacial acetic acid (BDH) and 30% peroxide (Fisher Scientific)) for 48 hours at 70°C. Following the reaction, the maceration solution was removed by emptying the mixture into coarse crucibles and rinsing with deionized water. The pulped wood was vacuum filtered with several washings of deionized water until a neutral pH was achieved. The pulped wood was then placed into pre-weighed 20mL scintillation vials and dried in a 105°C oven for 12 hours to determine the oven-

dry weight. The wood samples were further immersed in 10mL of water and stored at 4°C for fibre analysis.

The wood samples were mixed and the fibres were completely separated in 1L of distilled water using a Hamilton Beach mixer. Using a 50mL scoop, the solution was stirred to an equal consistency and three 50mL scoops were removed to a 5L beaker and the weight was recorded. The solution was further diluted to 4L and the weight was recorded. A waffle mixer was used to mix the 4L solution and approximately 50mL was transferred to a 700mL beaker and the weight was again recorded. This sample was then autodiluted to 600mL volume in the Fibre Quality Analyzer (Code LDA96 OpTest Equipment Inc.). Fibre quality analysis was performed in triplicates for a total 24 samples. The final dilution was adjusted to a target fibre frequency between 10-20 fibres per second. Fibre length and fibre coarseness were evaluated by the FQA using the following equations:

(10) Coarseness (mg/m) = $\dfrac{\text{Mass of oven dried fibre tested (mg)}}{L_T(m)}$

L_T (total fibre length) = Fibre total × Ln[mean arithmetic length (mm)] × 1m/1000mm

(11) $L_n = \dfrac{\sum n_i L_i}{\sum n_i}$

Where:

Ln = mean arithmetic length (mm)

n = fibre count

L = contour length (mm)

2.17 Fibre Quality Analysis of Pulp

Fibre Quality (FQ) Analysis of kappa 30 pulp samples from sound and infested heartwood and sapwood as well as from the topwood and butt section (fibre length and fibre coarseness) was determined according to a modified method from the FQ Analyzer operation

manual (Code LDA96, OpTest Equipment Inc., Hawkesbury, ON, 1996). Fibre quality analysis of pulp samples was performed in triplicates for a total of 24 samples.

One handsheet for each sample was torn in half and one half of the handsheet was labeled, weighed and then oven-dried for 24 hours at 105°C. Moisture content was determined for each sample. Using the other half of the handsheet, approximately 0.03200g of representative pulp from each sample was carefully teased away (avoiding shive) from the air-dry pulp sample and measured to a precision of 0.00001g. The pulp samples were placed in labeled scintillation vials and the vials were filled with deionized water to allow the pulp to soak for 12 hours. After 12 hours, the vials were vigorously shaken to disperse the fibers. A 4L beaker was then placed on a scale and the scale was tared. The vial with pulp slurry was then emptied and rinsed with deionized water into the 4L beaker. Deionized water was further added into the 4L beaker to bring up the volume to approximately 4L. The weight of the 4L beaker with pulp suspension was recorded. The 4L beaker was then removed from the scale using a 50mL scoop, and the pulp suspension was stirred using the waffle mixer to an equal consistency. Approximately 220 ± 10g (recorded to the nearest 0.01g) were removed from the 4L beaker into a 600mL beaker (previously 'zeroed' on a scale) using the 50mL scoop. This aliquot of pulp suspension was then auto-diluted and auto-sampled by the FQ Analyzer. The approximate aliquot weight of pulp slurry depends on the fibre content of each sample where fiber frequency should be between 10-20 EPS (events per second) during the FQ analysis.

Fibre lengths (expressed as arithmetic means) are calculated from the following equation:

(12) Mean arithmetic length $L_n = \dfrac{\Sigma n_i L_i}{\Sigma n_i}$

Where:

n = fibre count

L = contour length (mm)

2.18 Statistical Analysis

Statistical analysis was conducted using a SPSS-X Version 10 software program. Two-way analysis of variance (ANOVA) was performed on the wood quality and chemistry analyses to evaluate whether significant differences between samples at the 95% confidence level were apparent.

CHAPTER 3. THE EFFECT OF MOUNTAIN PINE BEETLE ATTACK ON WOOD CHARACTERISTICS

3.1 Introduction

As the mountain pine beetle infestation continues to expand at significant rates, there is a considerable volume of dead material available to the forestry industry in British Columbia. Although the dead timber is a large reservoir of potentially utilizable material, it may also have adversely altered wood and fibre characteristics. The principle deterrent to using grey stage mountain pine beetle killed wood is that dry wood (wood below fibre saturation point) is generally believed to be of little commercial value due to checking, blue stain and low chip recovery.

3.2 Moisture Content

Following the attack by mountain pine beetles and prior to death, lodgepole pine trees deteriorate through the inoculation of blue stain fungi. After death, several levels of deterioration are observed, which include additional invasion of blue stain, reduction in moisture content, loss of foliage and bark, development of stemwood checks and the onset of decay (Koch, 1996). Reid et al. (1967) found that after the beetle constructs its egg gallery vertically up the stem, the tissues surrounding the gallery undergo several changes in the following sequence: (i) formation of white streaks on the sapwood surface; (ii) moisture loss in the sapwood and in the inner bark; (iii) hydrolysis of starch; (iv) bark adhering to the sapwood; (v) resinosis; (vi) death of cells; and (vii) traumatic duct formation. An important consequence at the onset of beetle attack is therefore, the severe water stress that it causes.

Water is an essential constituent of the living tree. The xylem or wood of the tree not only provides mechanical support and storage of food, but also provides the conduits by which water transports nutrients from the roots to the foliage. The active transport of water is restricted

to the sapwood, and more specifically to the most recently formed sapwood (Skaar, 1972). Skaar (1972) and Siau (1984) suggest that the process by which water is carried from the roots to the foliage is predominantly due to the diffusion pressure shortage as a result of the transpiration of leaves. Liquid water is pulled upward in the water column in the sapwood and this replaces the water, which has transpired and is consumed during photosynthesis. However, when a tree is cut or wounded, the mechanism of water transport is interrupted. The inherent moisture in wood is located in the cell wall as bound water or in the cell cavities in vapour or liquid form. The liquid water in the cell cavities is referred to as free water. When wood dries, water initially leaves the cell cavity, as the forces holding the water are not as strong as those holding the water in the cell wall. When the cell wall is fully saturated, but the cell cavity is empty of liquid water, this is called the fiber saturation point. It usually ranges from 25-35 percent of the dry weight of the wood (Skaar, 1972; Siau, 1984).

As expected in sound trees, the moisture content in the sapwood was shown to be higher than that of the heartwood (Figure 3-1). This can be explained by the fact that heartwood does not contain living cells and does not function in conducting water from roots to foliage. Sapwood, however, contains a significant proportion of living cells, which continue to engage in metabolic processes, and it is through sapwood that water and nutrients are transported from roots to foliage (Koch, 1996). The infested sapwood and heartwood also had substantially lower moisture content when compared to the sound sapwood and heartwood. The sound sapwood had an average moisture content of 110%, whereas the infested sapwood had an average of 25% which is an 85% reduction in moisture content. In contrast, the average moisture content of the sound heartwood was 34% and the infested heartwood had a 27% moisture content. The results concur with other studies, which demonstrated that the moisture content of logs from beetle-killed lodgepole pine were frequently below 30% of oven dry weight (Reid, 1961; Giles, 1986).

Figure 3-1. Moisture content of sound and infested lodgepole pine sapwood and heartwood at different tree heights for a total of 24 samples. Error bars indicate range.

Reid (1961) found that the moisture content of sound sapwood varied from 100 to 170%, and that the heartwood was typically about 30%. In addition, Reid (1961) found that moisture content varied at different levels of the stem; however, he concluded that there was no consistent trend apparent. In the current study, we observed that sound sapwood moisture content does not appear to vary with tree height; however, the sound heartwood moisture content tends to decrease with increasing tree height. Similarly, the infested sapwood and heartwood moisture content also tends to decrease slightly as tree height increases.

Moisture content reduction in sapwood generally occurs in the first year after mortality, whereas the heartwood loses it moisture more slowly. The substantial reduction in moisture content of the sapwood is believed to be associated with the presence of blue stain in the sapwood, and with the successful development of the bark beetle broods. However, it has not been established which or any of these conditions are causal and which are merely effect (Reid, 1961). Nebeker *et al.* (1993) suggest that after beetle attack there appears to be secondary symptoms arising from water stress which include: (1) rapid decrease in turgor pressure of the living cells of the bole (indicated by the lower oleoresin exudation pressure immediately after beetle infestation); (2) aspiration of tracheids and associated loss in volume of water transported to the crown; and (3) drying of outer bole tissues as water is withheld from below and within. Again the cause and effect relationships have not been explained or extensively studied. Nebeker *et al.* (1993) further suggested that the water stress may be likely due to the blockage of xylem tracheids by toxic fungal metabolites produced from fungal hyphae, or by aspiration of tracheids when propagating hyphae penetrate the tracheid walls. In addition, although the role of staining in host colonization is not entirely clear, earlier investigations demonstrated that stained portions of the sapwood were generally drier than unstained portions, and that water conduction was impaired in the stained portions (Nelson, 1934; Bramble and Holst, 1940). Any or all of the

above may occur after fungal inoculation, but none have been proven responsible for the loss in moisture content and subsequent tree death (Nebeker *et al.*, 1993).

3.3 Wood Density

As a porous material, wood is made up of a matrix of fibre walls and air spaces. The air spaces exist primarily in the form of lumens and to a lesser degree as voids within fibrous walls. The fibre wall has three major components: cellulose, hemicellulose and lignin. Wood density is a measure of the total amount of solid-wood substance in a piece of wood (Jozsa and Middleton, 1994). Length and diameter of cells, proportion of tissue types, thickness of cell walls, proportions of earlywood and latewood, percentages of cellulose and lignin, proportions of extractives, and amount of trace materials all have been shown to affect the density of wood (Jozsa and Middleton, 1994; Koch, 1996).

The wood density of the permeability cores showed that the infested sapwood (390-425 kg/m^3) and heartwood (395-435 kg/m^3) were statistically less dense than the sound sapwood (445-480 kg/m^3) and heartwood (470-480 kg/m^3) (Figure 3-2). Koch (1996) suggested that the decline in density in infested (dead) trees is a function of time since death. The decrease in density in the infested wood also suggests that the chemistry of the infested wood may be altered compared to that of the sound wood. Koch (1996) reported that sapwood density ranged from 401-476 kg/m^3 and between 405 to 502 kg/m^3 for heartwood in live trees. Both Koch (1996) and Lieu *et al.* (1979) found that heartwood had significantly higher density values than sapwood and this trend is also demonstrated in the current study. Lieu *et al.* (1979) hypothesized that the higher density of heartwood is likely due to the greater extractive content present.
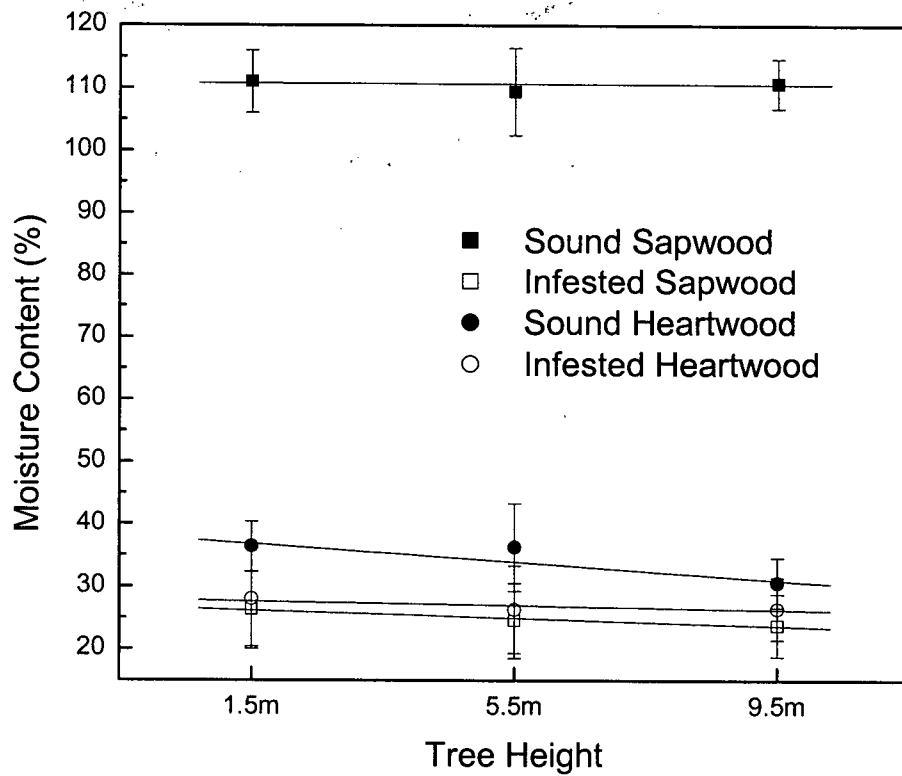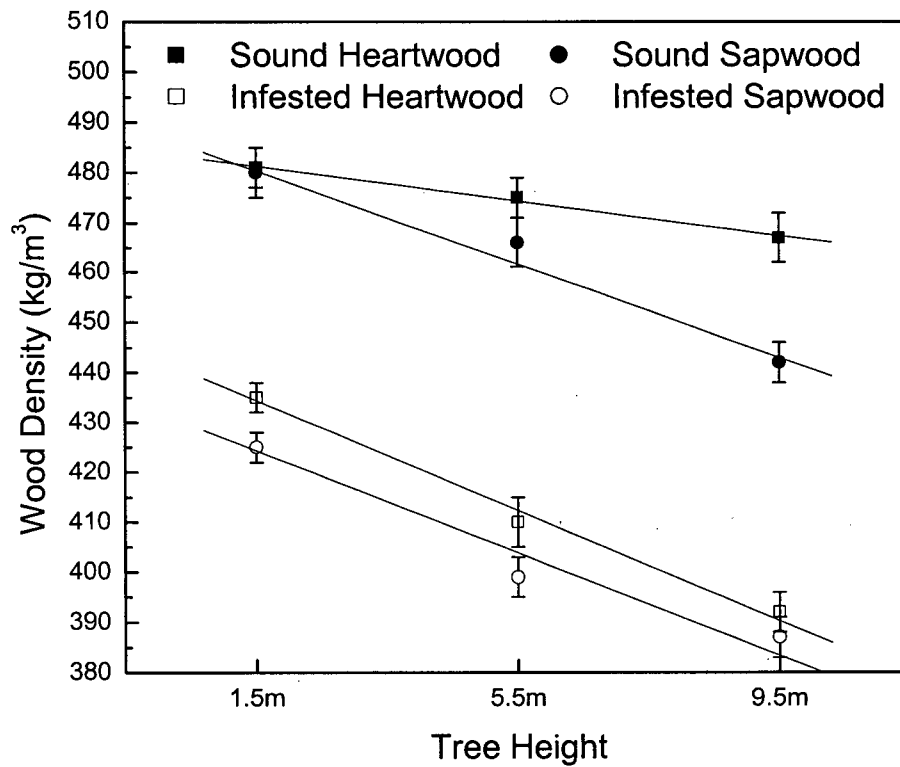
Figure 3-2. Wood density (at 12% moisture content) of sound and infested lodgepole pine sapwood and heartwood at different tree heights for a total of 36 samples. Error bars indicate range.

In addition, for both sound and infested heartwood and sapwood, significant differences in density distribution at different tree heights were observed, as density tended to decrease with increased tree height. This may be attributed to the fact that with increased tree height, more juvenile wood is present and therefore density is lower. Juvenile wood tends to have shorter wood cells compared to mature cells and have different cell structures. Juvenile wood has relatively few latewood cells and has a high proportion of thin-wall layered cells. The end result is lower density and concomitant lower strength (Jozsa and Middleton, 1994).

3.4 Wood Extractive Content

Wood extractives are a heterogeneous group of compounds, which can be extracted from wood by means of polar and non-polar solvents. Basically, extractives are compounds, which are soluble in organic solvents. Although extractives are considered nonstructural wood constituents, they represent a significant diversity of individual compounds of both lipophilic and hydrophilic molecules in wood. Extractives perform requisite biological functions; for example, fats and waxes are responsible for energy supply, whereas phenolics, resin acids and some terpenoids obstruct and hinder insect and microbial damage to the tree. The extractives are concentrated in the resin canals and the ray parenchyma cells; lower amounts are also found in the middle lamellae, intercellular and cell walls of tracheids. Typically, resin acids are located in the resin canals, whereas the fats and waxes are found in the ray parenchyma cells (Stenius, 2000; Sjostrom, 1993).

Extractives are typically classified as aliphatic and alicyclic (wood resins) compounds, phenolics, gums and other compounds. Wood resins are comprised of four classes of lipophilic components: fats and fatty acids, steryl esters and sterols, terpenes/terpenoids and steroids, and waxes. Typically, triglycerides (Figure 3-3) are the predominant form of fats (glycerol esters of fatty acids) in wood. The fats are partially hydrolyzed during wood storage but are also

extensively hydrolyzed during alkaline pulping. During storage or pulping, the free fatty acids and glycerol are liberated. Terpenes usually evaporate during solvent extraction when wood resin is gravimetrically determined. Terpenoids typically make up the resin of secretory tissues (resin canals) and the basic structural component of terpenoids as well as steroids is the isoprene unit, 2-methyl-1,3-butadiene. The common terpenes and terpenoids found in softwoods are monoterpenes, monoterpenoids, sesquiterpenes-terpenoids, and diterpenes-terpenoids (Figure 3-4). Sterols and steryl esters (Figure 3-4) generally occur in parenchyma cells in the form of fatty acid esters and waxes, which are long chain alcohols and are generally a minor component of wood resin (Stenius, 2000; Back and Allen, 2000).

The phenolic extractives are mainly located in the heartwood and bark. The phenolics are generally a variety of aromatics and are derived from the phenylpropanoid (C_6C_3) structure. The phenolic compounds include stilbenes, lignans, hydrolyzable tannins and flavonoids (Figure 3-5). Stilbenes and lignans are extractives generally found in the heartwood and bark of pines with small traces found in the sapwood. Both types of phenolics have fungicidal properties that help protect the tree against microbiological attack. Lignans are primarily responsible for the colouration of heartwood. Hydrolyzable tannins are uncommon in wood and some flavonoids have been shown to inhibit delignification in pulping (Sjostrom, 1993; Stenius, 2000; Back and Allen, 2000).

Some extractives are classified as gums, which are exuded as viscous fluids at the site of injuries or wounds. One of the common types of gums is oleoresin, which is a lipophilic gum exuded on the stem from secretory tissues of the resin canals. Generally, the volatile resin from the resin canals such as monoterpenes evaporate after being exposed to air while leaving the non-

Fatty Acids

/\/\/\/\/\/\COOH   Palmitic acid (16:0)

14 /\/\/\/\/\COOH 1   14-methyl hexadecanoic acid
16   (anteiso- 17:0, shorthand 17:0 ai)

18   10 9 /\/\/\=/\/\/COOH 1   Oleic acid (9-18:1)

/\/\/=\/=/\/\/COOH   Linoleic acid (9, 12-18:2)

\/=\/=\/=/\/\/COOH   Linolenic acid (9, 12, 15-18:3)

/\/_/_/\/=/\/COOH   Pinolenic acid (5, 9, 12-18:3)
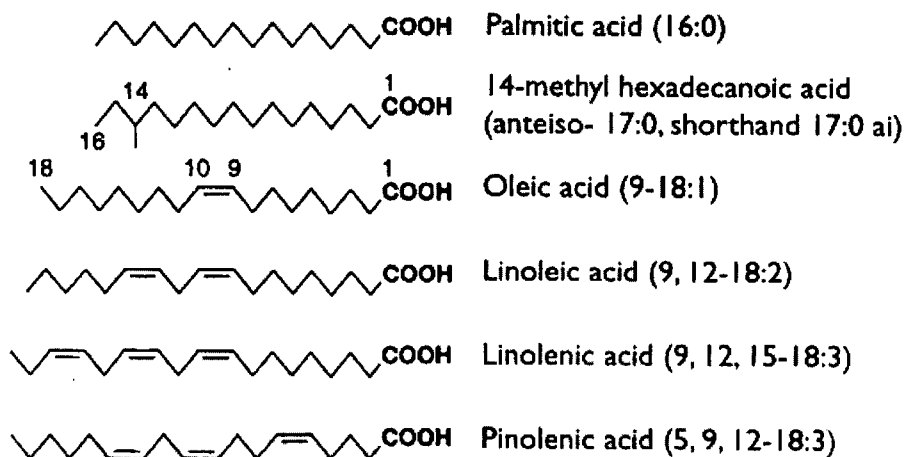
Triglycerides (triacylglycerols):

/\/\/=\/=/\/\/CO – O – CH₂
/\/\/=\/=/\/\/CO – O – CH   Trilinolein
/\/\/=\/=/\/\/CO – O – CH₂

Fatty acid esters:

$R_1 - \overset{O}{\overset{\|}{C}} - O - [CH_2]_n - CH_3$ (n = ca. 19 - 23) R_1 = fatty acid chain
"waxes"

$R_2 - \overset{O}{\overset{\|}{C}} - O -$ sterol
steryl esters R_2 = chains of saturated
and unsaturated, mainly
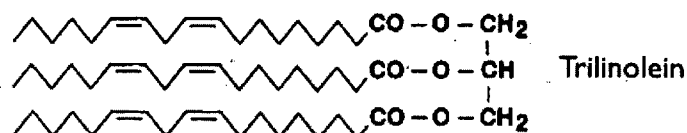
$R_2 - \overset{O}{\overset{\|}{C}} - O -$ triterpenyl alcohol $C_{14} - C_{20}$ fatty acids
triterpenyl esters
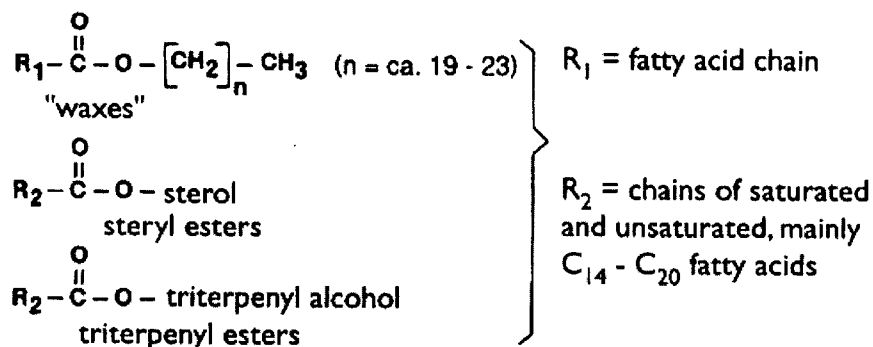
Figure 3-3. Predominate parenchyma wood resin constituents such as fatty acids, triglycerides and fatty acid esters (Back and Allen, 2000).

MONOTERPENES AND MONOTERPENOIDS

(-)-Limonene   α-Pinene   3-Carene   Camphene   Borneol
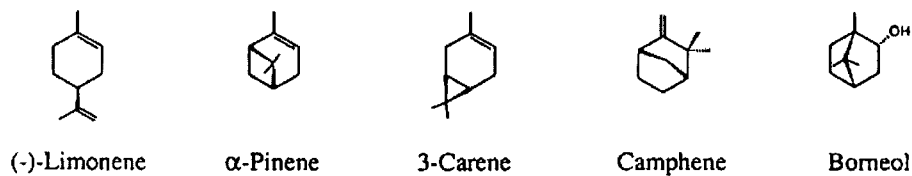
SESQUITERPENES AND SESQUITERPENOIDS

Farnesene   α-Muurolene   α-Cadinol   Longifolene
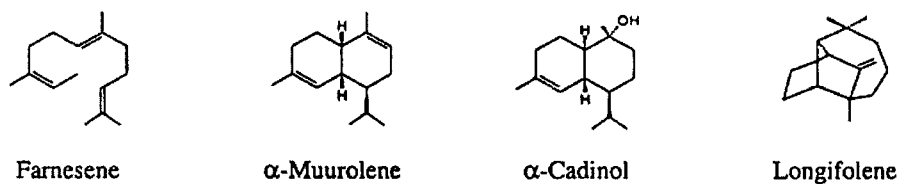
DITERPENES AND DITERPENOIDS

Thunbergene   Abietic acid   Pimaric acid
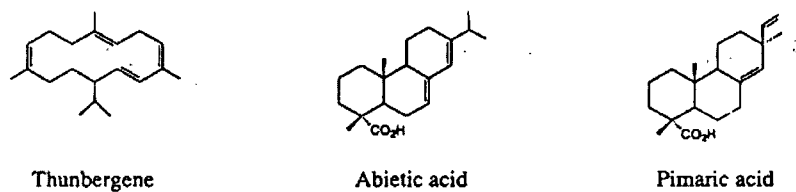
TRITERPENES AND STEROIDS

Cycloartenol   Betulinol   Sitosterol

sitosterol   citrostadienol   cycloartenol
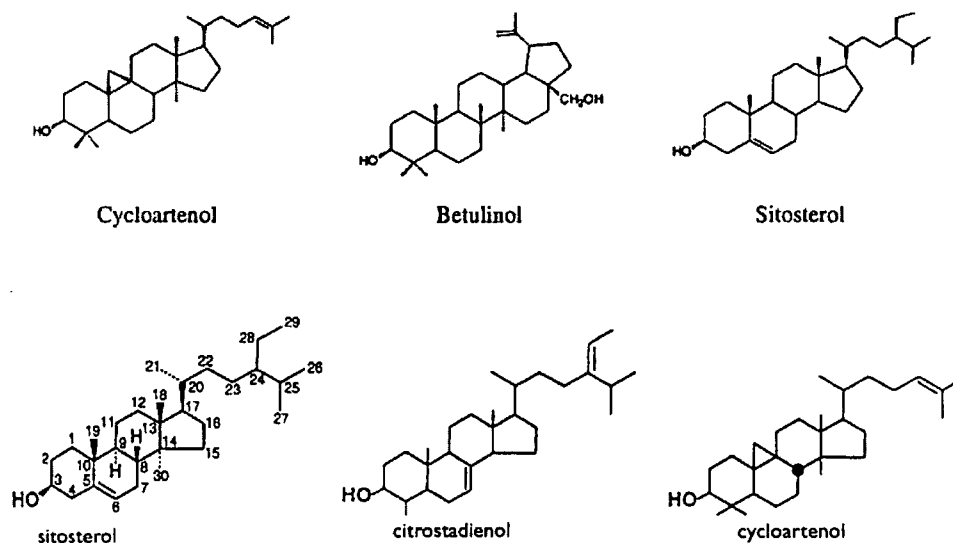
Figure 3-4. Chemical structures of some common terpene, terpenoid, steroid and sterol wood extractives (Stenius, 2000).
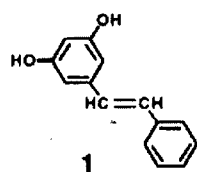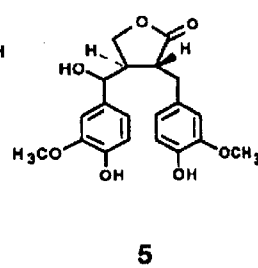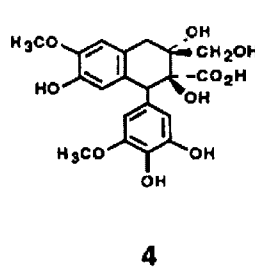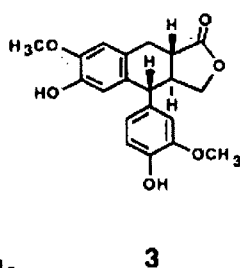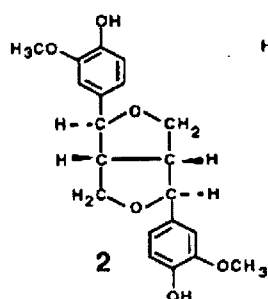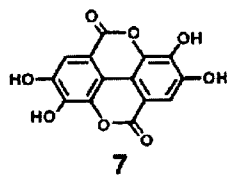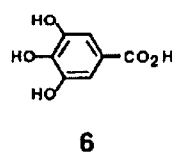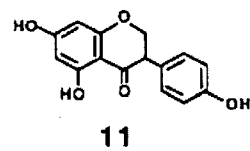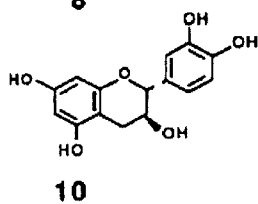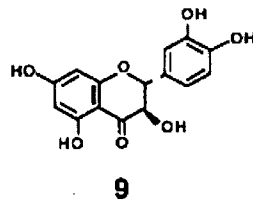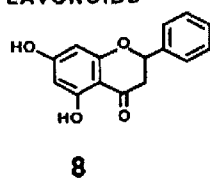
Figure 3-5. Chemical structures of some common phenolic extractives and associated constituents (Sjostrom, 1993).

volatile terpenoids to seal the wounds. Additionally, other extractive compounds include sugars and amino acids (Back and Allen, 2000).

Extractives are present in low quantities (less than 5%) in the wood of coniferous trees from temperate zones and generally are found in branch bases, heartwood, roots, and areas of irritation. The amount and composition of wood extractives and wood resin can vary significantly between trees. Generally, extractive content is higher in heartwood than sapwood, and the resin content generally decreases with stem height (Fengel and Wegener, 1984). Quijada (1967) found that the extractive content of corewood decreased with tree height up to 15-20 feet and was then relatively constant in the upper tree. In contrast, the resin present in sapwood is lowest at the base of the tree and increases with height (Quijada, 1967). Moreover, tree age is the most important factor affecting extractive content because it affects the sapwood/heartwood ratio, and density, which is related to juvenility. In addition, rapidly growing trees have a higher extractive content than slowly grown trees, as earlywood contains more extractives than latewood. The environment can also modify extractive content (Fengel and Wegener, 1984; Kim, 1988). Extractives are important from an industrial perspective because they can impact the pulping process by causing pulp colour reversion and by forming pitch deposits. Economic losses related to pitch problems in kraft mills have been estimated to account for as much as 1-2 % of sales (Back and Allen, 2000).

The percentage of extractive content in sound lodgepole pine varied from 1 to 2% in sapwood and 2 to 4% in the heartwood (Figure 3-6 and Figure 3-7). These results correspond with Kim (1988), Shrimpton (1973), and Lieu (1979), who observed that green lodgepole pine contained moderate amounts of extractives ranging from 1 to 4%. Extractive content in the heartwood was higher compared to sapwood, as expected. However, it was apparent that infested sapwood had lower levels of extractives than the corresponding sound sapwood. Shrimpton (1973) hypothesized that the reduced level of extractives in infested sapwood could

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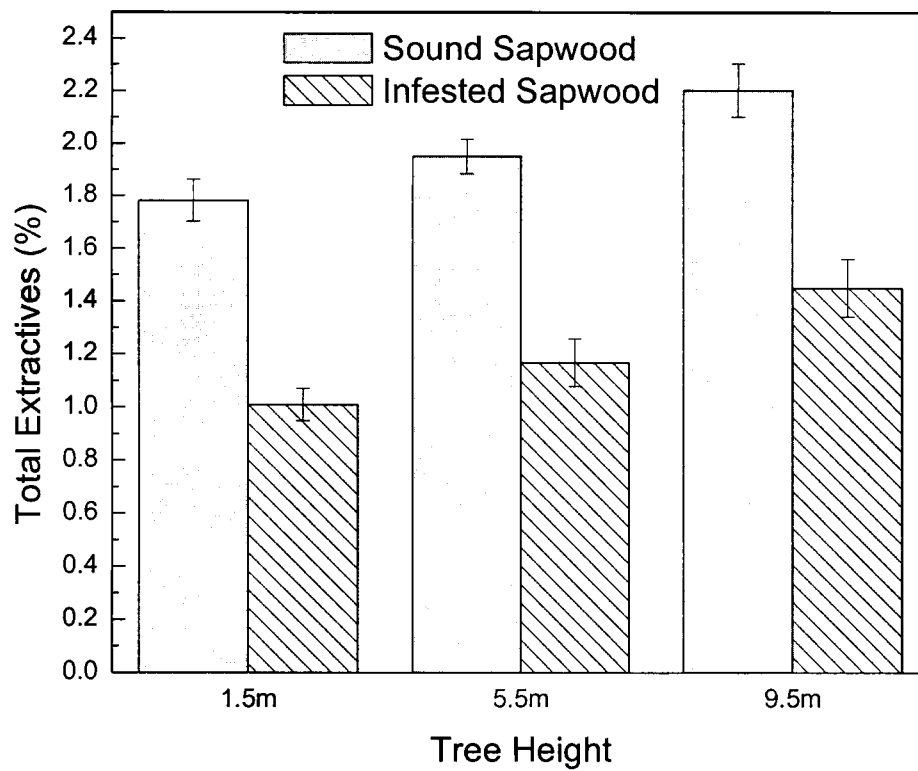
Figure 3-6. Total extractive content in sound and infested lodgepole pine sapwood at different tree heights for a total of 30 samples. Error bars indicate 95% confidence interval.
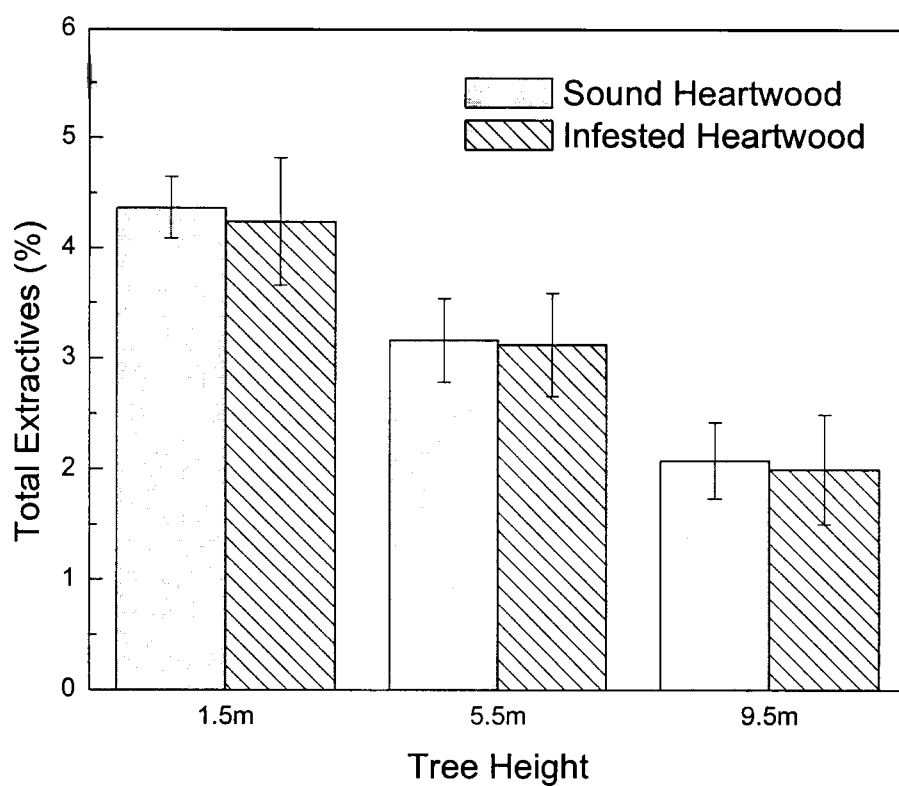
Figure 3-7. Total extractives in sound and infested lodgepole pine heartwood at different tree heights for a total of 30 samples. Error bars indicate 95% confidence interval.
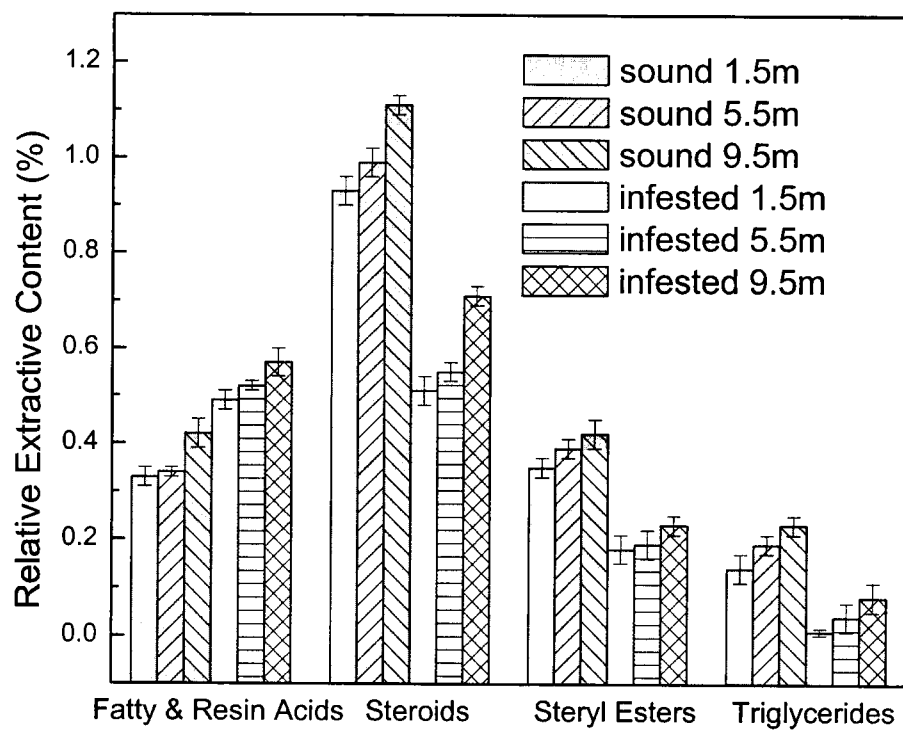
Figure 3-8. Relative proportion of extractives in sound and infested lodgepole pine sapwood at different tree heights for a total of 30 samples. Error bars indicate standard deviation.
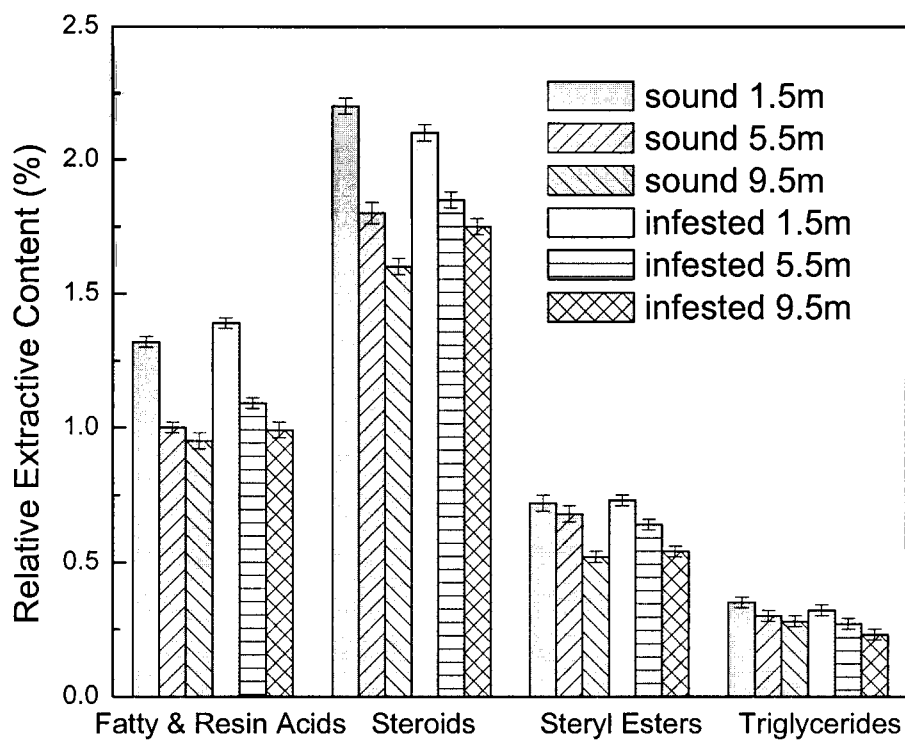
Figure 3-9. Relative proportion of extractives in sound and infested lodgepole pine heartwood at different tree heights for a total of 30 samples. Error bars indicate standard deviation.

be attributed to the attacking beetle and invading fungi that utilize the cellular components. The heartwood extractive content was not significantly different between sound and infested wood; however, it decreased as tree height increased in both sound and infested wood, which supports Quijada's (1967) findings. In the case of sapwood extractive content, there was a statistically significant difference in tree height, and between infested and sound wood. Extractive content in sapwood increased particularly towards the crown wood, which also concurs with the findings of Quijada (1967).

A comprehensive analysis of the individual classes of extractives indicated that the relative proportion of extractives in the infested sapwood had changed due to the beetle (fungal) infestation (Figure 3-8 and Figure 3-9). The infested sapwood generally had less extractives than sound sapwood and the results demonstrated a higher proportion of fatty and resin acids, and a lower proportion of steroids, steryl esters and triglycerides when compared to sound sapwood. It is fair to conclude that a decrease in these proportions of extractives is a result of fungal invasion such that fungus most readily degrades triglycerides, steryl esters and steroids (Shrimpton, 1973; Lieu *et al.*, 1979). Higuchi (1985) reported that as wood triglycerides are the most readily degraded extractive components and with the liberation of fatty acids, an increase in fatty acid components will be observed as indicated in our results. Back and Allen (2000) also noted that an increased presence of fatty acids in the sapwood may be due to early death of parenchyma cells. In addition, the relative proportion of extractives in both sound and infested sapwood tended to increase with tree height, which concurs with the trend demonstrated in total extractives content. The relative proportion of extractives in sound and infested heartwood were relatively similar, however, the extractive content tended to decrease with increased tree height. These results correlate with the trend found in the analysis of total extractives content.

Although extractives make up only a small percentage of the total chemical composition of wood, it has a significant impact on the pulp and paper process. Extractive content affects the

63

time required for chip seasoning, which is the outdoor storage of chips that allows for hydrolysis and oxidation of extractives to prevent pitch (wood resin deposits) and paper machine friction problems (Back and Allen, 2000). The results in the current study imply that chips from heartwood and the topwood section in the sapwood will require more seasoning as more extractives are present. Seasoning for the infested wood will not be a priority, as immediate use of this resource will be required to prevent further wood loss from decay.

3.5 Wood Lignin Content

Lignin is an abundant, aromatic polymer constituting 25-30% of conifers (Koch, 1996), and contributes to the mechanical strength of trees. Lignin is present in fine spaces within the cell wall where it acts as a bulking agent and reduces the dimensional changes in the wall. The rigidity of the lignin helps increase the cell wall stiffness. The composition and distribution of lignin varies within the cell wall and in the tree. The structure of lignin consists of phenylpropane units linked together in three dimensions. Lignin consists of derivatives from trans-p-coumaryl alcohol (the precursor of p-hydroxyphenyl monomers), trans-coniferyl alcohol (the precursor of guaiacyl monomers) and trans-sinapyl alcohol (the precursor of syringyl monomers) (Figure 3-10). Lignin of softwoods are primarily comprised of guaiacyl monomers (95%) with minor amounts of syringyl units. The most prevalent intermonomeric linkages in the formation of lignin macromolecules are β-O-4 ether linkages (C-O-C), which makes up between 40-60 % of most lignin bonds in softwoods. Other common linkages include carbon-carbon bonds and ester bonds (Figure 3-11) (Baucher *et al.*, 1998; Fengel and Wegener, 1984). During chemical pulping operations for the liberation of cellulosic fibres, the three linkages between the propane side chains and benzene rings are broken (Smook, 1992).

The klason lignin analysis demonstrated that sound (30.1%) and infested (29.8%) heartwood contained more lignin than sound (28.5%) and infested (27.2%) sapwood (Figure 3-
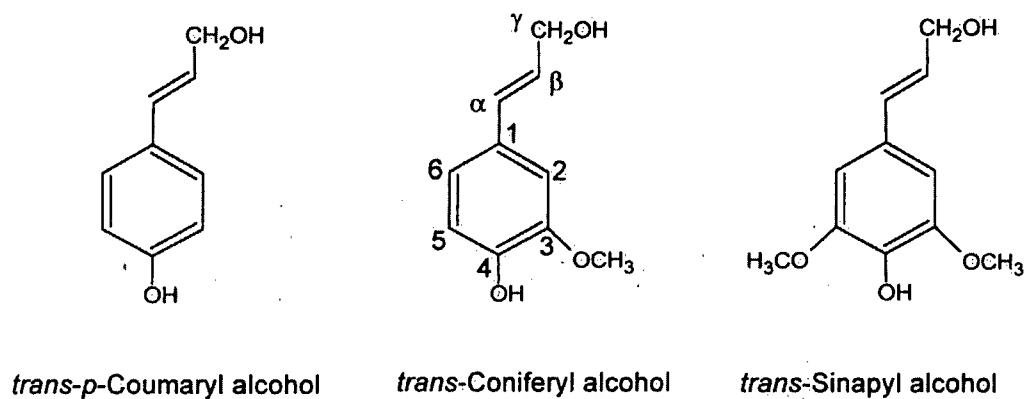
Figure 3-10. Chemical structures of three lignin precursors (Baucher *et al.*, 1998).

ETHERS

β-O-4
(40 - 60 %)

α-O-4
(5 - 10 %)

γ-O-4
(< 5 %)

5-O-4
(5 - 10 %)

γ-O-α
(< 5 %)

glyceraldehyde or
glycerol 2-aryl ether
(< 5 %)

CARBON-CARBON BONDS

5-5
(and 5-6)
(5 - 20 %)

β-5
(both ring and
open structures)
(5 - 10 %)

β-β
(< 5 %)

β-1
(< 5 %)

β-6
(and β-2)
(< 5 %)
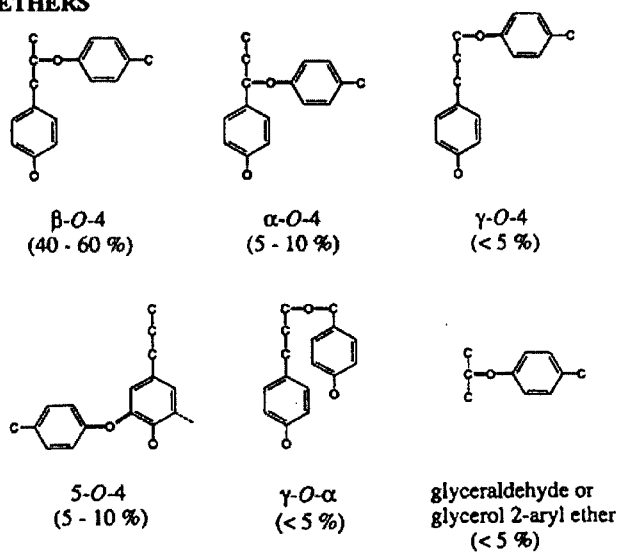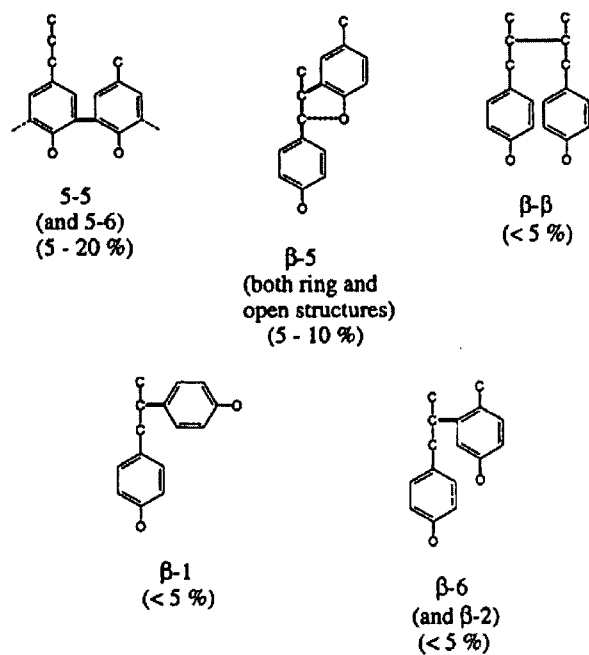
ESTERS

α-ester
(< 5 %)

γ-ester
(< 5 %)
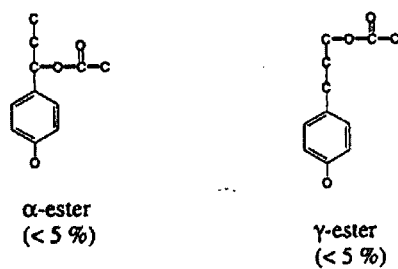
Figure 3-11. Types of linkages between phenylpropane units (Stenius, 2000).

12 and Figure 3-13). Lieu *et al.* (1979) reported similar results that lodgepole pine sapwood had a lower lignin content (26.85%) than heartwood (27.14%). Heartwood contains more juvenile wood than sapwood and sapwood is composed mainly of mature wood. Juvenile wood is known to contain a higher lignin content than mature wood and therefore, an increase in heartwood lignin content compared to sapwood may be found (Fengel and Wegener, 1984; Haygreen and Bowyer, 1996). There appeared to be no significant difference in the percentage of total lignin content between sound and infested heartwood as the results were statistically similar. The heartwood normally is not attacked as it contains defensive compounds (extractives), and requires more than a year for the beetles to reach the heartwood; which by then the larvae will have already developed into adults and have left the tree (Reid, 1962; Amman and Walter, 1983). In contrast, infested sapwood lignin content was significantly different in comparison to sound sapwood. These results correlated with the results found in the studies performed by Lieu *et al.* (1979) who suggested that the decrease in lignin content in the sapwood was attributed to beetle infestation. As blue stain fungi are the primary colonizers in mountain pine beetle killed wood, Scott *et al.* (1996) and Koch (1996) suggest that other fungal decay species are likely present and associated with the incipient decay that often is invisible and difficult to detect. Therefore, the decrease in lignin content may be attributed to the presence of the accompanying decay fungi. Current research is underway in determining the other fungal species present in mountain pine beetle killed wood following blue stain fungi.

In addition, sapwood and heartwood lignin content do not appear to vary with tree height. From the klason lignin analysis, we also found that infested sapwood and heartwood had significantly more acid soluble and less acid insoluble lignin than sound wood (Table 3-1 and
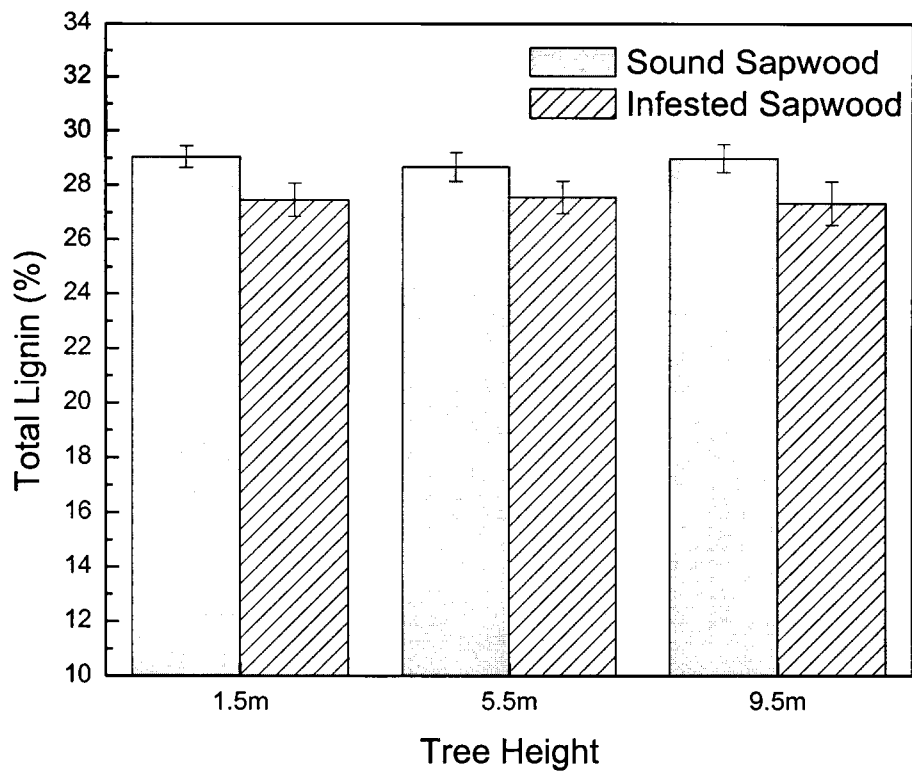
Figure 3-12. Total lignin content in sound and infested lodgepole pine sapwood at different tree heights for a total of 30 samples. Error bars indicate 95% confidence interval.
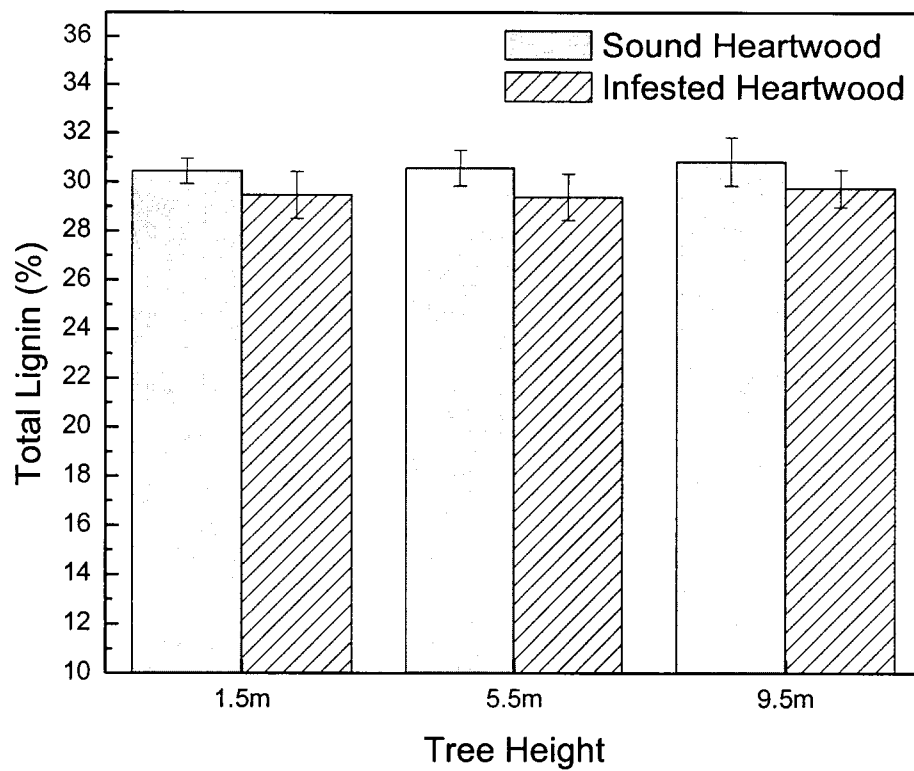
Figure 3-13. Total lignin content in sound and infested lodgepole pine heartwood at different tree heights for a total of 30 samples. Error bars indicate 95% confidence interval.

Table 3-2). The difference in acid soluble and acid insoluble lignin in the infested wood is likely a result of the presence of fungi associated with beetle attack (insoluble lignin may be slightly modified to increase the total amount of soluble lignin present), but requires further examination before a firm conclusion can be drawn. Again, there was no apparent trend in terms of lignin content with tree height. These results may in turn affect pulp quality in terms of kappa number, pulp yield and chemical consumption in the pulping process because chemical pulping processes separate wood fibres by dissolving away lignin (Jozsa and Middleton, 1994). The results imply that the infested sapwood and heartwood might be more readily pulped as there is significantly lower total lignin content and more acid soluble lignin.

Table 3-1. Acid soluble and acid insoluble lignin content in sound and infested lodgepole pine sapwood at different tree heights.

Tree Height (Sound)	Acid Soluble Lignin (%)	Acid Insoluble Lignin (%)	Total (%)
1.5m	1.25 (0.2)	27.79 (0.5)	29.04 (0.4)
5.5m	1.31 (0.2)	27.05 (0.3)	28.36 (0.5)
9.5m	1.29 (0.1)	27.28 (0.2)	28.57 (0.4)
Tree Height (Infested)	Acid Soluble Lignin (%)	Acid Insoluble Lignin (%)	Total (%)
1.5m	2.25 (0.2)	25.60 (0.4)	27.85 (0.3)
5.5m	2.33 (0.4)	25.31 (0.2)	27.64 (0.3)
9.5m	2.30 (0.2)	25.22 (0.2)	27.52 (0.5)

* Standard deviation shown in parentheses.

Table 3-2. Acid soluble and acid insoluble lignin content in sound and infested lodgepole pine heartwood at different tree heights.

Tree Height (Sound)	Acid Soluble Lignin (%)	Acid Insoluble Lignin (%)	Total (%)
1.5m	1.63 (0.3)	28.41 (0.2)	30.04 (0.3)
5.5m	1.55 (0.3)	28.65 (0.4)	30.20 (0.4)
9.5m	1.52 (0.3)	29.21 (0.2)	30.73 (0.5)
Tree Height (Infested)	Acid Soluble Lignin (%)	Acid Insoluble Lignin (%)	Total (%)
1.5m	2.45 (0.5)	27.00 (0.4)	29.45 (0.6)
5.5m	2.66 (0.3)	26.71 (0.3)	29.37 (0.4)
9.5m	2.47 (0.3)	26.85 (0.2)	29.32 (0.5)

* Standard deviation in parentheses.

3.6 Wood Carbohydrate Content

Holocellulose is defined as the sum of the polysaccharides (cellulose and hemicellulose) inherent in wood. Polysaccharides are complex molecules consisting of a large number of monosaccharide units joined together by glycosidic linkages. Monosaccharides are simple sugars, whereby D-glucose, D-mannose, D-galactose, D-xylose, and L-arabinose are the most common components of the cell wall polysaccharides in wood. Hemicellulose is a branched low molecular weight carbohydrate polymer, whereas cellulose is a linear, high molecular weight polymer. Cellulose is the main constituent of wood and comprises 40-45% of the dry substance in most wood species, and is located primarily in the secondary (S_2) cell wall. Hemicellulose, similarly to cellulose, functions as a supporting material in the cell walls, however, hemicelluloses are relatively easily hydrolyzed by acids and constitute 20-30% of the dry weight of wood. As for cellulose, which is initially joined to lignin, it requires intensive chemical

treatment in order to separate the structural constituents (Fengel and Wegener, 1984; Sjostrom, 1993; Kim, 1988).

Cellulose, the most prevalent molecule in wood, is a long, linear homopolysaccharide comprised of β-D-glucose monosaccharides connected by (1→4) glycosidic linkages (Figure 3-14A). Three hydroxyl groups per anhydrous glucose unit in cellulose are responsible for cell wall structural properties, as well as for several physical and chemical wood properties. The hydrogen bonding between the continuous cellulose molecules produce crystalline microfibrils interrupted by amorphous regions. The average degree of polymerization for wood cellulose is approximately 10,000. As cellulose is characterized by the chair conformation ($^4C_1$) of pyranose rings (Figure 3-14B) and consists of repeating units of disaccharide, 4-O-(β-D-glucopyranosyl)-D-glucopyranose (cellobiose), this makes the cellulose chain highly stable. There is a 180° angle of separation between the glucose units where the microfibrils are parallel to one another and hydrogen bonding occurs between the adjacent microfibrils (Figure 3-14C). Cellulose molecules fit appropriately together over long segments developing regions of crystallinity that are difficult to penetrate by solvents or reagents. The amorphous regions are more readily penetrated and therefore more susceptible to hydrolysis reactions (Zabel and Morrell, 1992; Sjostrom, 1993; Smook, 1992).

In contrast to cellulose, which is a polymer of glucose, hemicelluloses are polymers of several different sugars, which include glucose, mannose and galactose (which are hexoses) as well as xylose and arabinose (which are pentoses) (Figure 3-15). Uronic acids can also be found in hemicelluloses. Hemicelluloses are heterogeneous polysaccharides and have short chain lengths, side chains that are sometimes branches and a degree of polymerization of approximately 200. Softwood primary hemicellulose components are galactoglucomannans and arabinoglucouronoxylan (xylan). Galactoglucomannans (15-20%) consists of a linear backbone
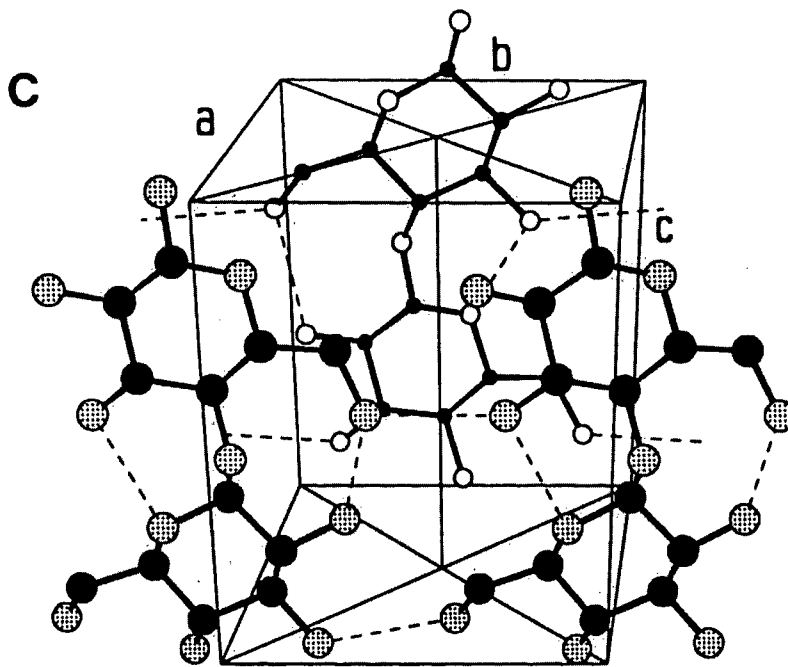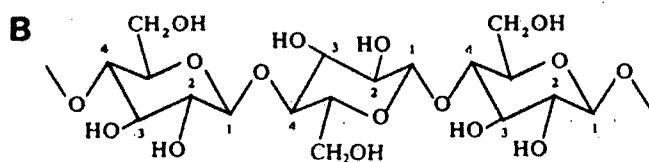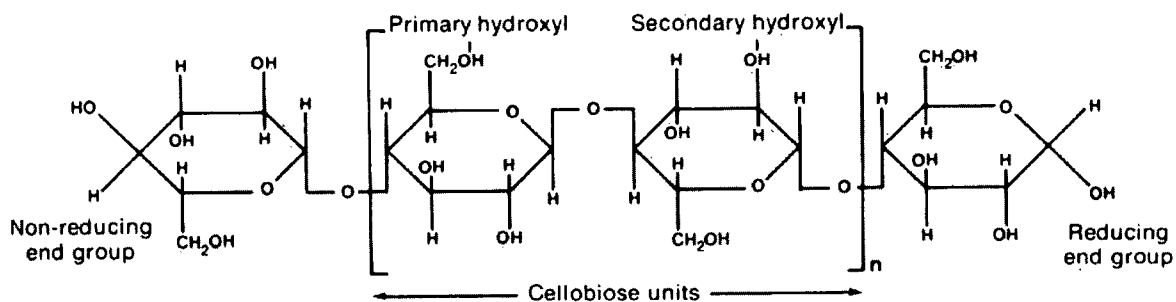
Figure 3-14. The cellulose polymer. (A) Chemical structure of cellulose (Smook, 1992). (B) Chemical structure of cellulose in the chair conformation (Zabel and Morrell, 1992). (C) The unit cell of parallel cellulose chains with hydrogen bonding in between (Zabel and Morrell, 1992).

73

β-D-Glucopyranose   α-L-Rhamnopyranose   α-L-Fucopyranose

α-L-Arabinofuranose   β-D-Xylopyranose   β-D-Mannopyranose

β-D-Galactopyranose   α-D-Galactopyranosyluronic acid   β-D-Glucopyranosyl-uro nic acid

β-D-Apiofuranose   2-keto3-deoxy-D-*manno*-2-octulosonic acid (Kdo)   3-deoxy-D-*lyxo*-2-hep tulosaric acid (Dha)

β-L-Aceric acid
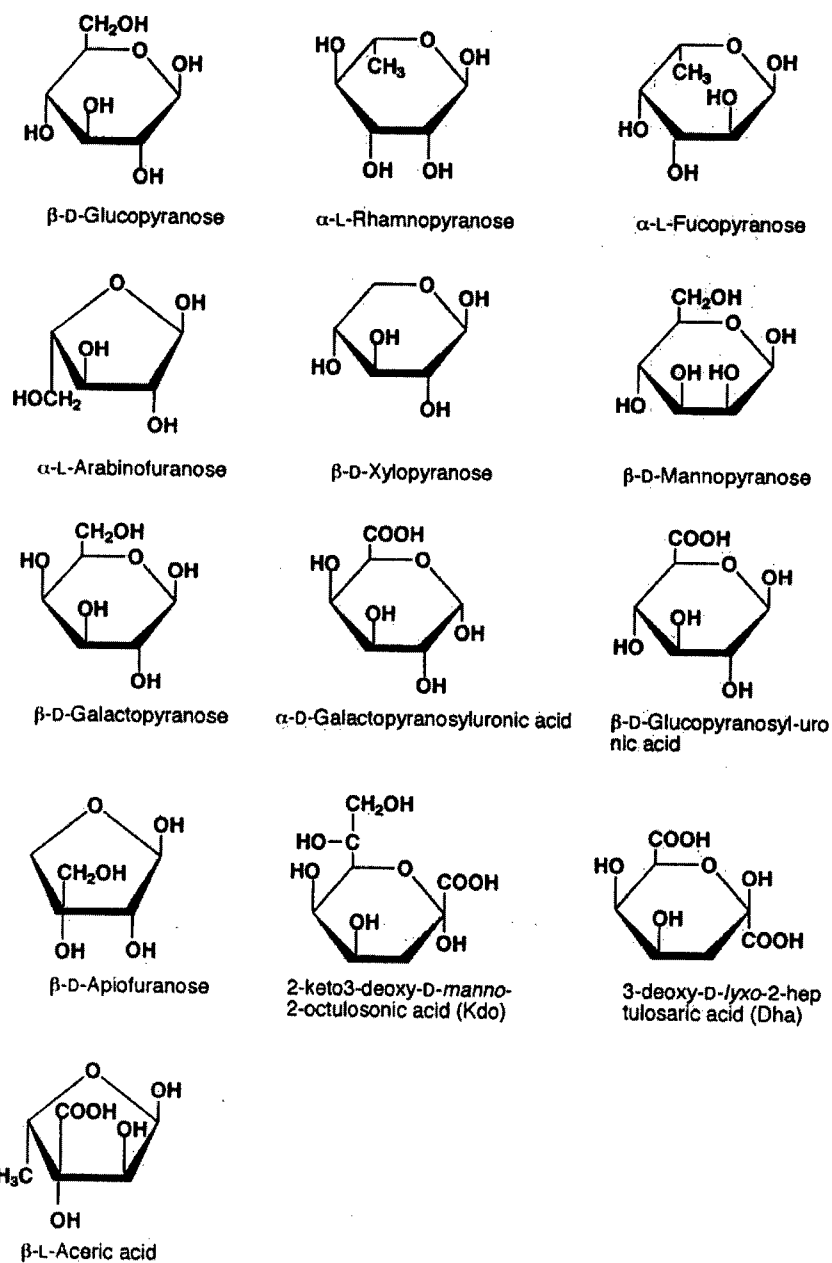
Figure 3-15. Chemical structures of common monosaccharides in the cell wall (Ishii and Shimizu, 2001).

74

→ 4)-β-D-Glc*p*-(1→ 4)-β-D-Man*p*-(1 ⟶[→ 4)-β-D-Man*p*-(1 ⟶]₂ →

⏜
6

↑

1
⏝
α-D-Gal*p*

Galactoglucomannan

→ 4)-β-D-Xyl*p*-(1 ⟶[→ 4)-β-D-Xyl*p*-(1 ⟶]₂ 4)-β-D-Xyl*p*-(1 ⟶[→ 4)-β-D-Xyl*p*-(1 ⟶]₄ →

⏜   ⏜
2   3

↑   ↑

1   1
⏝   ⏝
4-*O*-Me-α-D-Glc*p*U   α-L-Ara*f*
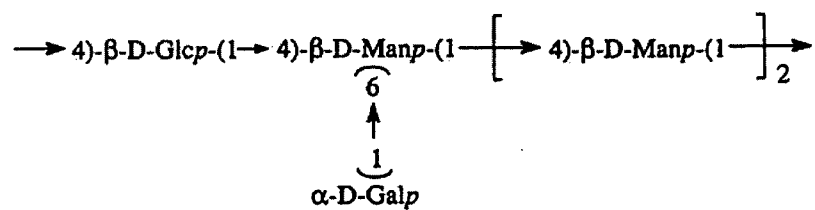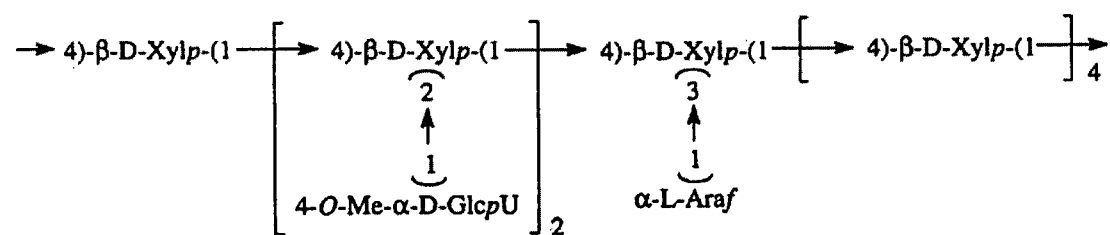
Arabinoglucuronoxylan

Figure 3-16. Schematic representation of the two major hemicelluloses in softwoods (Stenius, 2000).

of (1→4) linked β-D-glucopyranose (β-D-Glcp) and β-D-mannopyranose (β-D-Manp) units (Figure 3-16). The other constituent, xylan (5-10%) is made up of a linear framework of (1→4) linked β-D-xylopyranose (β-D-Xylp) with branches of both (1→2) linked pyranoid 4-O-methyl-α-D-glucuronic acid (4-O-Me-α-D-GlcpU) and (1→3) linked α-L-arabinofuranose (α-L-Araf) (Figure 3-16). Softwoods generally contain fewer hemicelluloses than hardwoods where mannose is the most common hemicellulose constituent. During chemical pulping, the amount, location and structure of the various hemicelluloses change dramatically. As hemicelluloses are more easily degraded and dissolved than cellulose, the hemicellulose percentage is always less in pulp than in the original wood. Hemicellulose in wood serves as a structural function whereby it coats and binds the cellulose microfibrils into a common matrix. In addition, hemicelluloses are significantly more soluble than cellulose (shorter chain lengths and branching), and therefore is the primary reason that these polymers are among the first cell wall components to be attacked by decay fungi (Stenius, 2000; Zabel and Morrell, 1992; Smook, 1992).

The carbohydrate analysis indicated that sound sapwood had higher average total carbohydrate content (66.6%) than infested (62.5%) sapwood (Figure 3-17). Earlier studies from McGovern (1951) and Lieu et al. (1979) also demonstrated that holocellulose content in sapwood (77.18%) of green lodgepole pine wood had slightly higher carbohydrate content than infested wood (75.11%). The results indicated that a difference in carbohydrate content between sound and infested sapwood is likely due to the consumption of low molecular, soluble carbohydrates by microorganisms in dead wood. Fungi typically penetrate, invade, externally digest and absorb soluble constituents when invading wood (Zabel and Morrell, 1992).

Figure 3-17. Total carbohydrate content in sound and infested lodgepole pine sapwood at different tree heights for a total of 30 samples. Error bars indicate 95% confidence interval.

Figure 3-18. Total carbohydrate content in sound and infested lodgepole pine heartwood at different tree heights for a total of 30 samples. Error bars indicate 95% confidence interval.

Total carbohydrate content in the sound sapwood was also found to increase with tree height, as there is a statistically significant difference when comparing carbohydrate content at diameter at breast height (1.5m) to midbole height (5.5m) and crown wood (9.5m). This result is attributed to the fact that more photosynthesis occurs higher up the tree, under the influence of the live crown, and thereby produces more sugars (Koch, 1996). However, carbohydrate content of the infested sapwood decreased with increasing tree height. This latter phenomenon is likely due to the fact that the tree is more prone to attack by mountain pine beetles near the midbole of the tree as mountain pine beetles have been reported to favour flying at a level corresponding to the midbole of lodgepole pine (Section 1.5.4) (Amman and Walter, 1983). As mountain pine beetles and associated fungi invade the host, they decompose and consume elements critical to plant photosynthesis (Zabel and Morrell, 1992). As a result, such invaders deplete important food sources, such as carbohydrates. Moreover, the heartwood carbohydrate content analysis did not demonstrate a statistical difference between sound and infested wood, nor did it reveal a difference at various tree heights (Figure 3-18).

A more specific evaluation of carbohydrates indicated that the infested sapwood had a significant decrease in hemicellulose-derived sugars for all tree heights (Figure 3-19). This result is due to the fact that hemicelluose sugars are soluble, and the first material to be consumed by fungi during incipient growth on lignocellulosic material (Higuchi, 1985; Zabel and Morrell, 1992). Table 3-3 and Table 3-4 demonstrate that aside from glucose, mannose is the dominant constituent of hemicellulose, followed by xylose, galactose and then arabinose. The infested sapwood had lower content of all hemicellulose sugars (with the exception of glucose); however, mannose, xylose and arabinose appeared to the most readily degraded sugars (Table 3-3). No significant difference with regards to glucose and hemicellulose content with tree height was apparent. The analysis of carbohydrates in both sound and infested heartwood
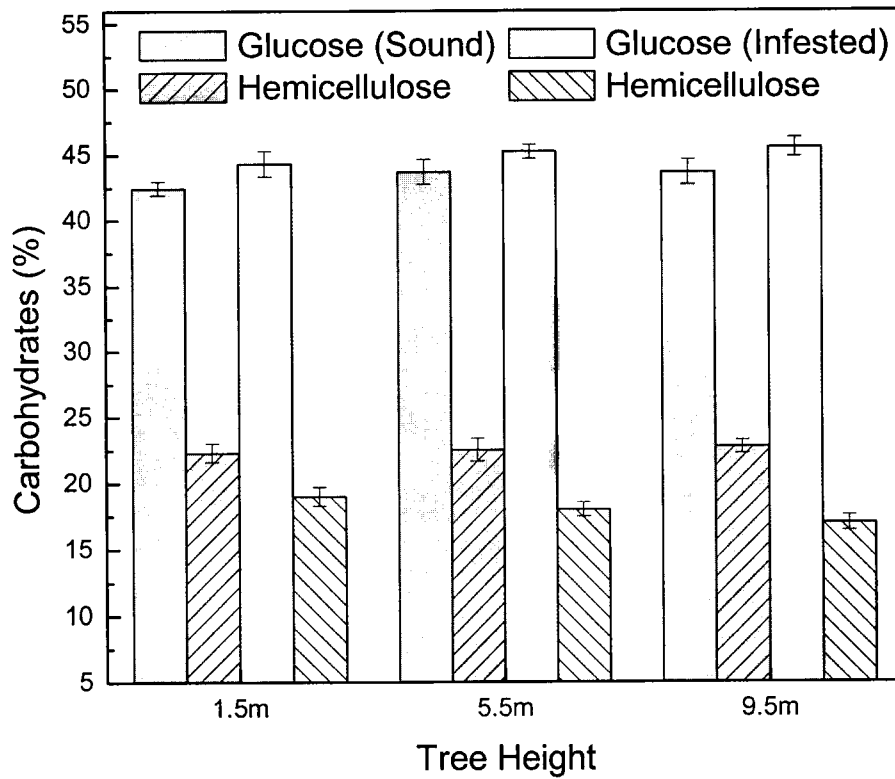
Figure 3-19. Glucose and hemicellulose content in sound and infested lodgepole pine sapwood at different tree heights for a total of 30 samples. Error bars indicate 95% confidence interval.
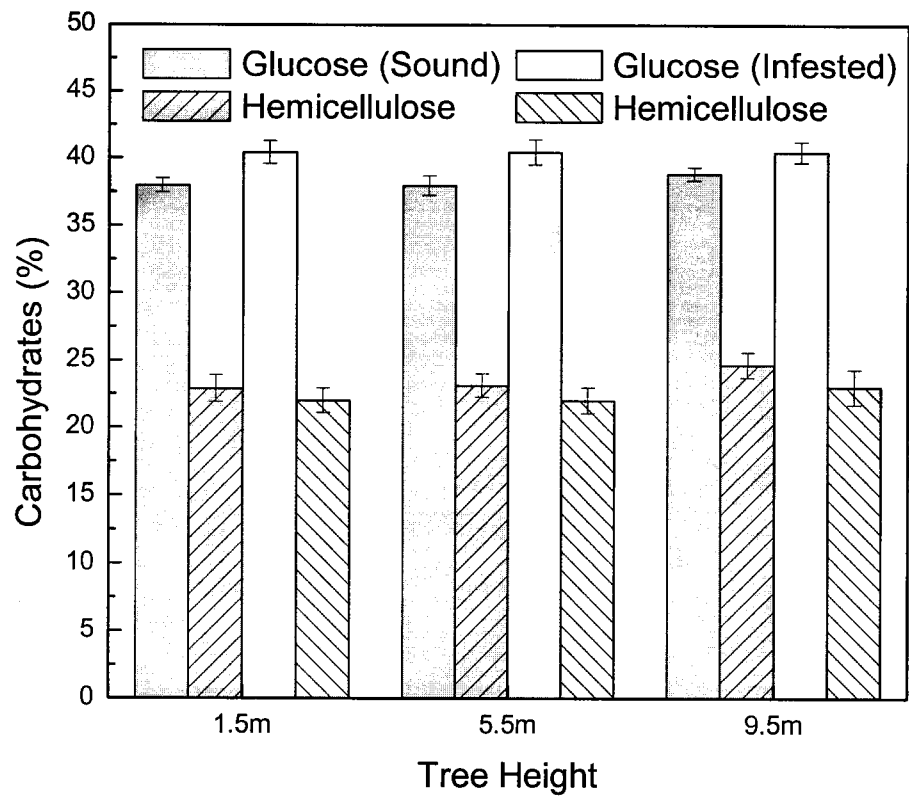
Figure 3-20. Glucose and hemicellulose content in sound and infested lodgepole pine heartwood at different tree heights for a total of 30 samples. Error bars indicate 95% confidence interval.

indicated that there was no significant difference in hemicellulose sugar content between trees at different tree heights (Figure 3-20 and Table 3-4). However, in both infested heartwood and sapwood, there is significantly more glucose content (by weight) compared to sound wood (Figure 3-19 and 3-20), which is likely related to the removal of other cell wall constituents during fungal infestation. The results indicate that infested sapwood contains less carbohydrate, which implies that the pulp yield will be lowered during the pulping process.

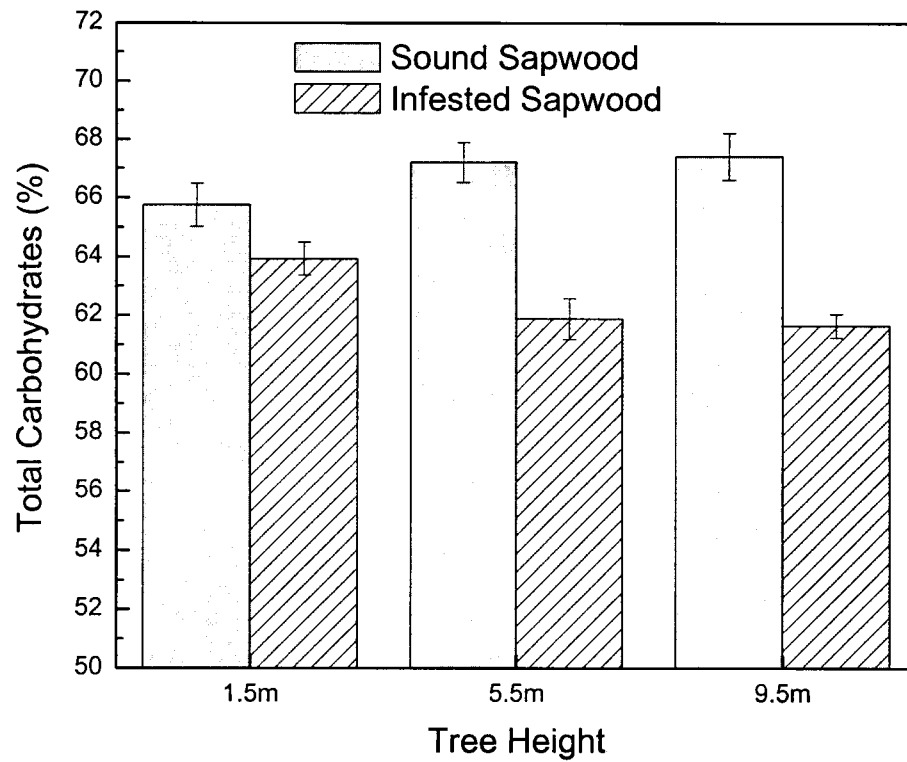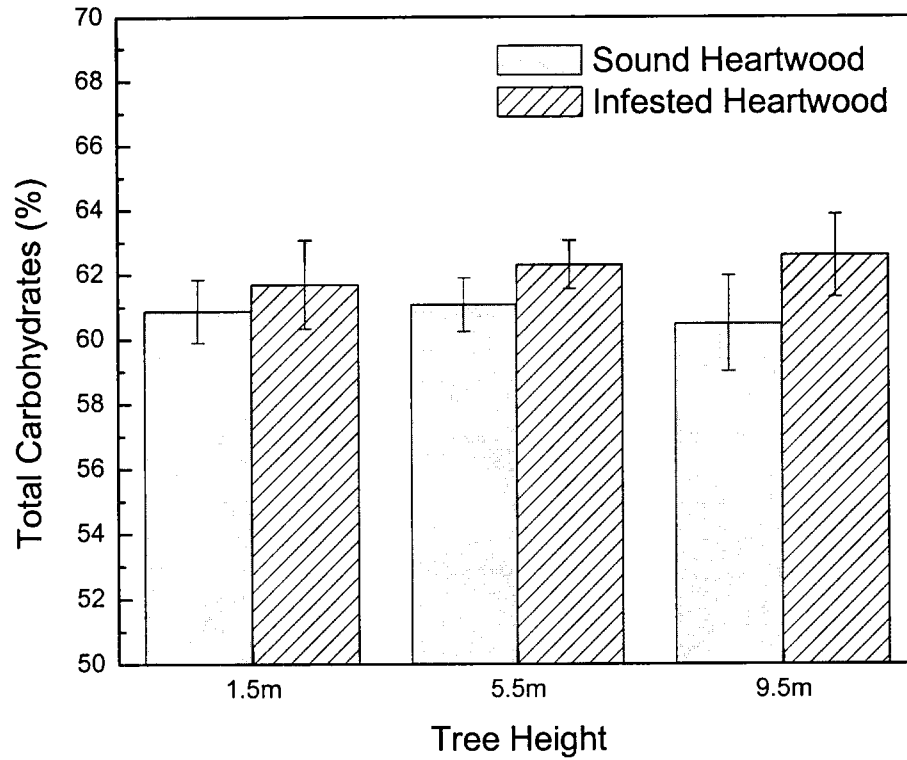**Table 3-3**. Carbohydrate composition of sound and infested lodgepole pine sapwood at different tree heights for a total of 30 samples.

Tree Height (Sound)	Arabinose (%)	Galactose (%)	Xylose (%)	Mannose (%)	Glucose (%)
1.5m	2.04 (0.1)	2.14 (0.2)	5.62 (0.4)	11.51 (0.2)	42.51 (0.5)
5.5m	2.13 (0.2)	2.23 (0.2)	5.98 (0.4)	11.44 (0.5)	42.72 (0.4)
9.5m	2.11 (0.1)	2.34 (0.3)	6.13 (0.3)	11.32 (0.3)	42.76 (0.4)
(Infested)	Arabinose (%)	Galactose (%)	Xylose (%)	Mannose (%)	Glucose (%)
1.5m	1.46 (0.5)	2.01 (0.3)	5.27 (0.3)	10.94 (0.4)	44.22 (0.3)
5.5m	1.45 (0.5)	2.00 (0.6)	5.30 (0.2)	10.35 (0.1)	44.53 (0.4)
9.5m	1.45 (0.4)	2.07 (0.4)	5.34 (0.2)	10.21 (0.2)	44.70 (0.3)

Table 3-4. Carbohydrate composition for sound and infested lodgepole pine heartwood at different tree heights for a total of 30 samples.

Tree Height (Sound)	Arabinose (%)	Galactose (%)	Xylose (%)	Mannose (%)	Glucose (%)
1.5m	1.99 (0.2)	4.54 (0.6)	6.01 (0.5)	10.40 (0.2)	38.05 (0.4)
5.5m	2.01 (0.2)	3.72 (0.3)	5.56 (0.6)	10.62 (0.1)	38.12 (0.3)
9.5m	2.08 (0.4)	4.88 (0.5)	6.38 (0.3)	10.91 (0.4)	38.84 (0.5)
(Infested)	Arabinose (%)	Galactose (%)	Xylose (%)	Mannose (%)	Glucose (%)
1.5m	1.98 (0.1)	3.72 (0.5)	5.98 (0.2)	10.19 (0.3)	40.40 (0.4)
5.5m	1.94 (0.2)	4.14 (0.1)	5.95 (0.4)	10.60 (0.1)	40.47 (0.3)
9.5m	1.98 (0.3)	3.78 (0.3)	5.91 (0.4)	10.74 (0.2)	40.44 (0.4)

3.7 Longitudinal Specific Permeability

The permeability of wood is a measure of the efficiency of fluid transport through a porous solid under the influence of static or dynamic pressure gradient. A solid must be porous to be permeable, but not all porous solids are permeable. A solid is permeable when the void spaces are interconnected by openings; of which softwoods are permeable because the tracheid lumens are connected by pit pairs with openings in the membranes. Pits are gaps in the secondary wall that regulate the conduction of water and a variety of solutes between adjacent cells. If these membrane openings are occluded or encrusted, or if the pits are aspirated, the wood will form a closed-cell structure and permeability may approach zero (Siau, 1984; Bao et al., 1999). Pits are the primary cell wall region initially penetrated by hyphae during colonization of wood and degradation caused by decay and staining fungi (Zabel and Morrell, 1992).

Softwood cells are characterized by secondary walls that are not continuous. The secondary walls are interrupted by regions in the secondary portion of the wall referred to as pits, which

typically occur in pairs. Softwood tracheids possess mainly bordered pits (Figure 3-21) and these pits are characterized by a thickened center called the torus, which is surrounded by a microfibrillar network known as the margo (Figure 3-22). The spaces in the microfibrillar network are large enough to permit liquid flow and passage of small particles between contiguous cells. The pit membrane is flexible, and therefore can shift to one side of the pit cavity, resulting in the blockage of the aperture by the impermeable torus. A pit in this condition is said to be aspirated (Figure 3-23), and results as a function of liquid tension (Haygreen and Bowyer, 1996; Zabel and Morrell, 1992). Wood with many aspirated pits is difficult to season or treat chemically.

Fluid transport through wood is segregated into two main classifications. The first classification involves the bulk flow of fluids through the interconnected openings of the wood structure under static or capillary pressure gradients. The second is diffusion, which is divided into two types: intergas diffusion, which involves water vapor transfer through the air in the lumens of the cells, and bound-water diffusion, which occurs within the cell walls of wood. In wood utilization, permeability plays an important role since it directly affects processing characteristics such as drying and treatment with preservatives as well as the penetration of kraft cooking chemicals to lignin active sites within the cell wall (Siau, 1984).
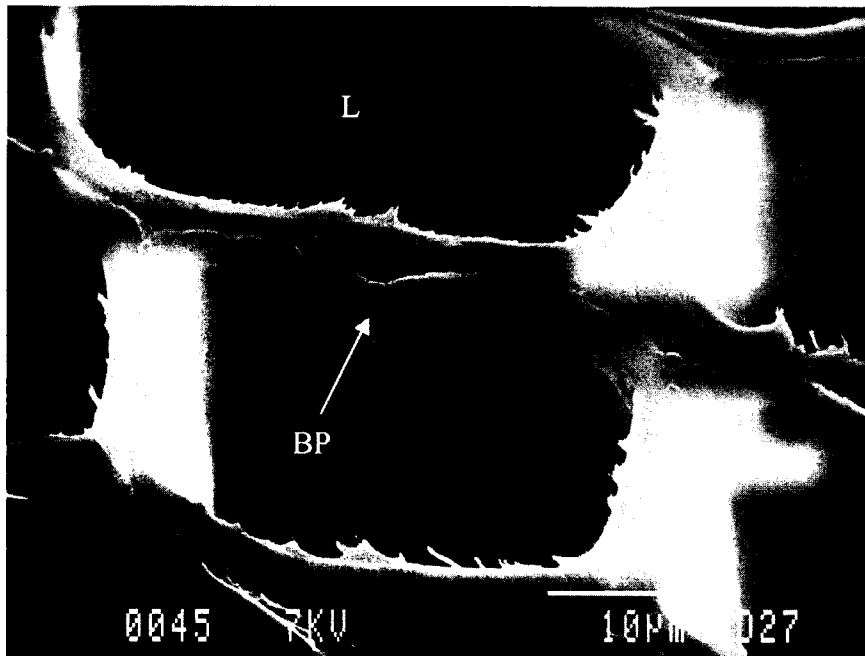
Figure 3-21. Scanning electron micrograph of a transverse section of infested lodgepole sapwood at midbole height showing a bordered pit (BP) and cell lumens (L) (2000× magnification)
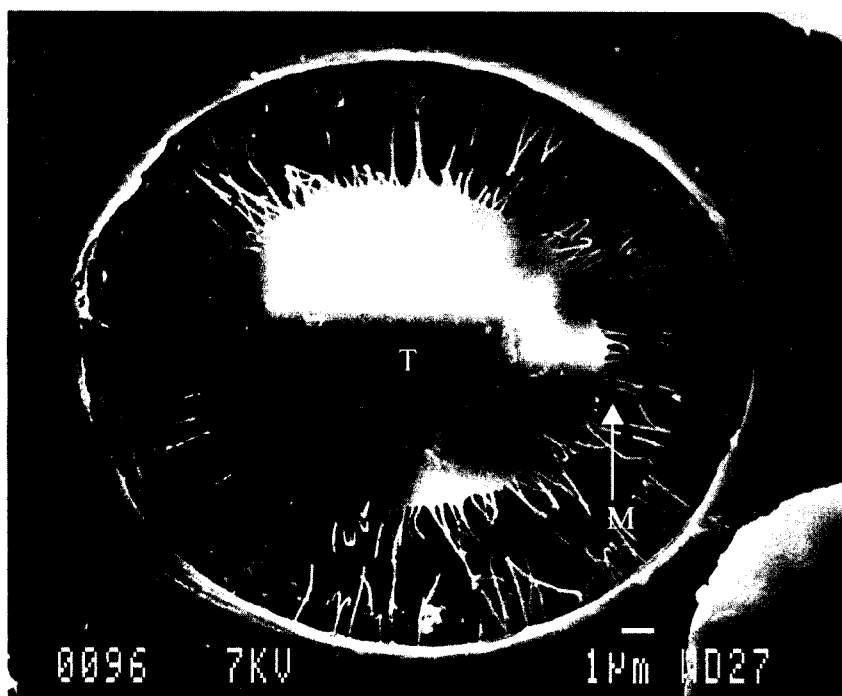
Figure 3-22. Scanning electron micrograph of a radial view of sound lodgepole pine sapwood at breast height showing the torus (T) and margo (M) of an unaspirated pit (4500× magnification).
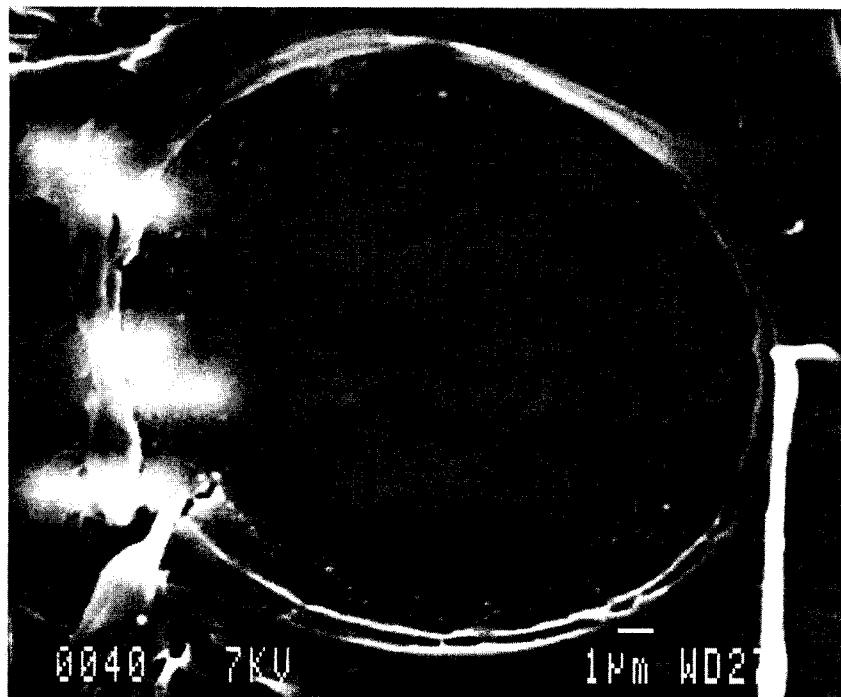
Figure 3-23. Scanning electron micrograph of a radial view of infested lodgepole pine heartwood at breast height showing an aspirated pit (5000× magnification).

Wiedenbeck *et al.* (1990) and Bao *et al.* (1999) both reported that the difference in permeability between heartwood and sapwood is due to more aspirated pits in the heartwood. As heartwood possesses a higher extractive content, it is more likely that pit membranes can also be encrusted with extractive material (Bao *et al.*, 1999). As expected, the results from our studies showed that sapwood was significantly more permeable than heartwood. The results further demonstrated that infested sapwood was significantly more permeable than sound sapwood, while sound heartwood was more permeable than infested heartwood (Figure 3-24 and Figure 3-25). As a consequence of these results, microscopic analyses were performed to determine possible reasons for the occurrence of such differences in sound and infested wood. The microscopic analyses showed the presence of fungal hyphae in infested sapwood (Figure 3-26). Fungi are filamentous eukaryotes without chlorophyll that digest various carbon compounds externally (Zabel and Morrell, 1992). In addition, more fungal hyphae were observed higher up the tree in the midbole and crown wood. Therefore, infested sapwood may be more permeable as a result of fungal infection as the fungi penetrate and proliferate within the pit membranes and primary cell walls of ray parenchyma cells. Majority of the decay fungi initially manoeuver through the wood mainly by direct pit penetration, and with the removal of the pit membrane, the wood becomes more receptive to the movement of fluids. Due the changes caused by fungi pit penetration, decayed wood absorbs and desorbs liquids more readily than sound wood (Zabel and Morrell, 1992). In addition, trabeculae were also present in the tracheids of the infested sapwood; these structures are rod-like extensions of cell wall material that occasionally transverse the lumens of wood cells from one tangential wall to another (Figure 3-27). Trabeculae have been thought to form as a result of tree wounding and cambial exposure to fungal attack (Butterfield and Meylan, 1979).
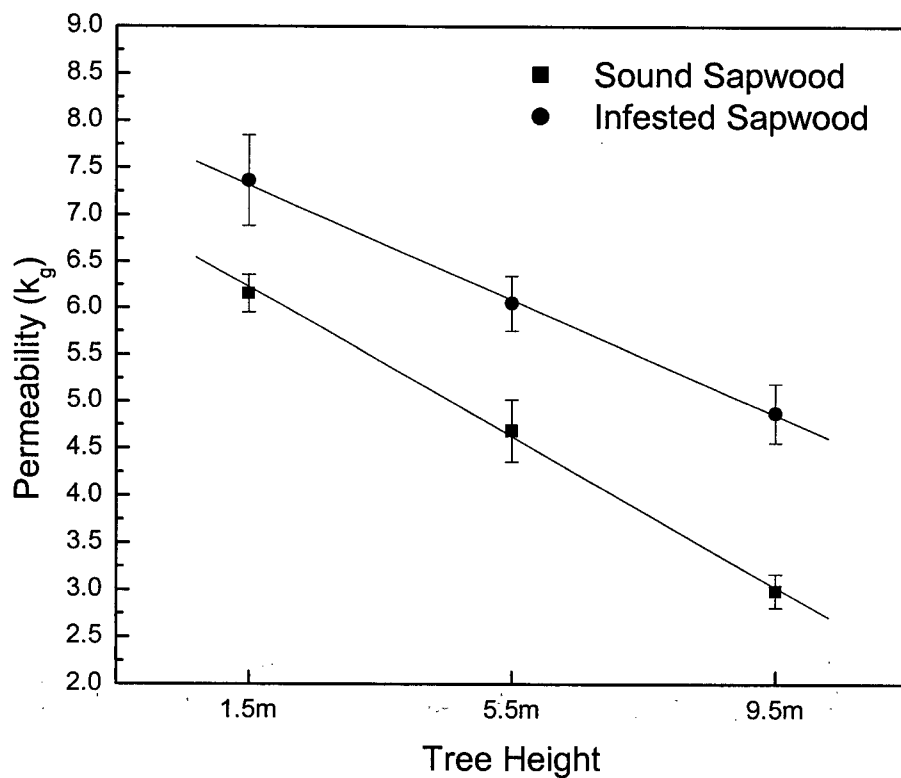
Figure 3-24. Longitudinal specific permeability of sound and infested lodgepole pine sapwood at different tree heights for a total of 18 samples. Error bars indicate 95% confidence interval.
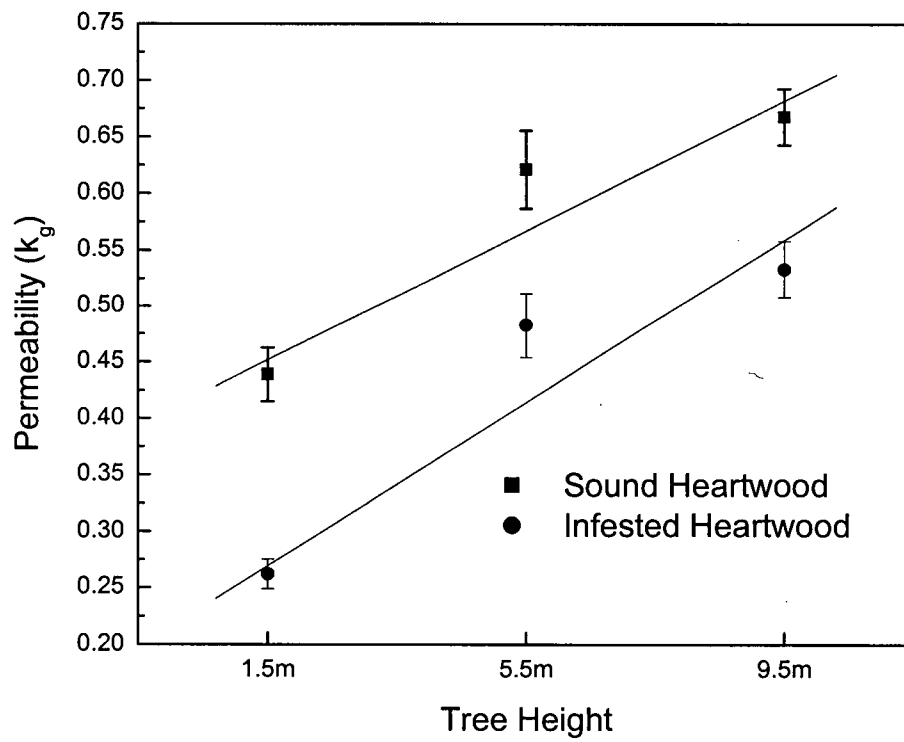
Figure 3-25. Longitudinal specific permeability of sound and infested lodgepole pine heartwood at different tree heights for a total of 18 samples. Error bars indicate 95% confidence interval.

Figure 3-26. Scanning electron micrograph of fungal hyphae in infested lodgepole pine sapwood at midbole height (1800× magnification).
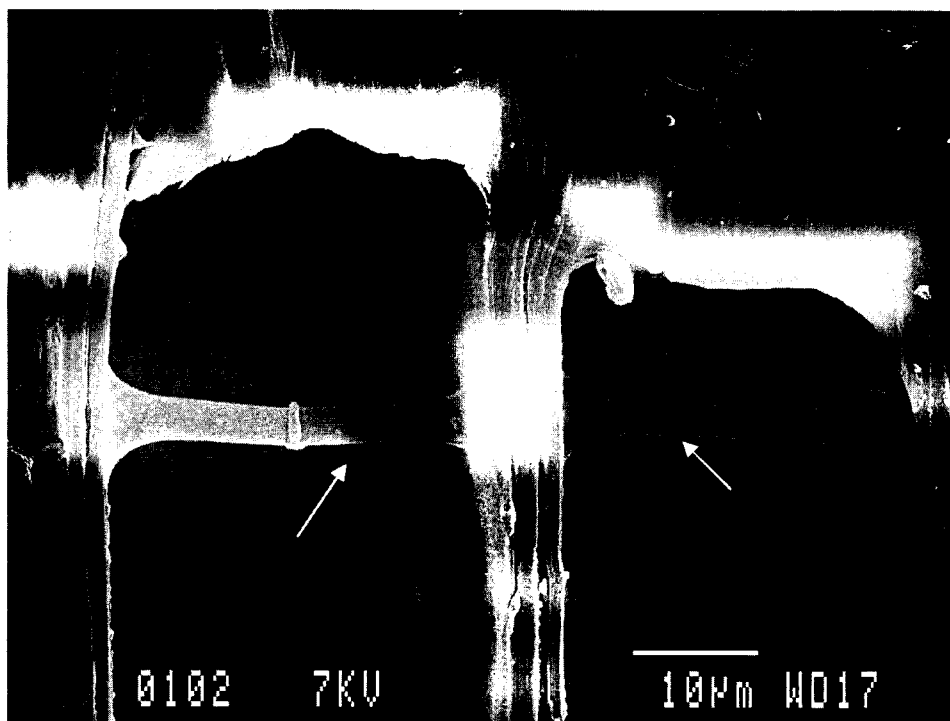
Figure 3-27. Scanning electron micrograph of trabeculae in infested lodgepole pine sapwood at midbole height (2400× magnification).
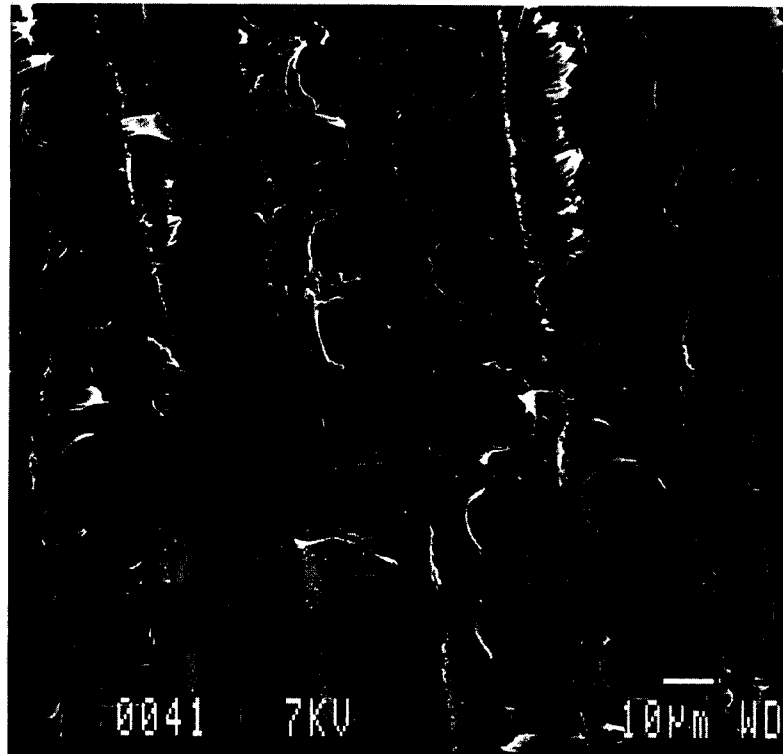
Figure 3-28. Scanning electron micrograph of aspirated pits in infested lodgepole pine heartwood at midbole height (2200× magnification).

Additionally, the heartwood contained an abundant number of aspirated pits, and therefore reduced permeability (Figure 3-28). In the infested heartwood, unaspirated pits were undetectable and this is likely due to the removal of water from the onset of infestation. Unlike the trend in sapwood, the permeability of sound heartwood was significantly higher than infested heartwood possibly because fungal penetration was in absence.

Furthermore, the effect of tree height on permeability showed that for both the sound and infested heartwood, permeability tended to increase with tree height. This increase in permeability with tree height is likely due to the decreased concentration of extractives towards the crown (Section 3.4). Koch (1996), Flynn (1995) and Rice and D'Onofrio (1996) suggested that differences in permeability are generally due to differences in aspiration and the amount of extractive content. Resin deposition can vary substantially within the tree and extractives in wood are known to impede flow through the cells and decrease permeability (Flynn, 1995; Rice and D'Onofrio, 1996). In addition, Koch (1996) suggested that the tree height at which heartwood no longer occurs varies from 7m to 14m, and is comprised of mostly juvenile wood and earlywood cells. The concentration of juvenile wood increases with tree height, and is distinguished by thinner cell walls and lower latewood content, which is known to possess fewer and smaller bordered pits compared to earlywood (Jozsa and Middleton, 1994). Since permeability is largely related to the number of pit membrane openings and is limited by the amount of latewood (Koch, 1996; Bao et al., 1999), the increase in permeability in heartwood towards the upper bole can also be explained by the increased presence of earlywood and juvenile wood and the decrease in latewood content. Conversely, the sapwood permeability results indicated that permeability tended to decrease towards the upper bole, which is likely related to the increased concentration of extractives (Section 3.4) towards the crown. Extractive content and permeability were also found to be closely correlated for both sound and infested sapwood and heartwood (Figure 3-29 and 3-30). Vologdin et al., (1979) also noticed that
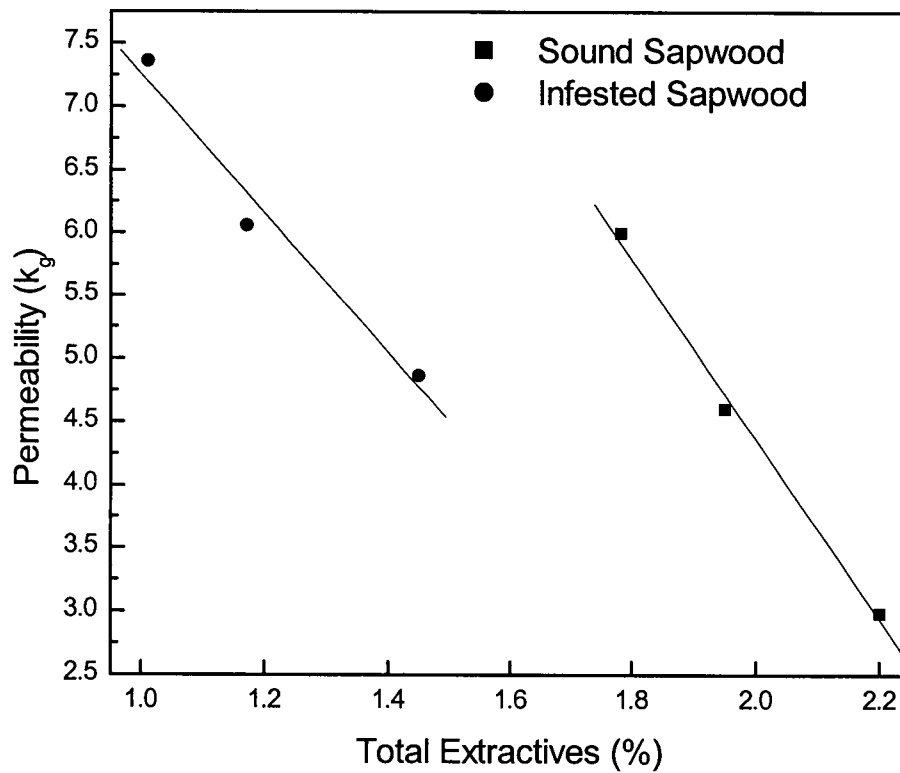
Figure 3-29. Longitudinal specific permeability versus total extractives content for sound and infested lodgepole pine sapwood.
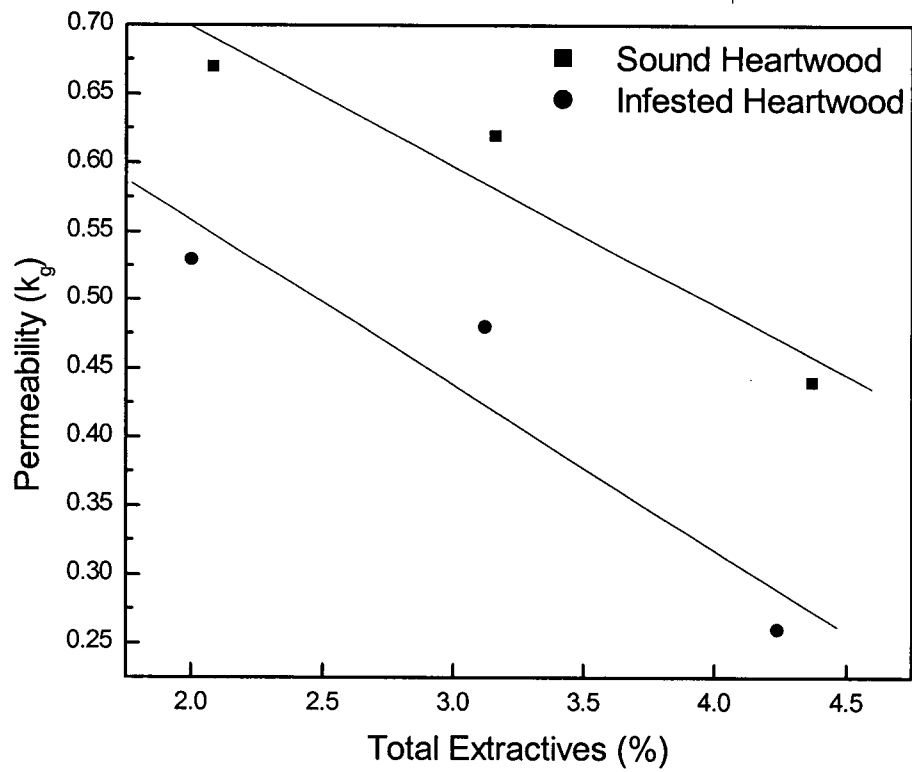
Figure 3-30. Longitudinal specific permeability versus total extractives content for sound and infested lodgepole pine heartwood.

permeability increased progressively with the removal of extractives (phenolics). Furthermore, Rice and D'Onofrio (1996) reported that there are variations in permeability with tree height; however, they concluded that there is no consistent trend in terms of tree height and permeability. Moreover, Booker (1977) suggested that gas permeability with relation to tree height effect has not been adequately investigated, and further research is needed.

The results imply that kraft liquor penetration for heartwood, and particularly the infested heartwood, will be more difficult than sapwood as the permeability of heartwood is lower. Therefore, more kraft pulping liquor will be required to pulp infested heartwood. However, since permeability of heartwood increases with tree height, the crown wood will have better liquor penetration than wood at breast height. Infested sapwood will be easier to pulp as the permeability is higher than sound sapwood, however, liquor penetrability in the crown wood will be lower than at breast height.

CHAPTER 4. THE EFFECT OF MOUNTAIN PINE BEETLE ATTACK ON PULP AND PAPER QUALITY

4.1 Introduction

As the catastrophic loss of lodgepole pine due to mountain pine beetle infestation rapidly increases, the ratio of pine traditionally harvested and entering the manufacturing sector will continue to increase. In effect, the abundant volume of infested lodgepole pine wood may constitute a predominant source of cellulosic fibres for pulp and paper manufacture for the foreseeable future in BC. It is well known that the use of dead wood is detrimental to chipping, pulp processing and paper end-product quality. One of the goals of this research is to ascertain how the use of infested lodgepole pine affects pulp and paper manufacture relative to sound lodgepole pine

4.2 Chip Quality

The quality of chips used for pulping is essential to the operation of the pulp mill and in the final pulp quality. As a result, it is critical to understand which variables affect chip quality. Chip quality variables generally include chip density (and consequently packing factor), chip size distribution. In kraft pulping, chip thickness is the most important factor determining chip quality (Mimms *et al.*, 1993; Koch, 1996), as this variable limits the penetration of cooking liquor into chips at approximately the same rate from all directions. If a chip is too thick, the cooking liquor will not have time to fully penetrate the center of the chip during cooking, and results in uncooked centers, which results in shives (uncooked wood) called rejects. Oversize chips can also cause non-uniform chip packing in the digester, which causes non-uniform liquor circulation and uneven cooking. Similarly, undersize chips can also cause several problems; pin chips (fines and sawdust), which are chips that have approximately the same width and thickness, can cause liquor circulation problems and digester plugging. Undersize chips will

normally be overcooked when digested with regular size chips. Therefore, pulp yield and pulp strength are significantly affected (Mimms et al., 1993).

A narrow size distribution and uniform chip thickness are very important to ensure uniform liquor penetration and steady cooking conditions. Typically the ideal chip dimensions or accept chips are a length of 10mm to 30mm and a thickness of 2mm to 6mm (Mimms et al., 1993; Hatton and Gee, 1994). Therefore, chip size distribution was evaluated in this research using a chip classifier. The chip screening system used in our investigation separated chips on the basis of chip thickness. Chip screening helps to remove oversize and undersize chips so that a narrow size distribution is generated that satisfies the mill's chip quality target. Moreover, chip screening also helps to remove some of the small size contaminants such as bark, sand and unwanted material (Smook, 1992; Mimms et al., 1993).

4.2.1 Chip Thickness and Chip Size Distribution

Chip screening analysis, indicated that the overall chip quality was generally comparable for both sound and infested wood, with the sound sapwood demonstrating an average of roughly 87% accept chips and the infested sapwood producing approximately 86% (Table 4-1). The heartwood had a slightly lower percentage of accept chips compared with the sapwood. Nonetheless, chip quality was relatively good such that sound heartwood had an average of 84% and infested heartwood had an average of 83% of the chips falling in the accept category (Table 4-2). However, the chipping of infested sapwood (average of 0.9%) and heartwood (an average of 0.8%), in general, produced a slightly larger percentage of fines than the chipping of sound sapwood (an average of 0.5%) and heartwood (an average of 0.4%). This is likely a result of the

Table 4-1. Lodgepole pine sapwood chip size distribution by tree height for a total of 24 samples.

Tree Height	% Accepts (2-6mm)		% Fines	
	Sound	Infested	Sound	Infested
2m	89.4 (0.8)	88.5 (0.1)	0.4 (0.01)	0.9 (0.03)
4m	84.7 (0.1)	86.7 (0.6)	0.5 (0.05)	1.0 (0.10)
6m	87.1 (1.0)	84.7 (0.9)	0.5 (0.20)	0.9 (0.10)
8m	88.6 (0.3)	86.8 (0.2)	0.5 (0.01)	0.9 (0.06)
10m	84.4 (0.7)	87.2 (0.4)	0.4 (0.04)	1.0 (0.02)
12m	88.1 (0.3)	87.5 (0.8)	0.5 (0.03)	0.9 (0.06)

* Range shown in parentheses.

Table 4-2. Lodgepole pine heartwood chip size distribution by tree height for a total of 24 samples.

Tree Height	% Accepts (2-6mm)		% Fines	
	Sound	Infested	Sound	Infested
2m	79.6 (1.0)	79.4 (0.4)	0.4 (0.02)	0.8 (0.07)
4m	83.0 (0.2)	83.5 (0.3)	0.3 (0.02)	0.8 (0.03)
6m	82.7 (0.9)	81.8 (0.9)	0.4 (0.05)	0.8 (0.10)
8m	84.1 (0.9)	83.7 (0.5)	0.4 (0.03)	0.9 (0.01)
10m	85.2 (0.4)	83.6 (0.7)	0.5 (0.05)	0.8 (0.02)
12m	87.3 (0.3)	83.7 (0.2)	0.6 (0.03)	0.9 (0.04)

* Range shown in parentheses.

lower moisture content in infested wood, which will have a pronounced effect on the way in which chips are generated, resulting in a higher percentage of fines. Lowery *et al.* (1977) observed similar results whereby sound and infested lodgepole pine produced a good percentage of accept chips, however, infested wood generated more fines. Lowery *et al.* (1977) attributed their results to the fact that infested wood had exceptionally lower moisture content, and as a result of the dryness of the wood, not only was the percentage of fines affected but also more power was required to chip the dead wood. Our results concur with these findings and clearly demonstrate that there was no apparent trend in the effect of tree height on chip size distribution.

4.3 Pulp Quality

Within the mountain pine beetle affected region in BC, kraft pulping accounts for 2,695,900 tonnes/year. In addition, BC softwood fibres remain the benchmark, as they are the strongest (tensile strength) fibres and the pulp produced from our managed natural forests are regarded as the gold standard worldwide. Generally, the pulping wood chips are a mixture of spruce, pine and fir (SPF), where pine has the coarses fibres and typically is inferior in tensile strength to other species. Chemical pulping dominates 70% of the total worldwide production, and currently about 80% of chemical pulps are produced by the kraft (sulfate) process (Stenius, 2000). The objective of kraft pulping is to chemically separate wood fibres and dissolve most of the lignin found in the fibre walls. Fibre separation is accomplished when the lignin in the middle lamella that binds the fibres together is dissolved. The chemicals in the cooking liquor also penetrate the cell walls and dissolve the lignin (Stenius, 2000; Mimms et al., 1993).

The cooking liquor, also known as white liquor, is an aqueous solution of sodium hydroxide (NaOH) and sodium sulfide (Na_2S) (Smook, 1992). The active components in the cooking liquor are the hydroxyl ion (HO^-) and the hydrosulfide ion (HS^-), which originate from NaOH and Na_2S. NaOH and Na_2S make up the active alkali (AA) of the cooking liquor as shown in the following:

$$NaOH \rightarrow Na^+ + OH^-$$

$$Na_2S \rightarrow 2Na^+ + S^{2-}$$

$$S^{2-} + H_2O \rightarrow HS^- + HO^-$$
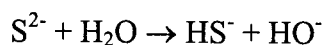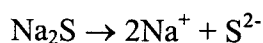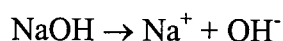
The concentration and total charge of the HS^- and HO^- ions are the key elements in all reactions; both in lignin dissolution and in unfavourable reactions such as cellulose degradation. The total HO^- ions present from the original caustic and part of the original sulfide is called the effective alkali (EA) as shown in the following (Mimms et al., 1993):

Effective Alkali = NaOH + ½ Na$_2$S

Sulphidity = Na$_2$S/NaOH + Na$_2$S

Total alkali = NaOH + Na$_2$S + Na$_2$CO$_3$ + Na$_2$SO$_4$

In addition, maintaining an alkaline environment throughout the kraft cooking process is important as it keeps the lignin in solution and protects the carbohydrates from peeling (degradation of carbohydrate polymers one monosaccharide at a time) which is important because this will preserve the pulp strength (Stenius, 2000).

The most important variable in kraft pulping is time and temperature. The time and temperature of the cooking cycle is usually represented by a numerical value called H-factor (Mimms *et al.*, 1993). Typically, the goal in kraft cooking is to obtain as low a kappa number as possible and minimize the impact on product quality (Stenius, 2000).

The reactions that occur during kraft pulping are complex and are not fully understood. However, it is known that the presence of hydrogen sulfide ions accelerates lignin dissolution without increasing the dissolution of cellulose (Mimms *et al.*, 1993). Hydrogen sulfide ions primarily react with lignin, whereas carbohydrate reactions are only affected by alkalinity (HO$^-$ ions). The overall effect of the reactions between lignin, hydrogen sulfide, and hydroxyl ions is that the lignin polymer is broken down into smaller molecules, which are dissolved in the cooking liquor and liberated from the wood fibers. After cooking, the cooking liquor (black liquor) is separated by washing from the pulp. During kraft pulping, more than 20% of the wood substance is degraded and dissolved. The remaining chemicals in the black liquor are made up of degradation products of lignin, polysaccharides, and wood extractives. Both cellulose and hemicellulose react with hydroxyl ions during cooking and these reactions are unfavourable, because the degradation of carbohydrates lowers pulp yield and strength. Hemicellulose degrades more quickly and in greater quantities than cellulose (Smook, 1992; Mimms *et al.*, 1993; Stenius, 2000). Cellulose is highly crystalline in nature, has a high degree of

polymerization and is therefore, more difficult to degrade. Stenius (2000) reported that approximately 90% of the lignin, 60% of the hemicelluloses, and 15% of the cellulose is dissolved during a traditional kraft cook.

4.3.1 Kappa Number

Kappa number is basically the residual lignin remaining in pulp. Based on kappa number determination, the results indicated that infested sapwood and heartwood has less residual lignin (lower kappa number) at any given H-factor compared to sound sapwood and heartwood (Figure 4-1 and Figure 4-2). For example at a H-factor of 1200 and at the tree level corresponding to breast height, the sound sapwood had a kappa value of 32 whereas the infested sapwood had a kappa of 29, which is a 3 kappa number difference. This result correlates with the klason lignin analysis, which showed that infested sapwood had a lower lignin content. Additionally, since pulping is a function of liquid penetrability (permeability), infested sapwood was shown to be more permeable and this will aid the pulping process, as the lignin will be more readily degraded. McGovern (1951) reported similar results, such that he also observed that infested wood in general produced pulp exhibiting lower kappa numbers when compared to sound. From the klason lignin analysis, it was also shown that infested sapwood and heartwood contained more acid soluble lignin, which may contribute to the reduced kappa number. However, the heartwood was typically shown to have more residual lignin than the sapwood. This result was expected as heartwood contains more juvenile wood, and juvenile wood is known to have higher lignin content than mature wood (Hatton, 1993). No clear trend was observed with regards to tree height on residual lignin. Moreover, for bleachable kraft pulps, typically a target of kappa 30 is desired and Table 4-3 indicates that to attain a kappa value of 30, the sound sapwood and heartwood required a cook with greater severity compared to infested sapwood and heartwood; these results imply that pulp production differences will arise.

Table 4-3. H-factor to kappa 30 values for sound and infested sapwood and heartwood.

Sample	H-factor to kappa 30
Sound Sapwood	1239
Infested Sapwood	1163
Sound Heartwood	1334
Infested Heartwood	1171

4.3.2 Effective Alkali Consumption

Residual black liquor analysis of sound and mountain pine beetle killed lodgepole pine indicated that infested sapwood consumed a lower percentage (an average of 10.5%) of effective alkali (EA) compared to sound wood (an average of 10.9%) (Figure 4-3). These results suggest that infested chips consume less alkali and therefore pulps more rapidly. This is likely due to the dryness of infested wood, which causes varied liquor penetration. This result also correlates with permeability as mentioned in chapter three because infested wood has a higher permeability than sound wood, and therefore, liquor penetrability is better. McGovern (1951) and Scott *et al.* (1996) found similar results such that they reported that infested wood tends to have lower consumption of effective alkali, which causes slightly faster cooking. Scott *et al.* (1996) also noted that the opening of the cell wall structure by fungal growth could increase liquor penetration into the cell wall thereby reducing the chemical consumption and increasing the pulping process.

Figure 4-1. Kappa number versus H-factor for sound and infested lodgepole pine sapwood at different tree heights for a total of 54 samples. Error bars indicate range.
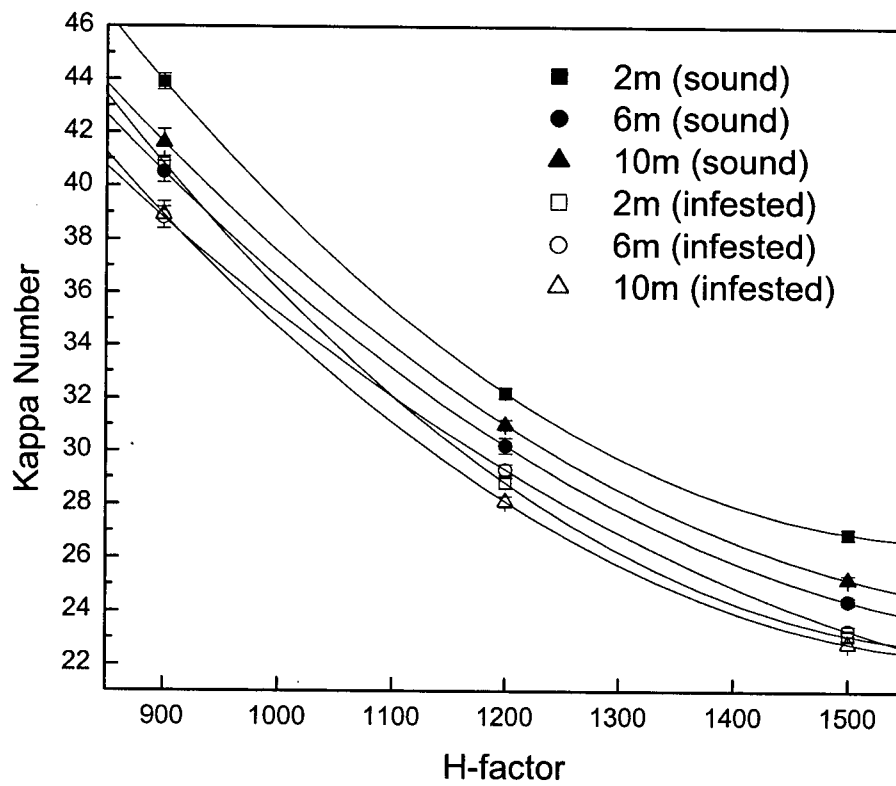
Figure 4-2. Kappa number versus H-factor for sound and infested lodgepole pine heartwood at different tree heights for a total of 54 samples. Error bars indicate range.

In contrast, it is likely that the consumption of effective alkali by infested (an average of 11%) and sound heartwood (an average of 11%) may not show much difference as beetle and fungal infestation is generally confined to the sapwood region (McGovern, 1951; Scott *et al.*, 1996). The heartwood however did consume slightly more effective alkali compared to sapwood (Figure 4-4). This is likely due to the fact that heartwood has significantly lower moisture content than sapwood and more aspirated pits. As a result, it is harder for cooking liquor to penetrate heartwood, and in turn more effective alkali is needed to mitigate lignin desolation. Mimms *et al.* (1993) suggests that when heartwood develops, extractives fill cell voids and impregnate the fibre walls, and therefore liquids do not easily penetrate heartwood. Moreover, no apparent trend was observed in terms of the effect of tree height on effective alkali consumption.

4.3.3 Pulp Yield

Pulp yield is critically important to pulp mills, for example, if a mill produces 1000 tonnes per day and the pulp yield increases by 1%, this implies that the mill will produce an extra 10 tonnes of pulp per day, which translates to 3600 tonnes per year. Likewise, if the yield decreases by 1 % than the pulp mill will have substantial economic losses. Pulp yield determination demonstrated that infested sapwood (48%) and heartwood (46%) have slightly higher yields than sound sapwood (47%) and heartwood (45%) (Figure 4-5 and Figure 4-6). The 1% increase in pulp yield in infested wood may be due to the fact that infested wood actually contains a larger percentage of glucose, on a weight basis, as much of the soluble carbohydrates and extractives have been consumed by beetle and fungal infestation (Section 3.4 and Section 3.6). Unlike infested wood where most of the hemicellulose content is degraded by beetle and fungal attack prior to pulping, the hemicellulose content in sound wood is still intact. Therefore, during pulping, when all the hemicellulose is degraded, the impact on overall yield is more
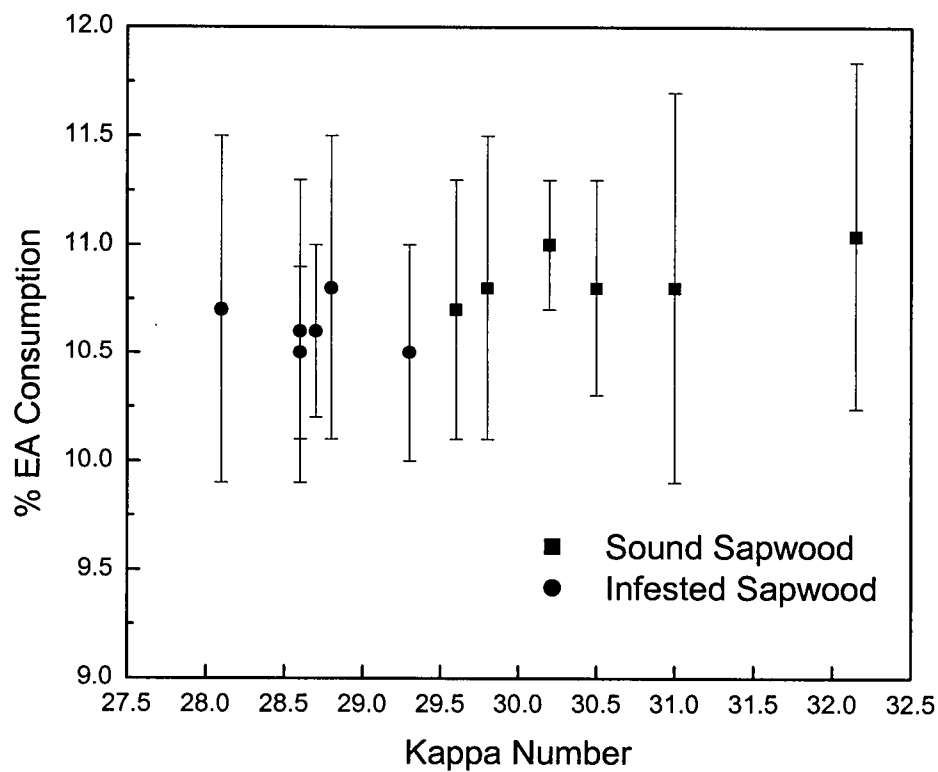
Figure 4-3. Effective alkali consumption (%) versus kappa number at 1200 H-factor for sound and infested lodgepole pine sapwood for a total of 24 samples. Error bars indicate range.

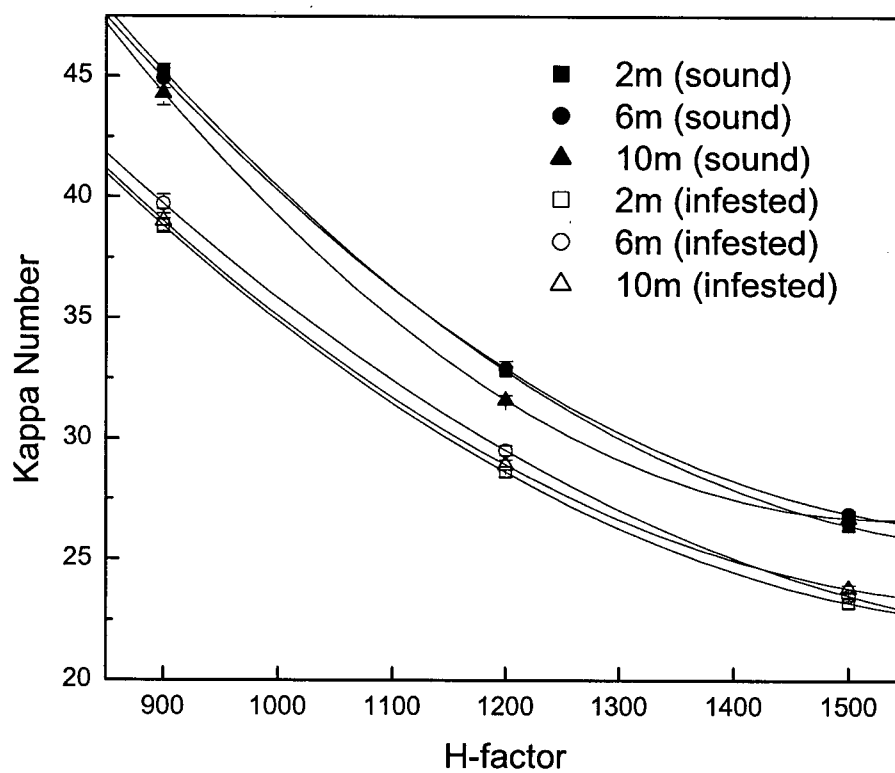**Figure 4-4.** Effective alkali consumption (%) versus kappa number at 1200 H-factor for sound and infested lodgepole pine heartwood for a total of 24 samples. Error bars indicate range.

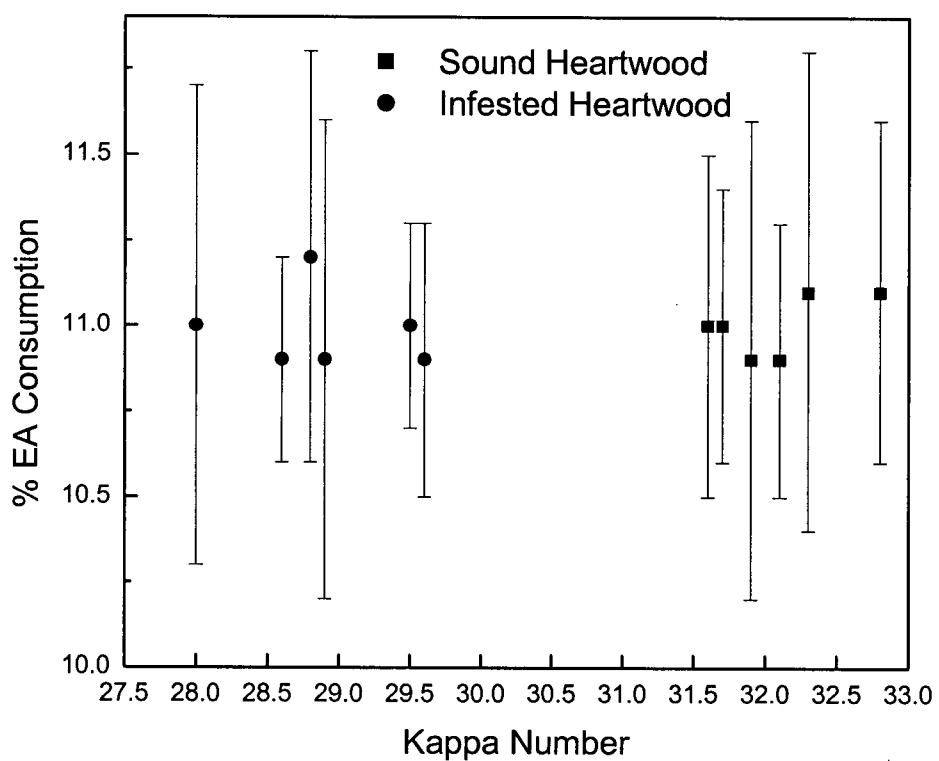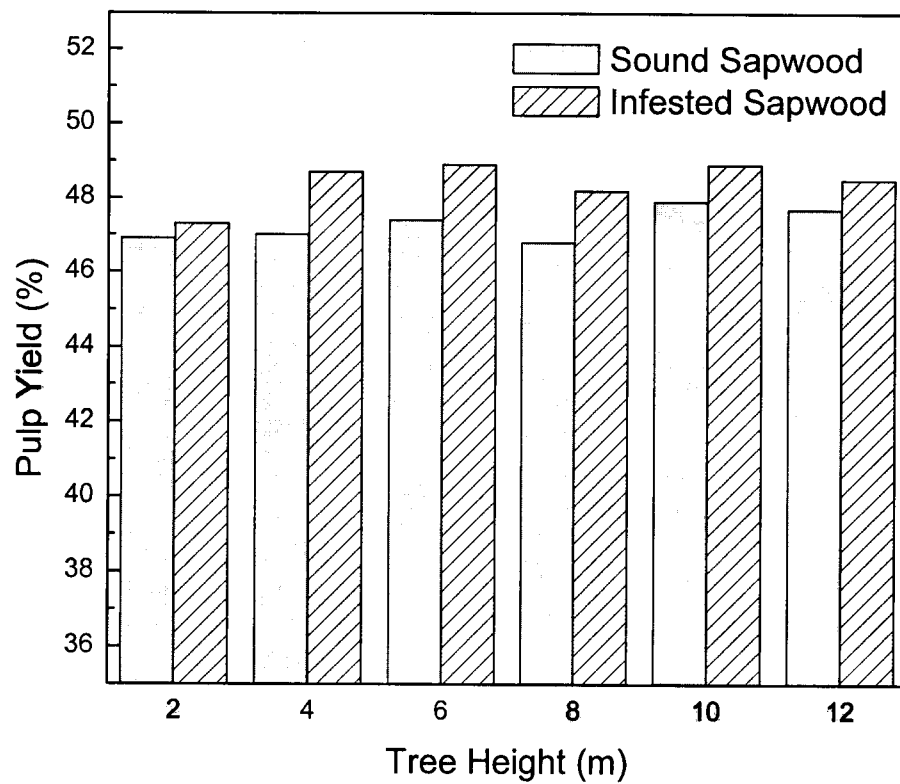**Figure 4-5.** Pulp yield of sound and infested lodgepole pine sapwood at different tree heights for a total of 12 samples.
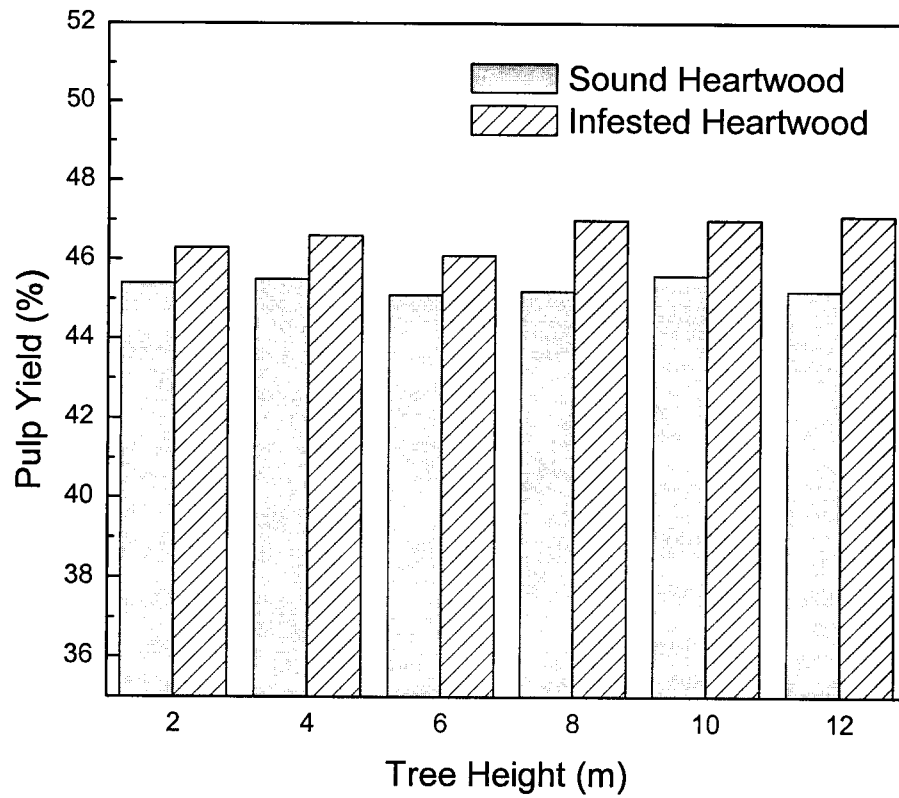
Figure 4-6. Pulp yield of sound and infested lodgepole pine heartwood at different tree heights for a total of 12 samples.

greatly decreased in pulp yield for sound wood. Wood carbohydrate analysis (Section 3.6) generally showed that infested wood contained slightly more glucose content than sound wood. As glucose is more difficult to degrade than hemicellulose, this results in increased pulp yield in infested wood compared to sound wood. Again, there was no apparent trend in terms of the effect of tree height on pulp yield. Moreover, it was apparent from the kappa number analysis that infested sapwood and heartwood required less severe cooking (Section 4.3.1) than sound wood to achieve a target kappa of approximately 30 (Figure 4-7 and 4-8). Thus, with greater severity in cooking the sound wood, more carbohydrates will be degraded causing a decrease in pulp yield when compared to the infested wood.

4.4 Handsheet Quality

In order to investigate the impacts on paper properties between sound wood and mountain pine beetle killed wood, handsheets were prepared and tested. Initially the pulp was beaten in a PFI mill to collapse and fibrillate the fibres and to determine maximum strength properties. Beating changes the fibre characteristics, for example it causes internal fibrillation and external fibrillation, fines formation, fibre cutting and fibre straightening. Alterations in individual fibre properties also correspond to the altered pulp and paper quality. For example, pulp drainage decreases as a result of increased surface area (mainly fines formation) during refining, while internal fibrillations increase fibre swelling and flexibility by loosening the cell wall structure. Similarly, external fibrillation increases the outer surface area of the fibres and therefore increases the potential for overall contact (Levlin and Soderhjelm, 1999).

Paper is formed fibres and fibre fragments bond. The dimensions of pulp fibres depend on the variations in the wood as well as the pulping processes used. Typically, the length of a softwood fibre is 100 times its width (Levlin and Soderhjelm, 1999). The variation in wood fibre dimensions within one species is related to the seasonal variations of earlywood and
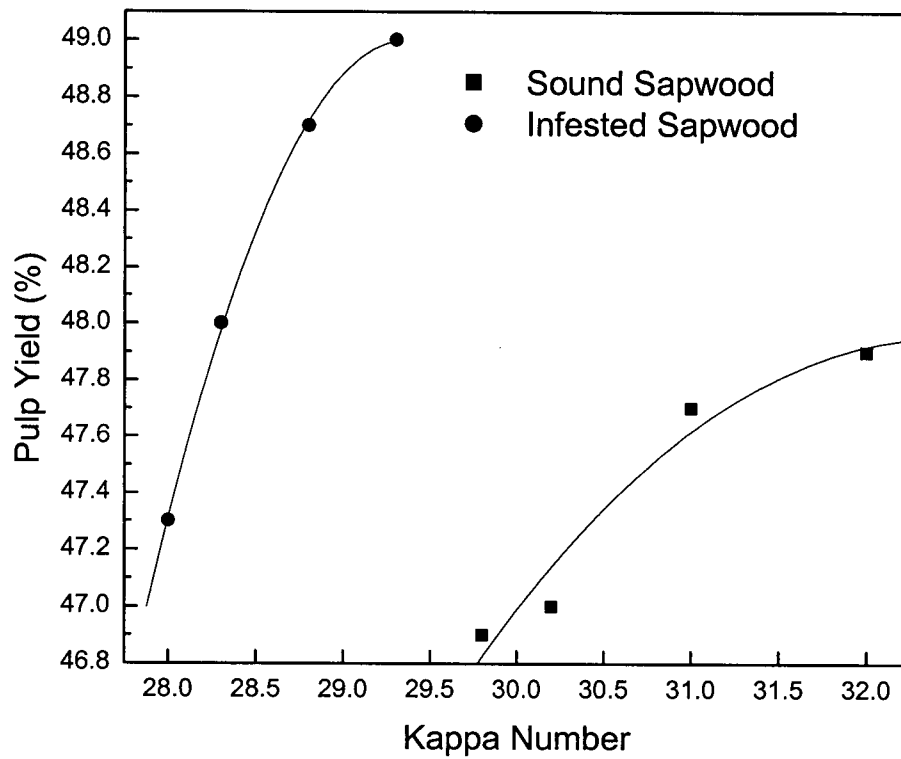
Figure 4-7. Pulp yield (%) versus kappa number for sound and infested lodgepole pine sapwood.
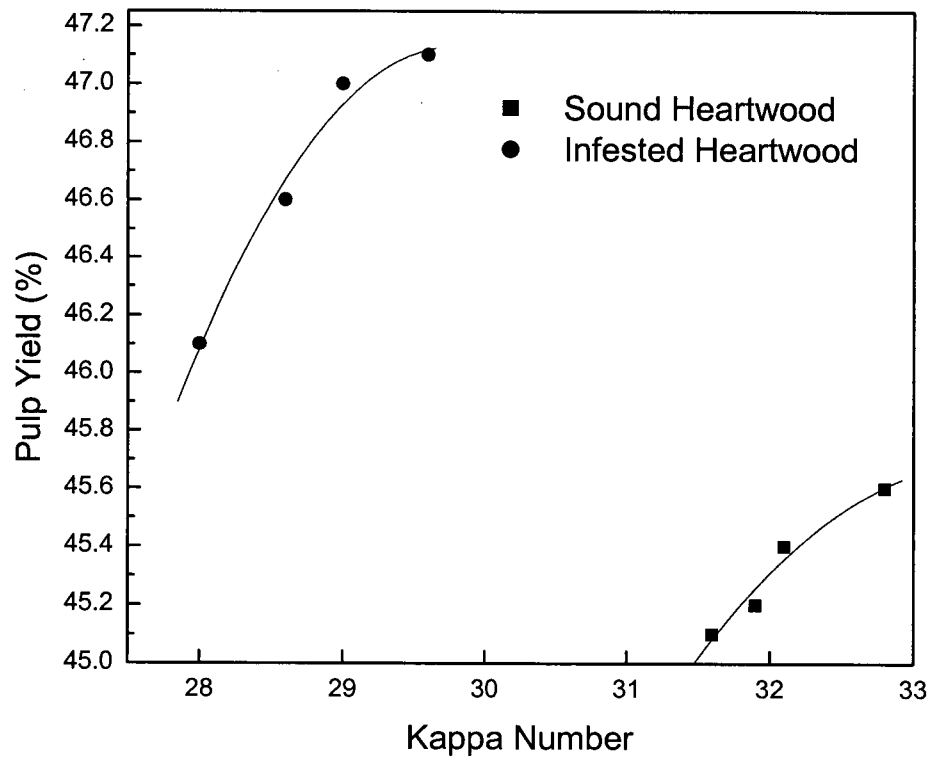
Figure 4-8. Pulp yield (%) versus kappa number for sound and infested lodgepole pine heartwood.

latewood cells, as well as the age and growing conditions of the stem. Fibre length increases generally with the age of the stem (Jozsa and Middleton, 1994). Simultaneously, fibril angle decreases while fibre wall thickness, fibre coarseness and wood density increases. Pulp fibre dimensions also change during chipping of wood and processing of fibres. Lignin and hemicelluloses are released from the fibre wall and the fibres become thinner and more flexible during chemical pulping. Mechanical processes such as mixing and pumping change the fibres and cause curl and kinks that influence the fibre length. Chemical pulp fibres contain approximately 5%-10% fines, 30% middle fraction, and the remainder is long fibres (Levlin and Soderhjelm, 1999).

4.4.1 Handsheet Density

There are many different physical properties that can describe paper, and some of these properties correspond with the end use of the paper. The basic properties of any type of paper include basis weight, thickness and density. Basis weight (also called grammage) is what connects the weight of the material to its surface area, and is the weight per unit area of the paper. Another important parameter that is related to basis weight is apparent sheet density. Apparent density is the mass per unit volume of paper calculated as the ratio between basis weight and thickness of the associated paper. Apparent sheet density is an important parameter because it can affect paper drainage and printing properties. An analysis of the paper produced from the pulp generated from cooks at the same kappa (30) indicated that both the infested sapwood and heartwood have a lower apparent density than that of sound wood (Figure 4-9 and Figure 4-10). For example, at 6000 PFI mill revolutions and at the butt section of the tree, sound sapwood (670 kg/m^3) and sound heartwood (710 kg/m^3) compared to infested sapwood (615kg/m^3) and infested heartwood (630kg/m^3) have a higher apparent density of 55kg/m^3 and 80kg/m^3, respectively. This result correlates with the wood density analysis described in chapter

three. The results show that increased refining of the pulp, increases sheet density because fibers are broken into smaller fibers, which increases interfibre bonding.

In addition, increased refining indicated that topwood generally has a higher sheet density than the butt section for both sapwood and heartwood, which is related to the fact that wood from the top of trees has more juvenile wood and is therefore comprised of thin-walled fibres, which collapse more effectively and increase interfibre bonding (Lowery and Host, 1977; Lieu *et al.*, 1979; Seth, 1995).

4.4.2 Pulp Drainability

The Canadian Standard Freeness test is designed to give a measure of the rate at which a dilute suspension of pulp can be dewatered. The drainage rate, or freeness, has been shown to be related to the surface conditions and swelling of the pulp fibres. Freeness is a useful parameter that helps to illustrate the amount of mechanical treatment applied to pulp, and provides some insight on the drainage behaviour of pulp material on a commercial paper machine (Levlin and Soderhjelm, 1999).

It was apparent from our results that increased refining reduces drainage, as expected. Both the infested sapwood and heartwood have higher freeness values than sound wood, which suggests that they were not developed to the same extent as the pulp originating from sound wood (Figure 4-11 and Figure 4-12), and this correlates well with the density profiles obtained from apparent handsheet density analysis. For example, the freeness value at 3000 PFI mill revolutions and at the topwood section of the tree indicated that the sound sapwood (675 mL) and sound heartwood (635 mL) compared to infested sapwood (710 mL) and infested heartwood (715 mL) had a lower freeness value of 35 mL and 80 mL, respectively. As infested wood has a lower apparent sheet density, higher freeness values were observed because water is able to drain more quickly in a less dense handsheet. When the pulp was extensively refined (12,000

116

Figure 4-9. Apparent sheet density versus refining of sound and infested lodgepole pine sapwood at the butt and topwood section for a total of 16 samples.

Figure 4-10. Apparent sheet density versus refining of sound and infested lodgepole pine heartwood at the butt and topwood section for a total of 16 samples.

Figure 4-11. Canadian standard freeness versus refining of sound and infested lodgepole pine sapwood at the butt and topwood section for a total of 32 samples. Error bars indicate range.

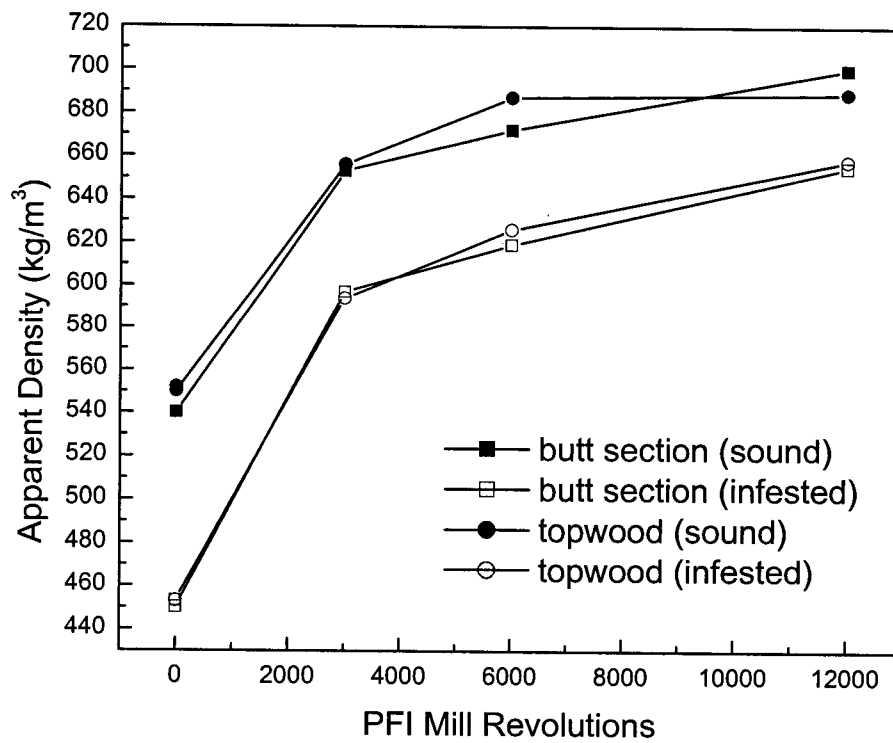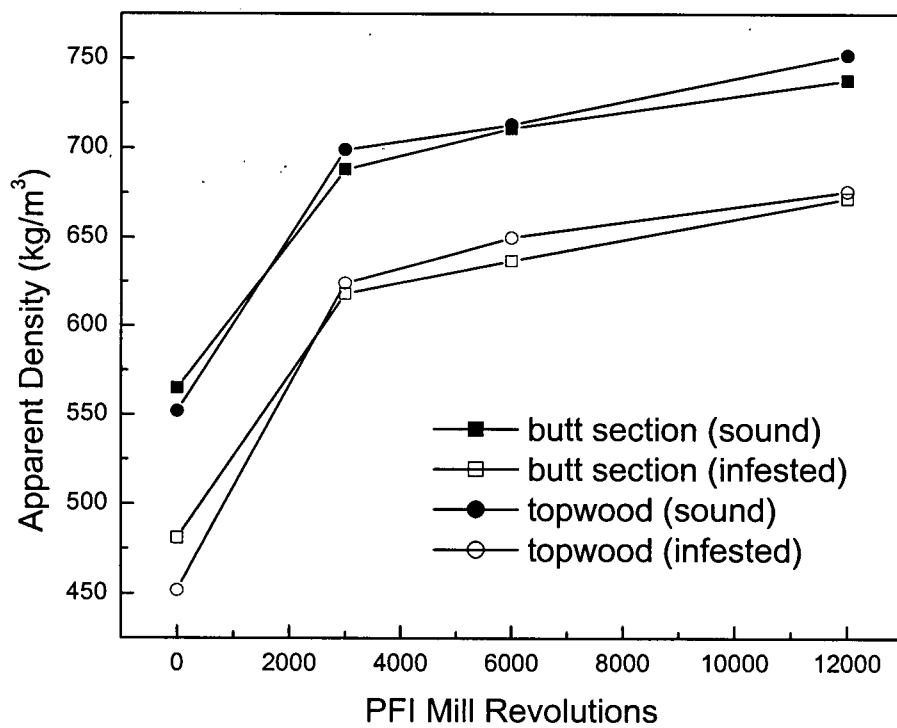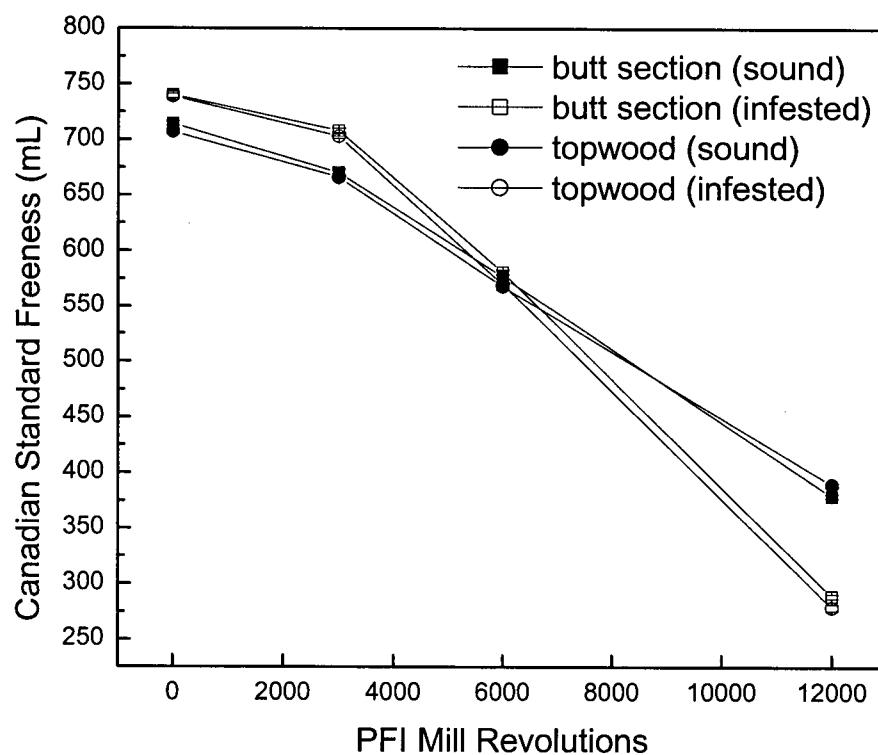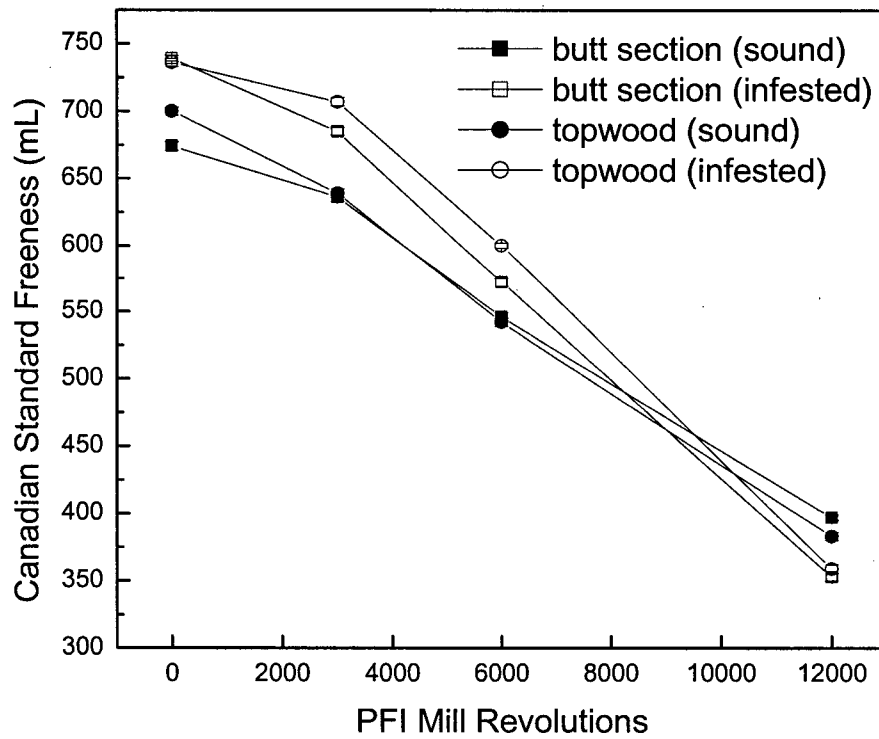**Figure 4-12.** Canadian standard freeness versus refining of sound and infested lodgepole pine heartwood at the butt and topwood section for a total of 32 samples. Error bars indicate range.

revolutions), sound wood was shown to exhibit a higher freeness value than dead wood. This is likely due to the fact that with increased refining, a greater production of fines is generated. The fines material produces a more compact sheet because there is more surface bonding and as a result a more compact sheet will reduce water drainage. Moreover, as the results demonstrate that at higher levels of refining, pulp drainage tends to decrease compared to sound wood, and this suggests that the infested wood is less compliant and perhaps the fibres are stiffer and therefore is not as susceptible to refining as the sound wood. For both sapwood and heartwood, there appears to be no apparent trend in terms of the effect of tree height on pulp drainability.

4.4.3 Handsheet Strength Properties

The most important strength properties of paper include tensile, burst and tear strengths. Tensile strength is a very useful property to describe the general strength of any material. For paper, it is the maximum force per unit width that a paper strip can resist before breaking when applying a load parallel to the length of the paper strip. Tensile strength is measured by calculating the tensile index, which is the ratio of tensile strength to basis weight (Levlin and Soderhjelm, 1999). The tensile index values relate strength to the amount of material being loaded. Therefore, tensile index is a very good indicator of pulp strength. While tensile strength of paper depends on individual fibre strength, it is also dependent on the degree of bonding between fibres. It has been suggested that the tensile index is an illustration of the force required to break interfibre bonds and some individual fibres (Levlin and Soderhjelm, 1999).

It was apparent that the tensile strength of infested sapwood and heartwood were lower than sound wood (Figure 4-13 and Figure 4-14). For example, the tensile strength at 6000 PFI mill revolutions from the topwood section of the tree indicated that the sound sapwood (118 N·m/g) and sound heartwood (120 N·m/g) compared to infested sapwood (110 N·m/g) and infested heartwood (110 N·m/g) had a higher tensile strength of 8 N·m/g and 10 N·m/g,

respectively. As a result, sound wood produces a stronger handsheet compared to infested wood. McGovern (1951) reported similar findings when he compared green versus insect-killed lodgepole pine.

In addition, generally topwood possessed a higher tensile index than handsheets produced in the butt section for both sapwood and heartwood. This is likely related to the fact that topwood is comprised of more juvenile wood, which has thinner cell walls and therefore collapse more effectively, and improves interfibre bonding. In effect, increased interfibre bonding increases the tensile index (Smook, 1992).

Bursting strength, which is another important paper strength property, is the maximum pressure that the paper can resist without breaking when pressure is applied perpendicular to the plane (Levlin and Soderhjelm, 1999). Bursting strength is measured by calculating the burst index, which is the ratio of bursting strength to basis weight. Thus, the burst index is the measure of pressure required to rupture paper. Lowery *et al.* (1977) and McGovern (1951) observed that dead wood, in general, showed lower burst index values than sound wood. Our research agreed with these findings, as sound sapwood and heartwood typically produced higher burst index values when compared to infested wood (Figure 4-15 and Figure 4-16). For example, the burst strength at 6000 PFI mill revolutions from the topwood section of the tree indicated that the sound sapwood (11.5 $kPa \cdot m^2/g$) and sound heartwood (10 $kPa \cdot m^2/g$) compared to infested sapwood (9.3 $kPa \cdot m^2/g$) and infested heartwood (9 $kPa \cdot m^2/g$) had higher burst strength of 2.2 $kPa \cdot m^2/g$ and 1.0 $kPa \cdot m^2/g$, respectively. Hence, handsheets produced from sound wood required more pressure to rupture. This is likely due to the fact that infested wood showed lower apparent sheet density values than sound wood and therefore is more easily ruptured as its handsheet is not as compact as that of sound wood. Moreover, handsheets from the topwood section for both sapwood and heartwood showed substantially higher burst index

values than handsheets from the butt section. Once again, topwood contains more juvenile wood and therefore there exhibits greater interfibre bonding as mentioned previously.

Another important paper strength parameter is tearing resistance. The tearing strength, or the internal tearing resistance, is the force required to continue the tearing of paper from an initial cut. The tearing strength is usually represented by the tear index, which is the ratio of tearing strength to basis weight (Levlin and Soderhjelm, 1999). The tearing strength of paper depends on several factors such as fibre length, fibre strength, and the degree of bonding between fibres. Longer and stronger fibres provide higher tearing strength. At low levels of bonding, the tearing strength increases with increased bonding. However, at higher degrees of bonding, fibre strength determines the level of tearing strength (Seth, 1995; Levlin and Soderhjelm, 1999). In general, an increase in tensile index will generally lead to a decrease in the tear index because the tearing force is concentrated in a small area as the number of interfibre bonds increase (Koch, 1996).

As expected, the tear index analysis indicated that both sound sapwood and heartwood showed lower tear index values when compared to infested wood (Figure 4-17 and Figure 4-18). For example, the tear strength at 6000 PFI mill revolutions from the butt section of the tree indicated that the sound sapwood (11.2 mN·m^2/g) and sound heartwood (9.3 mN·m^2/g) compared to infested sapwood (14.8 mN·m^2/g) and infested heartwood (12.9 mN·m^2/g) had a lower tearing strength of 3.6 mN·m^2/g, respectively. Lowery *et al.* (1977) and McGovern (1951) reported similar results. Conversely, handsheets from the butt section of both sapwood and heartwood produced slightly higher tear index values than the topwood section. This is likely due to the fact that the butt section contains more mature wood, which is known to have longer fibres than juvenile wood. Furthermore, the sound wood exhibited higher sheet densities, which is known to cause reduction in tear strength.

Figure 4-13. Tensile index versus refining of sound and infested lodgepole pine sapwood in the butt and topwood section for a total of 80 samples. Error bars indicate standard deviation.

Figure 4-14. Tensile index versus refining of sound and infested lodgepole pine heartwood at the butt and topwood section for a total of 80 samples. Error bars indicate standard deviation.
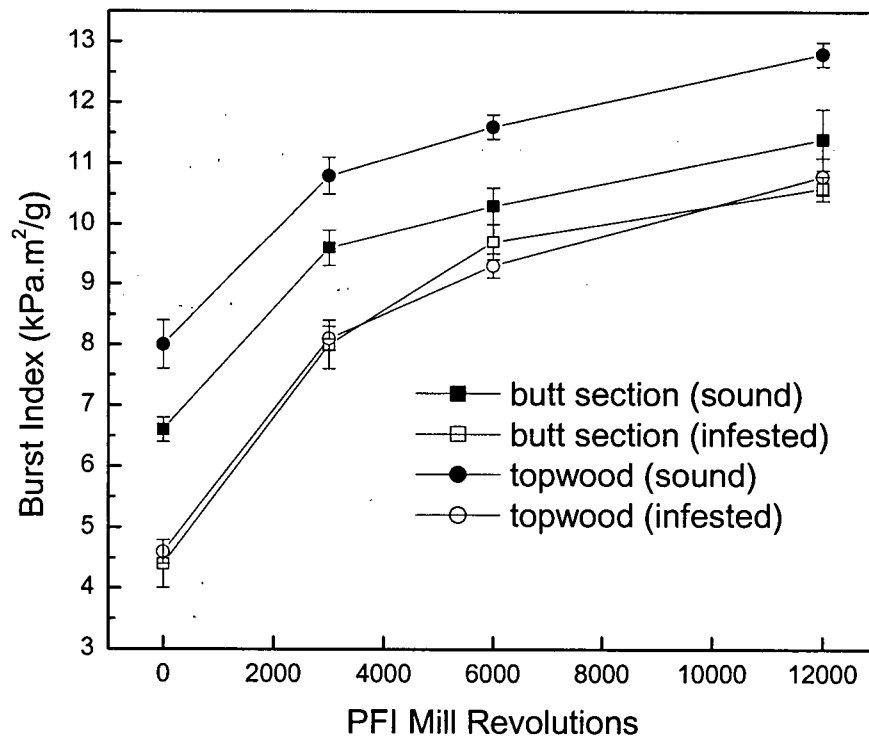
Figure 4-15. Burst index versus refining of sound and infested lodgepole pine sapwood at the butt and topwood section for a total of 80 samples. Error bars indicate standard deviation.

126

Figure 4-16. Burst index versus refining of sound and infested lodgepole pine heartwood at the butt and topwood section for a total of 80 samples. Error bars indicate standard deviation.

Figure 4-17. Tear index versus refining of sound and infested lodgepole pine sapwood at the butt and topwood section for a total of 80 samples. Error bars indicate standard deviation.

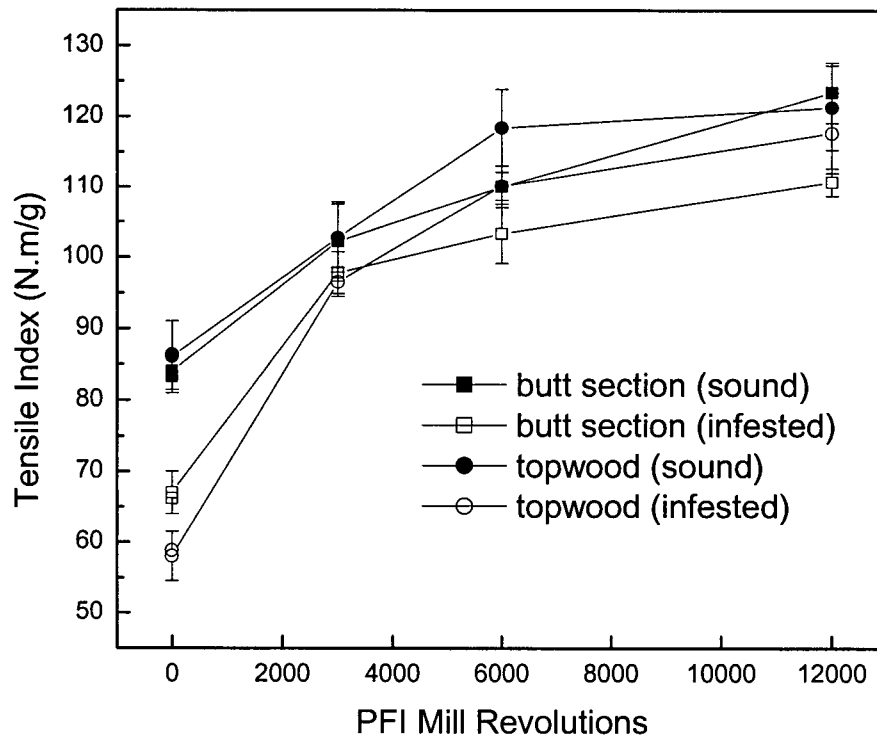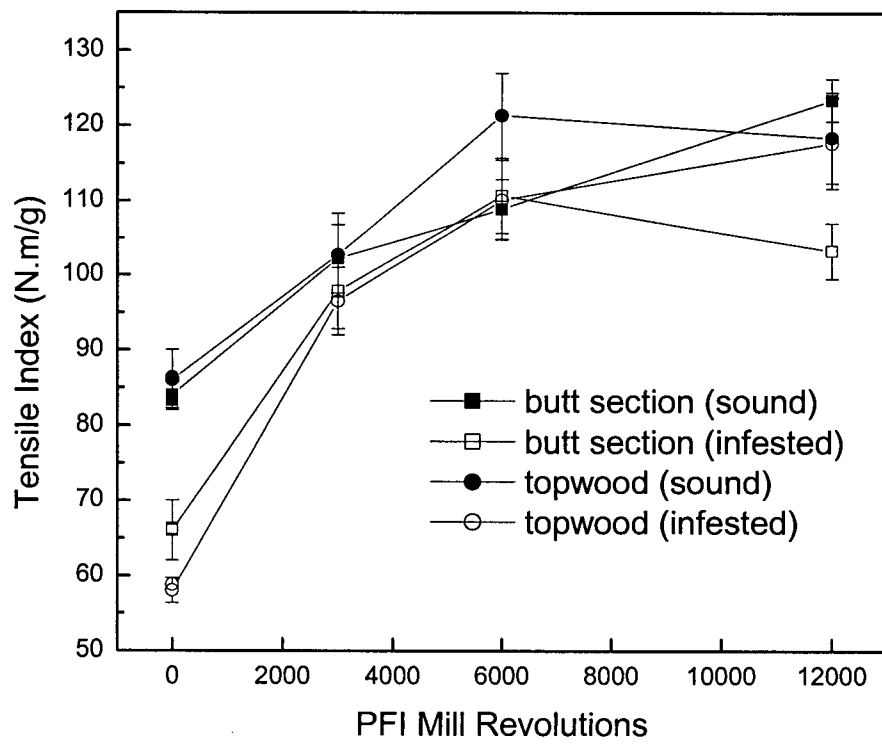**Figure 4-18.** Tear index versus refining of sound and infested lodgepole pine heartwood at the butt and topwood section for a total of 80 samples. Error bars indicate standard deviation.
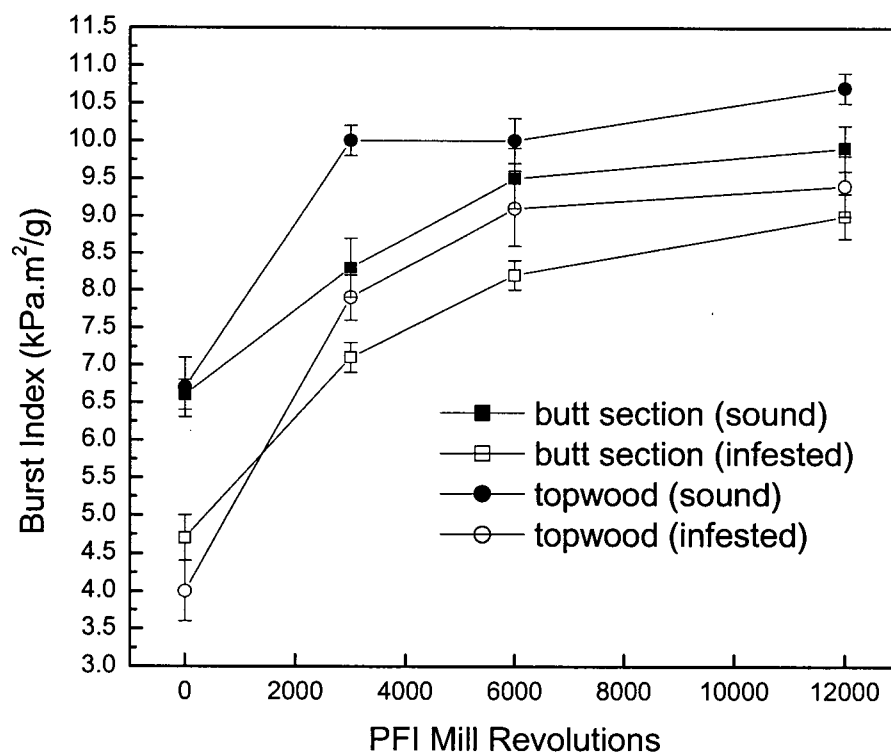
4.4.4 Handsheet Surface Properties

Air porosity is the measure of the compactness of fibres in paper and is normally determined by measuring the flow of air through a defined area of paper (Levlin and Soderhjelm, 1999). The results indicate that with increased refining, both sapwood and heartwood (Figure 4-19 and Figure 4-20), demonstrated increased air resistance and therefore lowered air porosity. This can be attributed to the increased apparent sheet density, which is an indication of sheet compactness. As a result, a more compact handsheet causes a decrease in porosity. In addition, infested sapwood and heartwood showed lower air resistance compared to sound wood, which indicates that paper produced from infested wood is more porous. For example, the air resistance at 6000 PFI mill revolutions from the butt section of the tree indicated that the sound sapwood (23 sec/100 mL) and sound heartwood (40 sec/100 mL) compared to infested sapwood (5 sec/100 mL) and infested heartwood (10 sec/100 mL) had a higher air resistance of 18 sec/100 mL and 30 sec/100 mL, respectively. However, there was no apparent trend in terms of the effect of tree height on air porosity.

The smoothness or roughness is a measure of the topography of the paper surface. Smoothness is measured by the rate at which air leaks across the surface paper. The surface property of paper is crucial to the paper industry such that it influences printing quality because uniform ink coverage of the paper is dependent on smoothness. Paper surface properties also affect coating absorption and gloss (Levlin and Soderhjelm, 1999; Smook, 1992). The results from the smoothness tests showed that for both sound sapwood and heartwood, smoother surfaced paper is produced than the corresponding infested wood (Figure 4-21 and Figure 4-22). For example, Sheffield roughness at 6000 PFI mill revolutions from the butt section of the tree indicated that the sound sapwood (180 Sheffield units) and sound heartwood (110 Sheffield units) compared to infested sapwood (270 Sheffield units) and infested heartwood (230 Sheffield

units) produced a paper that was less rough. Moreover, handsheets from the topwood section of the tree produced a smoother piece of paper for both sapwood and heartwood compared to handsheets made from the butt section. As the butt section is mainly composed of mature wood, it is known that mature wood possesses coarser fibres compared to juvenile wood (Koch, 1996). Since the topwood section contains more juvenile wood than the butt section, a smoother paper will be produced, as the fibres are less coarse.

4.5 Fibre Quality

Fibre length is an important attribute to the pulp and paper industry. It can be used to determine juvenility because juvenile wood tends to have shorter fibres. Fibre length is shortest next to the pith at all heights of the stem, and increases radially outward with age (Hatton and Gee, 1994). In addition to fibre length, fibre coarseness, which is the mass of fibres per unit length, is another important quality descriptor. Fibre coarseness is an important pulp parameter because it is a measure of fibre flexibility and the thickness of cell walls (Seth, 1995). Generally, thinner cell walls are favoured, as they are indicated by low coarseness values. Juvenile wood is known to have short fibres and lower fibre coarseness than mature wood. Finer fibres tend to form better quality paper sheets than coarser fibres, which are typical of mature wood (Seth, 1995). The results obtained from the wood fibre quality analysis showed that for both sapwood and heartwood, the infested wood possessed longer fibres with higher coarseness values (Table 4-4 and Table 4-5). Consequently, a similar trend was found for the pulp fibre quality analysis (Table 4-6 and Table 4-7). These results correlate with the tearing strength test, such that infested wood had higher tear index values than sound wood, which is an attribute heavily influenced by fibre length in low density sheets. Moreover, handsheets produced from infested wood were rougher and this correlates with the high coarseness values observed in infested wood. Sound wood was observed to have shorter fibres and lower coarseness values

Figure 4-19. Air resistance versus refining of sound and infested lodgepole pine sapwood at the butt and topwood section for a total of 80 samples. Error bars indicate standard deviation.
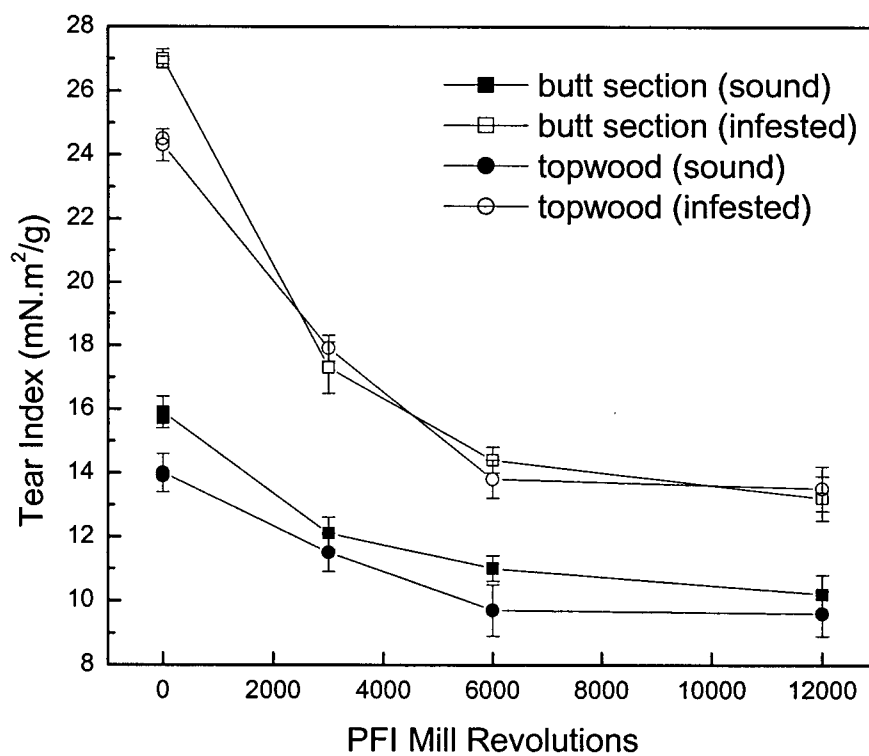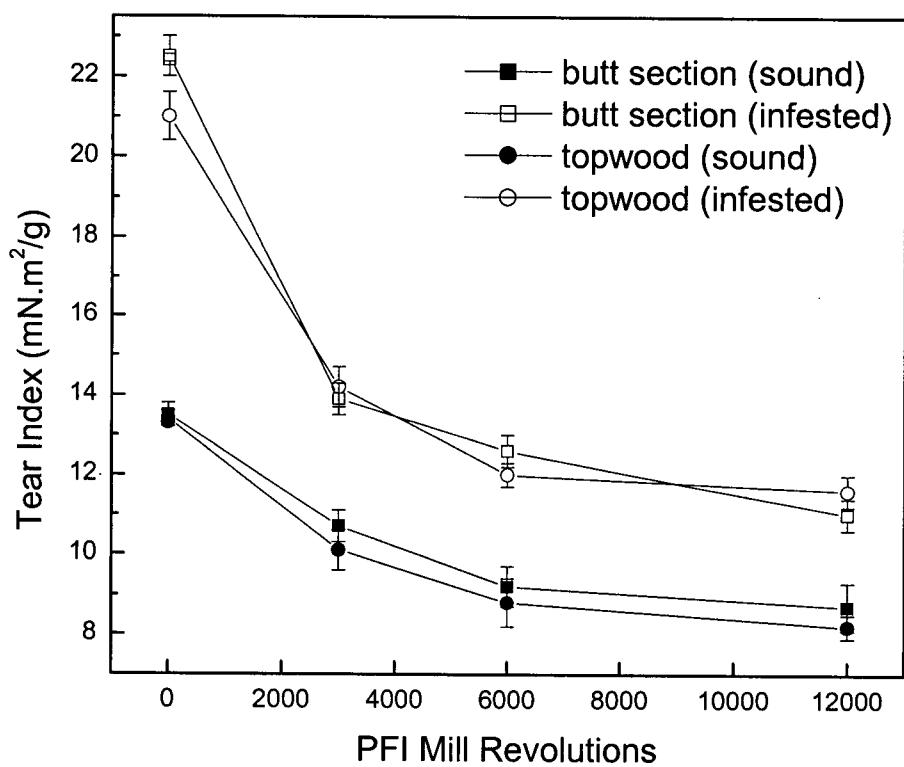
Figure 4-20. Air resistance versus refining of sound and infested lodgepole pine heartwood at the butt and topwood section for a total of 80 samples. Error bars indicate standard deviation.
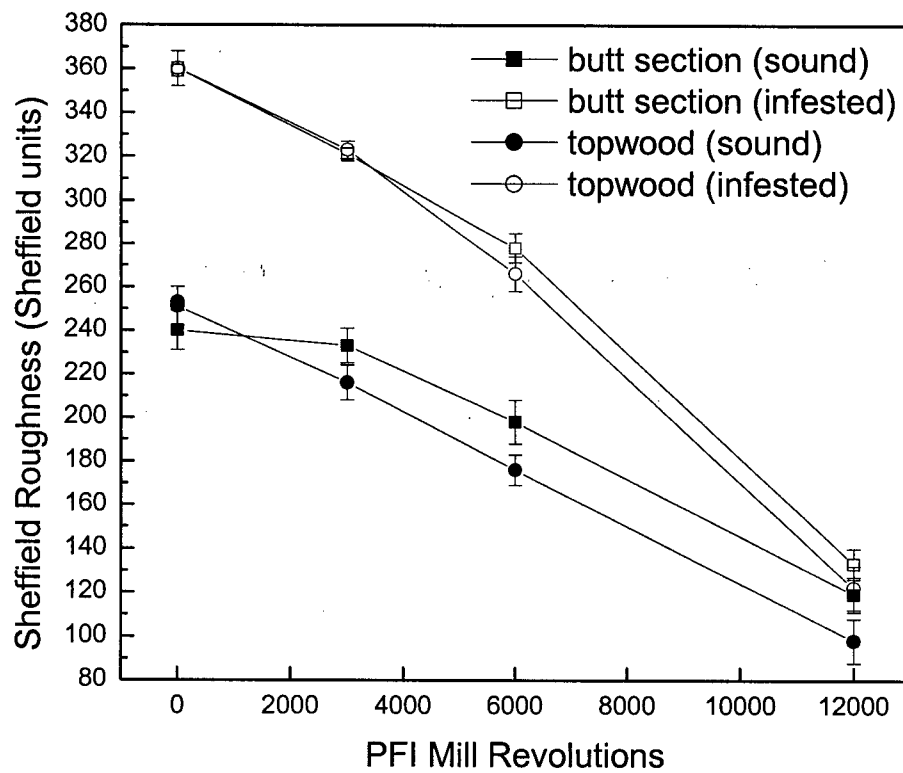
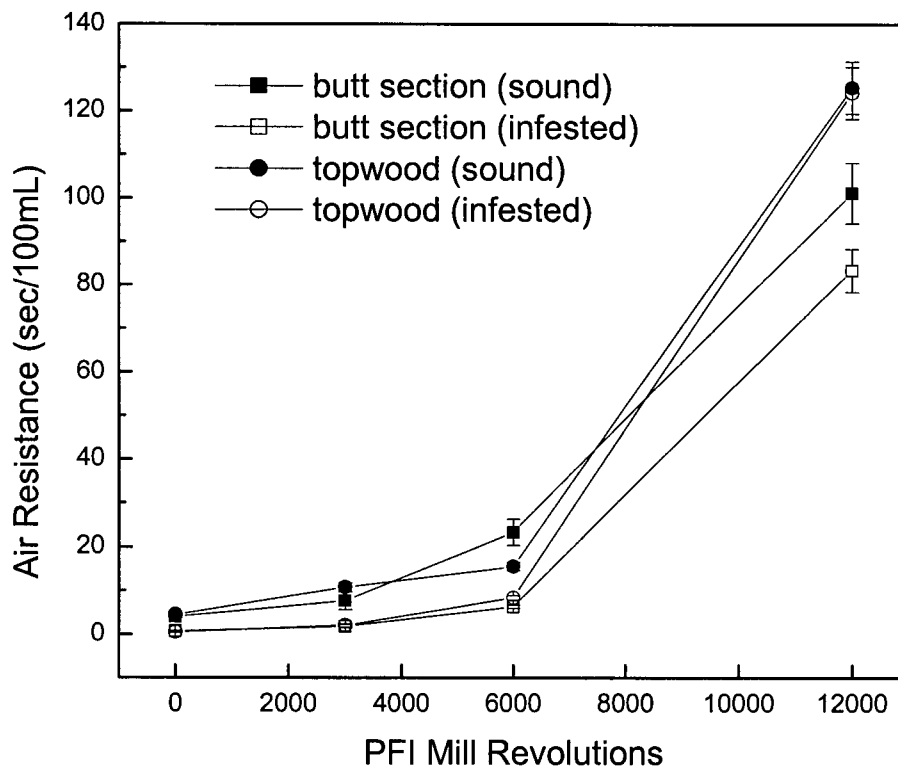**Figure 4-21.** Sheffield roughness versus refining of sound and infested lodgepole pine sapwood at the butt and topwood section for a total of 80 samples. Error bars indicate standard deviation.
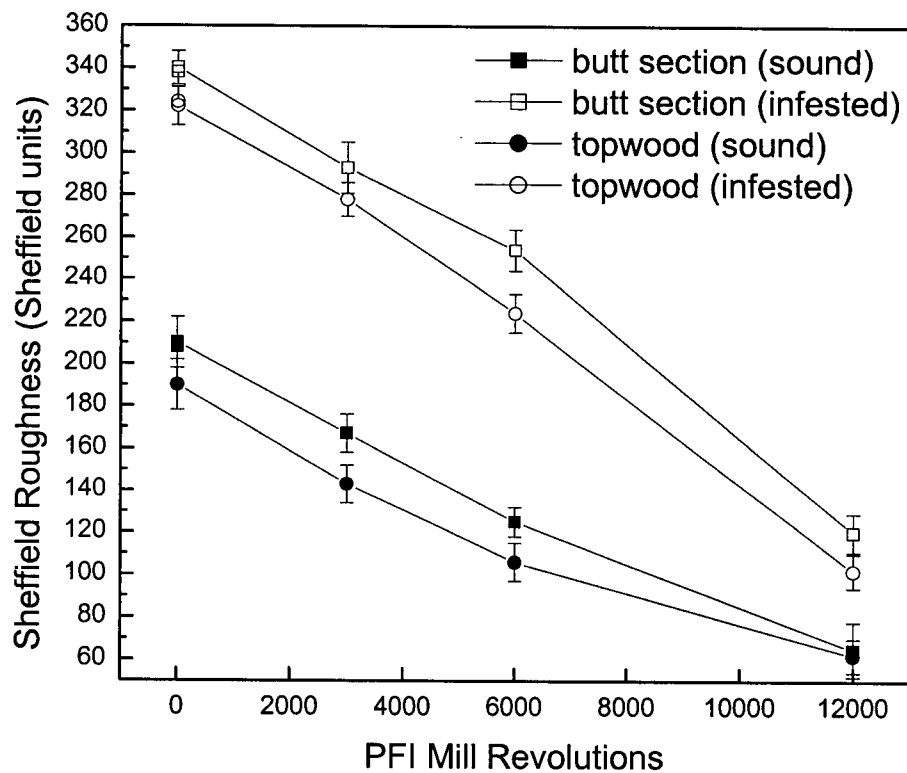
Figure 4-22. Sheffield roughness versus refining of sound and infested lodgepole pine heartwood at the butt and topwood section for a total of 80 samples. Error bars indicate standard deviation.
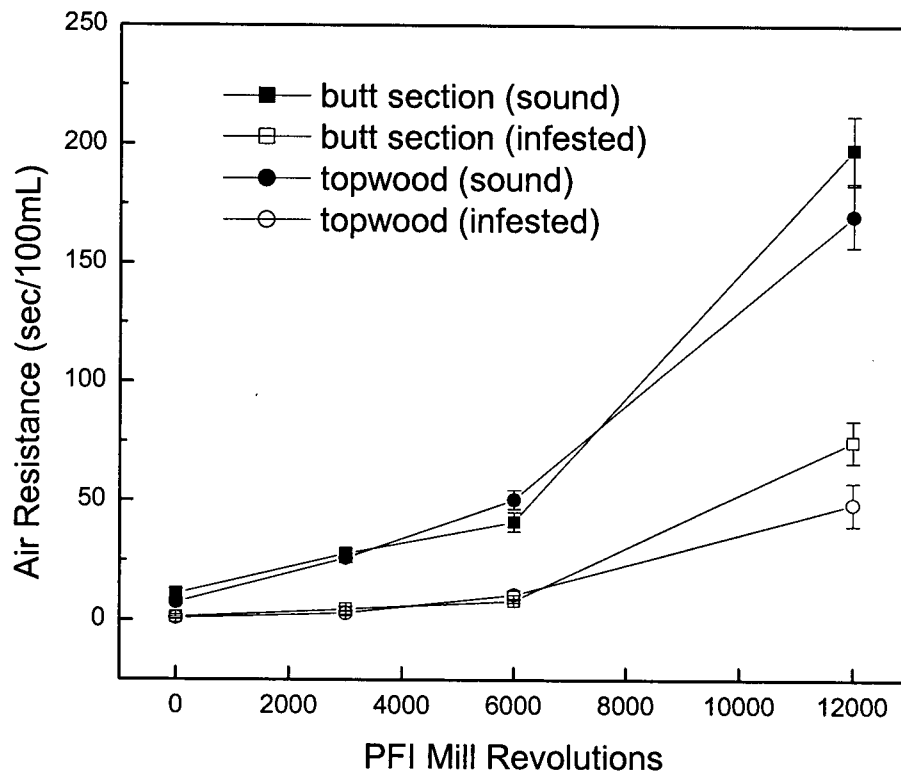
Table 4-4. Macerated wood fibre length and coarseness values for sound and infested lodgepole pine sapwood at the butt and topwood section.

Tree Position	Fibre Length (mm)		Coarseness (mg/m)	
	sound	infested	sound	infested
butt section	2.78 (0.03)	3.31 (0.02)	0.134 (0.004)	0.174 (0.002)
topwood	2.50 (0.04)	3.15 (0.02)	0.131 (0.006)	0.156 (0.005)

* Values in parentheses show standard deviation.

Table 4-5. Macerated wood fibre length and coarseness values for sound and infested lodgepole pine heartwood at the butt and topwood section.

Tree Position	Fibre Length (mm)		Coarseness (mg/m)	
	sound	infested	sound	infested
butt section	2.14 (0.05)	2.56 (0.03)	0.157 (0.002)	0.172 (0.006)
topwood	1.84 (0.03)	2.14 (0.04)	0.133 (0.003)	0.155 (0.002)

* Values in parentheses show standard deviation.

Table 4-6. Pulp fibre length and coarseness values for sound and infested lodgepole pine sapwood at the butt and topwood section.

Tree Position	Fibre Length (mm)		Coarseness (mg/m)	
	sound	infested	sound	infested
butt section	3.11 (0.02)	3.29 (0.02)	0.166 (0.006)	0.221 (0.002)
topwood	2.81 (0.02)	3.28 (0.02)	0.167 (0.003)	0.216 (0.005)

* Values in parentheses show standard deviation.

Table 4-7. Pulp fibre length and coarseness values for sound and infested lodgepole pine heartwood at the butt and topwood section.

Tree Position	Fibre Length (mm)		Coarseness (mg/m)	
	sound	infested	sound	infested
butt section	2.48 (0.02)	2.67 (0.02)	0.157 (0.003)	0.218 (0.005)
topwood	2.19 (0.02)	2.65 (0.03)	0.148 (0.003)	0.218 (0.003)

* Values in parentheses show standard deviation.

which suggest that sound wood fibres are more flexible, have thinner cell walls, and collapse more efficiently. Therefore, enhanced collapse of fibres, produces increased interfibre bonding and thus higher tensile and burst indices (Seth, 1995).

In terms of tree height effect, typically the topwood had shorter fibres exhibiting lower coarseness values because topwood is composed of juvenile wood compared to the butt section. In addition, the results also indicated that heartwood, in general, had shorter fibres and lower coarseness values when compared to sapwood. This is likely due to the fact that heartwood also contains more juvenile wood compared to sapwood.

CHAPTER 5. CONCLUSIONS AND FUTURE WORK

5.1 Conclusions and Implications

The mountain pine beetle infestation has devastated the lodgepole pine resource in Canada. The effect of mountain pine beetle killed wood on product quality has been the major concern for the utilization of dead lodgepole pine in the pulp and paper industry. However, there are a paucity of studies that are available on the impact of mountain pine beetle killed lodgepole pine on wood chemistry, morphology and pulping properties.

The primary objective of the present investigation was to provide some insight on the influence of mountain pine beetle killed wood on pulp fibre quality using matched sound and infested samples from the same site. As demonstrated from this study, it is apparent that upon beetle attack, the tree undergoes several changes. The gravest changes that occur are the incursion of blue stain fungi and the severe water stress in sapwood that follows beetle invasion, which are believed to be the primary causes of tree death. The results indicate that infested sapwood and heartwood exhibit substantial moisture loss and that moisture content decreases with increased tree height. Additionally, the wood density analysis demonstrated that infested wood is less dense than sound wood and this is likely related to the fungal degradation of lignin and carbohydrates. Wood density was also shown to decrease with increased tree height for both sound and infested wood. This result may be attributed to more juvenile wood being present with increased stem height. Moreover, the chemical analysis indicated that infested sapwood contains less extractive content when compared to sound sapwood. No significant difference was found in infested and sound heartwood. The lower extractive content in infested sapwood is likely due to the fact that beetles and fungi utilize the cellular components to sustain life. Typically, increased extractive content towards the crown was shown in sound and infested sapwood and a decrease in extractive content towards the upper bole was shown in heartwood.

138

In addition, through a comprehensive analysis of the individual classes of extractives indicated that infested wood consisted of higher fatty acid proportions and lower steroid, steryl ester and triglyceride proportions. The reduced extractive proportions are typically the most readily degraded components by fungi present in infested wood. Moreover, klason lignin analysis demonstrated that less lignin content was present in infested sapwood compared to sound, and no trend was found for heartwood. The reduced lignin content in infested sapwood can be attributed to not only beetle infestation, but also increased acid soluble lignin in infested sapwood. No apparent trend was found in the influence of tree height on lignin content. Furthermore, the carbohydrate analysis showed a reduction in carbohydrate content for infested wood as fungi consume the carbohydrates more vigorously. Carbohydrate content generally increased with increased tree height in sound sapwood, as more stable sugars are produced towards the crown. However, carbohydrate content decreased with increased tree height in infested sapwood as the tree is more prone to beetle attack particularly towards the stem midbole. Again, no trend was found for heartwood. An evaluation of carbohydrate content indicated that infested wood contained less hemicellulose-derived sugars as hemicellulose content is typically the first material to be consumed by fungi.

Consequently, the permeability analysis demonstrated that infested sapwood is more permeable than sound sapwood; while sound heartwood is more permeable than infested heartwood. From the microscopic analysis, it was found that fungal hyphae are present in infested sapwood, which is likely the primary reason for increased permeability in infested sapwood. The presence of aspirated pits may contribute to the heartwood being less permeable than the sapwood. Variation in permeability with tree height was shown in both sapwood and heartwood and is likely related to the differences in concentration of extractives towards the upper bole.

Generally, lodgepole pine is used as wood chips in the pulp and paper industry. The chip quality analysis revealed that infested wood generated more fines, as this is likely due to the reduced moisture content. However, tree height did not appear to affect chip size distribution. As chip quality directly influence pulp quality, the results from the pulping analysis showed that infested wood has less residual lignin at any given H-factor for sapwood and heartwood. No clear trend indicating an effect of tree height on residual lignin was apparent. Infested wood also consumes less alkali, which allows it to pulp more rapidly. These results correlate with permeability, as infested wood is more permeable than sound wood; liquor penetrability is more effective. The pulping of infested wood generated a higher pulp yield compared to sound wood as infested wood contains a larger percentage of glucose, as on a weight basis.

As timber is harvested and sold on the basis of volume, using the results obtained from this study, it is apparent that to produce one tonne of unbleached kraft pulp, $5.25m^3$ of sound sapwood (Figure 5-1) and $5.70m^3$ of sound heartwood (Figure 5-2) versus $5.83m^3$ of infested sapwood and $6.49m^3$ of infested heartwood will be required. Therefore, a greater volume of infested wood (roughly 10% and 13%) is required to produce the same quantity of pulp compared to sound wood. Differences in wood density increases the cost of purchasing the required amount of wood necessary to produce 1 tonne of pulp.

Figure 5-1. Volume of wood required to produce 1 tonne of pulp for sound and infested lodgepole pine sapwood.
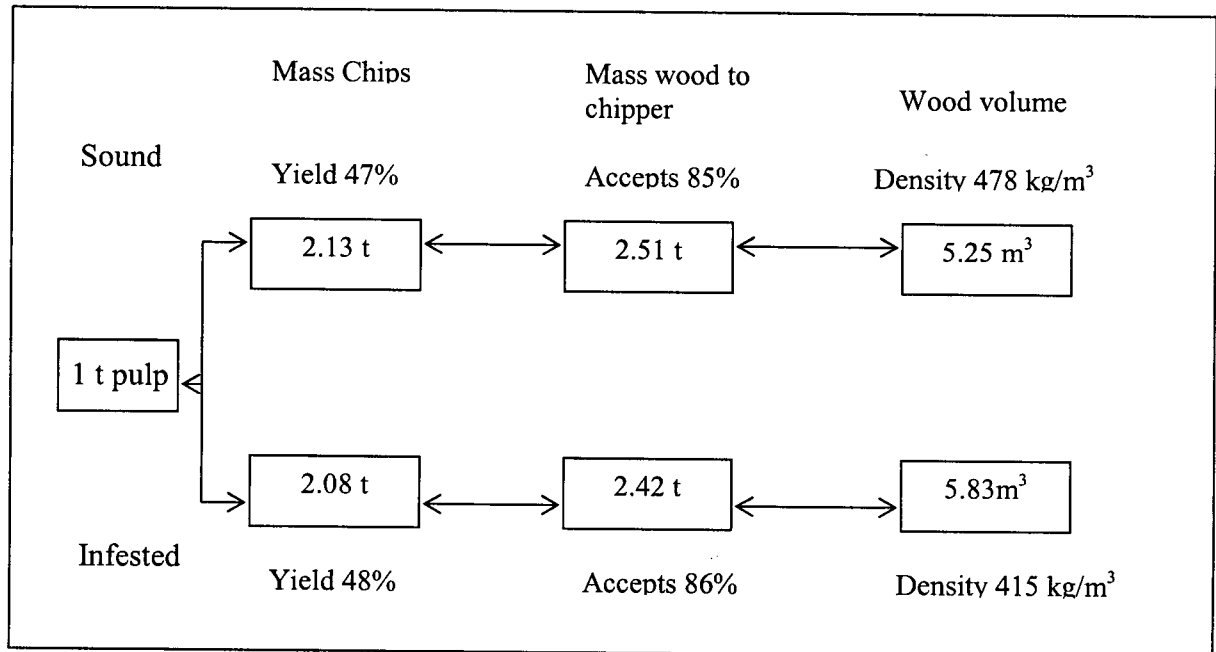
Figure 5-2. Volume of wood required to produce 1 tonne of pulp for sound and infested lodgepole pine heartwood.
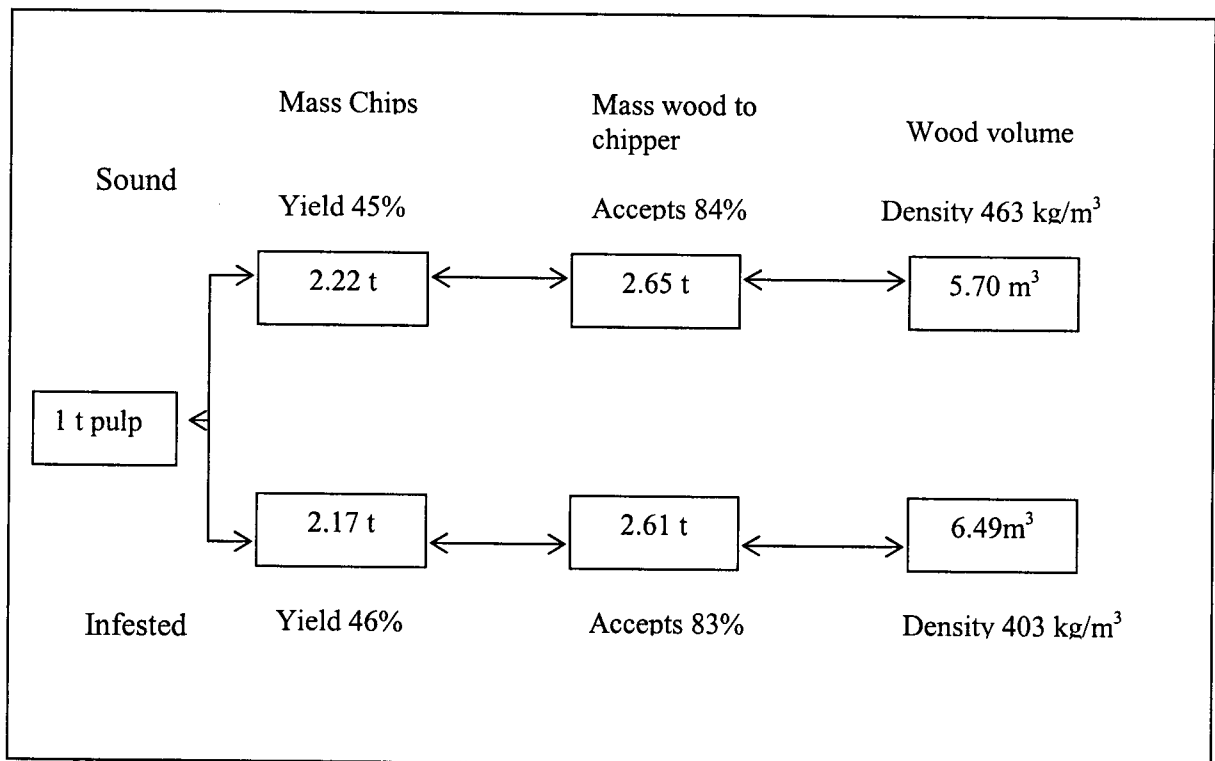
Additionally, the paper quality analyses demonstrated that infested wood produces an inferior paper to sound wood, as paper from infested wood has a lower burst and tensile indices, and higher tear strengths. The paper quality from infested wood is also more porous, less smooth and less dense. The differences in strength and paper quality of infested wood may be more a result of the inherent original fibre attributes, rather than the effect of beetle or fungi in that the results from the fibre quality analysis demonstrate that infested wood has longer and coarser fibres. As infested wood has longer fibres, it exhibits a higher tear index. Similarly, as a result of infested wood having coarser fibres, it produces a rougher paper. Moreover, sound wood has shorter fibres and lower coarseness values, which may enhance fibre collapse and increase paper strength.

Clearly, mountain pine beetle killed wood affects the morphology, chemistry and pulpability of the wood. However, the quality differences in mountain pine beetle killed wood disclosed in the current investigation are not so significant that the resource cannot be utilized. If decay is severe, it will have a significantly negative impact on industrial efficiency. Therefore, provided that decay is not too far advanced, beetle killed wood can be used for most of the purposes as sound wood.

5.2 Future Work

As the present investigation endeavours to provide some insight into the effect of mountain pine beetle on wood and pulp fibre quality, much more research is necessary to expand our understanding. As we intend to effectively use the ever growing quantities of mountain pine beetle killed lodgepole pine currently available, we must understand the interrelationships between time of tree death, beetle activity, pulp yield, pulp quality and paper properties.

As an extension to the present investigation, a further analysis with increased tree sample size within the stand is necessary to determine whether changes apparent in infested lodgepole pine are attributed to mountain pine beetle infestation or within tree-to-tree variations. In addition, the essential questions relative to the utilization of mountain pine beetle killed wood in pulping include:

(1) How long can a tree be left after beetle infestation before its quality deteriorates to a point beyond which it can no longer be utilized?

(2) What is the mechanism of wood decay (are other fungi present following incipient decay)?

(3) Is cellulose crystallinity affected by dryness?

(4) How is lignin distributed through fibres and how is it affected by fungal infestation?

(5) What kinds of storage processes can be used to store mountain pine beetle killed wood chips?

(6) Which pulping processes are more tolerant to the effects of reduced wood quality?

(7) What is the influence of beetle or fungi on bleaching chemical consumption?

Answers to the preceding key questions relative to the use of mountain pine beetle killed lodgepole pine can significantly impact the pulp and paper production in the near future.

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