## AN ABSTRACT OF THE THESIS OF

Adam A. Scouse for the degree of <u>Master of Science</u> in <u>Wood Science</u> presented on <u>September 13, 2012</u>.

Title: Essential Oil Treatment of VTC Wood.

Abstract approved:

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Western juniper and cinnamon essential oils were combined with ethanol at 2.5, 5, and 10% concentrations by weight and applied to hybrid poplar (*Populus trichocarpa x P. deltoids*) veneers by vacuum soaking to produce a naturally durable wood veneer with increased mechanical properties for use in structural composites. Half of these veneers were then modified using viscoelastic thermal compression to increase veneer density and modulus of elasticity. Following densification, unprocessed and VTC processed veneers receiving an essential oil treatment were subjected to an AWPA E21-06 Formosan termite exposure test, AWPA E24-06 mold box test, and brown rot (*Gloeophyllum trabeum*) decay bending test. While VTC processing drastically reduced the abundance of chemical components inherent within essential oil treatments, veneer specimens without VTC processing showed increased durability. A 10% juniper oil treatment drastically reduced Formosan termite attack on hybrid poplar veneers while a 10% cinnamon oil treatment significantly reduced mold growth. Timbor®, an industrial powdered borate treatment, withstood VTC

processing and inhibited Formosan termite attack and mold growth. Tests to evaluate the effectiveness of essential oil treatments against brown rot were unsuccessful. Results suggest that incorporating a disodium octaborate tetrahydrate (DOT) treatment prior to VTC processing could help improve VTC wood durability. ©Copyright by Adam A. Scouse September 13, 2012 All Rights Reserved Essential Oil Treatment of VTC Wood

by Adam A. Scouse

# A THESIS

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I understand that my thesis will become part of the permanent collection of Oregon State University libraries. My signature below authorizes release of my thesis to any reader upon request.

Adam A. Scouse, Author

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Essential Oil Treatment of VTC Wood

#### **CHAPTER 1 – INTRODUCTION**

#### **1.1 Background**

One possibility for improving the performance of wood products involves densification of wood veneers using steam, high temperatures, and compression. This process, termed viscoelastic thermal compression (VTC), has the ability to increase strength and stiffness properties of otherwise low density wood (O'Connor, 2007). VTC processed wood could be used to create stronger structural composite components from low density commercial species. These products made from VTC wood need to resist biological deterioration.

While the architectural design of buildings plays a key role in limiting wood decay, a common method for ensuring protection against mold, decay, and termites is to treat wood with preservatives. Preservative treatments are generally oil-soluble chemicals or waterborne salts. Identifying the most appropriate treatment option requires careful consideration of the performance requirements and end use. However, wood preservative systems are not perfect and some have legitimate performance and environmental concerns.

Chromated copper arsenate (CCA) was introduced into the U.S. market in the early 1950's (Townsend & Solo-Gabriele 2006). Concern about the environmental impacts of some CCA components led the industry to withdraw this preservative from residential use in 2003. The U.S. Environmental Protection Agency indicated that, as of 2003, "no wood treater or manufacturer may treat wood with CCA for residential uses, with certain exceptions" (Wormell 2011). Consumers are becoming increasingly concerned with the environmental impact of their purchases and want products that are perceived as environmentally friendly.

One example of this perception change is the Leadership in Energy and Environmental Design (LEED) Living Building Challenge. LEED attempts to define the most advanced measure of sustainability in the built environment and diminish the gap between current limits and ideal solutions. Buildings that meet the Living Building Challenge must conform to a material list identifying chemicals or materials that cannot be present in building projects. Arsenic, creosote and pentachlorophenol are among the materials that can not be used (McLennan 2010). This list issued a challenge to wood preservative manufacturers and researchers to develop alternative wood preservative systems that can meet performance requirements.

The purpose of this research was to investigate combinations of wood protectants and VTC processing to create a stronger, more durable wood product. Western juniper oil was combined with commercial grade cinnamon oil and ethanol at three different concentrations to create multiple wood preservative systems. Wood samples were treated with a preservative, subjected to VTC processing, and then strength and decay resistance properties were assessed.

## 1.2 Objective

Explore the potential for using natural product extracts to enhance the durability of VTC processed hybrid poplar.

#### **1.3 Rationale and Significance**

The VTC process increases wood strength at a rate that is directly proportional to the degree of compression and could improve the utility of low density wood species for creating wood composites with high strength and stiffness values. The VTC manufacturing process could create value-added uses for low value, low quality wood species. Hybrid poplar (*Populus trichocarpa x Populus deltoides*), a plantation grown and relatively abundant tree in Oregon, is an example of such a species. The principle concept behind this research is to investigate uses for under-utilized Oregon tree species by creating wood products with increased durability and commercialization potential for Oregon manufacturers.

Western juniper (*Juniperus occidentalis*), a pest species growing abundantly in Oregon's eastern rangeland, contains heartwood known for its natural decay resistance. The antimicrobial and anti-termitic properties of *Juniperus* species have been extensively studied. Essential oils extracted from the heartwood of these species contain many compounds including cedrol, widdrol, and sesquiterpene alcohol (Adams, 1988). These compounds are strongly termiticidal and *Juniperus* essential oil has shown an ability to inhibit some, but not all species of fungi and bacteria (Clark, 1990). Extracts from cinnamon tree components, native to Taiwan, also offer opportunities for use as a preservative ingredient. Steam distilled cinnamon oil can be used in multiple applications. Active compounds in cinnamon oil include cinnamaldehyde, eugenol, and a-terpineol. These compounds caused complete mortality of termites (*Coptotermes formosanus*) after fourteen days of exposure (Chang and Cheng 2002). Cinnamon oil has another distinct advantage due to its use as a perfume and food additive (Burt, 2004). Cinnamon oil is also widely available and could easily be purchased as a wood preservative ingredient.

#### **CHAPTER 2 – LITERATURE REVIEW**

#### 2.1 Introduction

Humans have used wood to carve out an existence since before recorded history. John Perlin noted that wood has "...been the principle fuel and building material of almost every society for over five thousand years, from the Bronze Age until the middle of the nineteenth century" (Perlin 1989). Although it now competes alongside building materials like steel and concrete, wood remains a relevant raw material for a number of products. In 2009, the United Nations Economic Commission for Europe (UNECE) and the Food and Agriculture Organization of the United Nations (FAO) reported that annual U.S. consumption of sawnwood, woodbased panels, and paper/paperboard was 517 million cubic meters, while consumption over the entire UNECE region during that time was 1.2 billion cubic meters. As wood continues playing an integral role in society our "...expectations of the region's forests have never been so high" (Pepke 2010).

Society's dependence on wood along with other natural phenomena have had a significant impact on the availability of trees and their relative size. The FAO estimated that 8.3% of world forests were lost between 1990 and 2000 and 5.2% of the worlds forests were lost between 2000 to 2010 (FAO 2011). Bowyer et al. (2003) predicted that human population growth and declining timber resources in the developing nations will reduce per capita forest area by half by 2100 from 0.6 hectares to 0.3 hectares. Changing forest resources will require alternative approaches to wood utilization like composite and engineered wood products.

Wood composite describes "any wood material adhesive-bonded together." Composites combine the natural strength properties of wood with modern production technology to create resource efficient products with enhanced properties (FPL 1999, Bowyer et al. 2003). Wood composites serve in roles where traditional timber is no longer available and allow utilization of both high-grade and low-grade forest resources. Examples include glue-laminated timber, laminated veneer lumber, strandbased composites, and engineered I-joists. Wood composites may offer potential solutions for meeting future worldwide wood demand.

#### 2.2 Viscoelastic Thermal Compression

Most structural applications require wood to have a high density to meet performance needs. Many mechanical properties of wood are closely correlated to density (Bowyer 2003) and controlling it offers unique opportunities to utilize low density species for new applications. Viscoelastic thermal compression (VTC), a wood densification process developed by Kamke and Sizemore (2008), presents an opportunity to control wood density using combinations of heat, steam, and mechanical pressure. Densification results from heating lignin above its glass transition temperature, softening cell wall components, and using mechanical pressure to compress wood in a non-destructive manner (Kutnar & Sernek 2007). The resulting wood has an increased modulus of elasticity (MOE), modulus of rupture (MOR), and reduced hygroscopicity.

VTC treatment closely resembles hydrothermal treatment which has multiple effects on hemicellulose, cellulose, and lignin. Wood being VTC treated is first exposed to 170°C (typical). After heating, steam is introduced, which causes hemicelluloses within the cell wall to partially depolymerize and form acetic acid (Tjeerdsma et al. 1998). Acetic acid encourages further carbohydrate depolymerization while cellulose is unaffected due to the stability of its highly ordered crystalline configuration (Tjeerdsma & Militz 2005, Boonstra & Tjeerdsma 2006). High temperatures also soften lignin, increasing the number of hydroxyl groups on the aromatic rings (Tjeerdsma et al. 1998). Hemicellulose and lignin softening during steaming causes wood cell wall plasticization and allows for transverse compression without fracturing cell wall material (Kutnar & Sernek 2007, Wolcott et al 1990). Following steaming and compression, the products are cooled and "...recondensed into a modified polymeric complex" which "results in a further cross-linking of the lignin network" where "cellulose microfibrils are surrounded by a firm and more inelastic network" (Tjeerdsma & Militz 2005, Boonstra & Tjeerdsma 2006, Tjeerdsma et al. 1998). The process results in wood veneer with an increased crystalline structure, increased relative lignin percentage, reduced hydroscopicity, and a darkened color (Boonstra & Tjeerdsma 2006).

Previous research with VTC wood was conducted on a small scale, 50-liter sealed cylindrical reaction chamber capable of processing wood veneers approximately 8.5 cm wide and 14 cm long. The device used two internal platens to densify wood samples to either a desired thickness or density (O'Connor 2007).

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Recently, a second VTC prototype, capable of more precise temperature, pressure, and steaming control, was constructed at the Oregon State University Green Building Materials Laboratory. This VTC machine allows researchers to produce larger VTC wood specimens, 25 cm wide and 60 cm long, using computer controlled VTC densification schedules (Kamke & Rathi 2011).



Figure 1 - VTC densification chamber at the Oregon State University Green Building Materials Laboratory

#### 2.2.1 Benefits of Viscoelastic Thermal Compression

Previous research has investigated the gains in wood performance resulting from varying VTC densification processes. Kutnar et al. (2009) found that density profile change during the VTC process depended upon the extent of densification. Wood cell walls buckled without fracturing and the overall amount of void space within the woody material was reduced. This reduced void space contributed to the enhanced mechanical properties observed in VTC wood (Kutnar et al. 2009).

VTC processing enhances wood bending properties at rates that are roughly proportional to the degree of densification. Kutnar et al. (2008a) reported hybrid poplar (*Populus deltoides* x *Populus trichocarpa*) MOE and MOR increases as high as 129% and 102%, respectively, with a 132% increase in density. These findings support Kamke's (2006) previous VTC research findings where MOE and MOR of radiata pine (*Pinus radiata*) veneer increased by 174% and 116% respectively, with a 170% increase in density. O'Connor (2007) densified the semi-porous hardwoods eastern cottonwood (*Populus deltoides*) and sweetgum (*Liquidamber stiraciflua*), and found MOE increases as high as 223% and 224%, respectively, while MOR increased 184% and 154%, respectively.

VTC processing not only improves wood strength, it also produces materials with good adhesion properties that bond well with commercial adhesives. Kutnar (2008a) found that lap-shear bond performance of VTC wood, glued using phenol formaldehyde (PF) resins, was "comparable or better" to non-densified wood. This effect may be a result of processing, which creates a smooth surface on VTC wood and improves adhesive application efficiency (Kamke 2006). Kutnar (2008b) also detected hydrophobic behavior on VTC wood surfaces, observing that water contact angles increased over 20% on densified specimens. Reductions in surface free energy for VTC wood, when compared to non-densified samples, indicate that VTC processes also decrease wood polarity (Kutnar 2008b).

#### 2.2.2 Detrimental Effects of Viscoelastic Thermal Compression

Dimensional stability is an important aspect of wood product performance, particularly for wood composites. While VTC processing tends to make wood cell substance more hydrophobic, increased density also increases swelling potential (O'Connor 2007). Gabrielli and Kamke (2010) found that impregnating VTC wood with PF resin at concentrations as low as 5% significantly reduced thickness swelling. However, the increased dimensional stability resulting from resin impregnation also resulted in 49% to 77% declines in MOE values compared to non-impregnated VTC samples (Gabrielli & Kamke 2010).

In some cases, thermal modification in a manner similar to VTC processing has been reported to limit biological decay. Norway spruce (*Picea abies*) and beech (*Fagus sylvatica*) blocks subjected to thermo-hydro-mechanical (THM) densification at 180°C showed significant reductions in weight loss compared to controls when exposed to the brown rot fungi *Coniophora puteana*, *Gloeophyllum trabeum*, or *Postia placenta* and white rot species *Trametes versicolor* and *Trametes pubescens* for 16 weeks. Increased decay resistance was attributed to occlusion of cell lumina during densification that restricted fungal hyphae growth and penetration into the secondary walls of tracheids (Schwarze & Spycher 2005, Skyba et al 2009).

Kutnar et al. (2010) observed conflicting results and concluded that viscoelastic thermal compression of hybrid poplar did not significantly enhance durability against the white-rot fungi *Trametes versicolor* and *Pleurotus ostreatus*. Susceptibility to fungal decay was independent of degree of densification and increased concentrations of cell wall material resulted in higher mass losses (Kutnar et al. 2010). In addition, Lesar et al. (2012) found that thermo-hydro-mechanically treated hybrid poplar (*Populus deltoides* x *Populus trichocarpa*) and Douglas-fir (*Pseudotsuga menziesii*) veneers exposed to the brown rot fungus *A. vaillantii* and the white rot fungus *T. versicolor* for eight weeks performed no better in durability tests than non-densified controls (Lesar et al. 2012). While heat treatment might degrade the hemicelluloses that many fungi use in the early stages of attack, it is doubtful that these changes markedly alter decay resistance of highly decay susceptible species such as poplar.

#### 2.2.3 Potential for Viscoelastic Thermal Compressed Wood

VTC wood holds potential as an engineered wood composite component which can be assimilated into multiple wood products. Rathi (2009) found that incorporating 40% VTC processed hybrid poplar strands into oriented strand composites increased MOE by 30% and MOR by 18%. Interviews with forest products sector employees and design professionals suggested that VTC processed wood could be used in both structural and non-structural applications. Interviewees identified over twenty products that could benefit from incorporating VTC wood including LVL, plywood, concrete forms, flooring, and transportation components. Barriers to VTC utilization identified by interviewees often focused on the capital and operational costs of the manufacturing equipment required for the process (Macias 2010). However, the ability to control wood density makes it possible to utilize new species of wood not typically considered for structural building components.

#### 2.3 Hybrid Poplar

Hybrid poplar (*Populus trichocarpa* x *P. deltoides* and other varieties) plantations cover over 100,000 acres in an area from British Columbia to southern Oregon and Idaho (Revels 2009). Originally planted as windbreaks for fruit crops, hybrid poplar is now used for a range of applications including wastewater treatment and as a biomass feedstock (Revels 2009). Hybrid poplar plantations emerged in Oregon in the late 1970's as the paper industry tried to develop steady sources of pulpwood (Stanton et al. 2002). When demand for wood chips and pulp declined in the late 1990's, plantation management shifted towards sawlog production using rotation ages of nine to ten years (Carlson & Berger 1998). Plantation and hybridization researchers also proposed utilizing these trees as carbon sinks and biofuel sources (Revels 2009). However, another alternative could be utilizing hybrid poplar for enhanced engineered wood products.

Hybrid poplar is a low density, diffuse porous hardwood. Hybrid poplar MOE values are lower than natural grown poplar due to the presence of a higher percentage of juvenile wood, but MOE values become comparable to commercial softwoods when the strength to density ratio is considered (Balatinecz et al. 2010). De Boever et al. (2007) evaluated hybrid poplar (*Populus trichocarpa* x *P. deltoides*) clones Beaupre, Hazendans, and Hoogvorst and found MOE values ranging from 6.51 to 7.86 GPa. These values are roughly comparable to those for American sycamore (7.31 GPa) (FPL 1999). While it is unlikely that solid-sawn hybrid poplar will be used in structural applications, hybrid poplar glue joints perform well, creating opportunities for utilization in composites (Carlson & Berger 1998). Low density and availability have also allowed hybrid poplar to be incorporated as an oriented strand board (OSB) component (Kenney et al. 1990). Another potential application for hybrid poplar is VTC processing, a technique that increases wood density, flexural properties, and allows for hybrid poplar utilization as a component in engineered wood products (Kutnar et al. 2008a).

# 2.4 Biological Degradation

Wood products can be subjected to biological decay. Although some timber species are naturally durable, most species are prone to biological attack. Biological attack can be caused by many agents including insects, molds, and fungi.

#### 2.4.1 Termites

Subterranean termites are important agents of wood deterioration in temperate and tropical areas. Termites are problematic for wood buildings and their infestations are destructive and expensive to remove (Bowyer et al. 2003). In the U.S., over 500,000 dwelling units a year receive termite control treatments (Bowyer et al. 2003). Beal et al. (1986) estimated that the cost of termite control ranged between \$100 million to \$3.5 billion annually. Subterranean termites are the most common species in the U.S., living along the Eastern Seaboard (Florida to Virginia), the Gulf of Mexico, and California (Bowyer et al. 2003). Subterranean termites are also found in drier, temperate regions of Europe, South America, southern Africa, Australasia, and sections of Asia (Eaton & Hale 1993).

Subterranean termites include both lower termites (*Coptotermes, Mastotermes, Reticulitermes, Schedorhinotermes*) or higher termites (family: Termitidae; *Microcerotermes, Microtermes, Nasutitermes*). Following World War II, the Formosan termite (*Coptotermes formosan*), a species that is far more aggressive than indigenous termites, was introduced to the continental United States in wood shipped from Asia. Formosan termite populations are now well established in Hawaii, Louisiana, and Florida (Morrell et al. 2011).

Termites live within a caste system consisting of adults, soldiers, and workers. Workers make up 80 to 90% of the population and colonies may contain anywhere from "...a few thousand to millions of termites" (Eaton & Hale 1993, Lund 1973). Termites enter buildings through small cracks or holes and travel through selfconstructed tubes to reach food sources as far as 100 meters from their original nest. Colonies, which typically survive around 15 to 20 years, appear in high moisture soils, trees, and wood structures (Eaton & Hale 1993).

Termites ingest wood using their mandibles to tear off small chunks. While some wood species resist termite attack (e.g. baldcypress, redwood, and cedar), none are completely immune (Nicholas 1973). Sapwood of all species is highly susceptible to attack and must be treated to protect against termites (Eaton & Hale 1993). Many methods have been used to eradicate termite colonies including fumigation and wood treatments. Complete prevention requires a combination of soil treatment with careful construction practices that incorporate physical barriers and regular inspections (Morrell et al. 2011). While historical prevention methods like wood (creosote and CCA) and soil treatments (chlorpyrifos) can effectively limit termite attack, alternative approaches emphasizing more environmentally acceptable solutions are being investigated (Eaton & Hale 1993).

## 2.4.2 Staining and Mold Fungi

Stain and mold fungi are Ascomycetes and Deuteromycetes that are particularly problematic in freshly felled logs and recently sawn lumber. Stain and mold fungi are different from decay fungi because they do not degrade wood cell walls, but instead utilize sugars and starches found within the cell lumens (Bowyer et al. 2003). Although they do not significantly reduce wood strength, stain and mold fungi are troublesome because they discolor wood surfaces and cause significant losses in commercial quality. Common mold genera in temperate regions include *Aspergillus*, *Trichoderma*, and *Penicillium*, while *Ophiostoma* and *Alternaria* are common stain fungi (Eaton & Hale 1993).

Blue stain, also called sap stain, is an economically important discoloration of the wood. Infection by blue stain spores and hyphae often comes from bark damage during felling or from transmission by insects on exposed wood surfaces (Eaton & Hale 1993). The bluish black to steel gray color results from a buildup of dark colored fungal hyphae that penetrate deeply into the sapwood. Stain fungi hyphae can increase wood permeability, but rarely influence wood strength other than moderately reducing toughness (Nicholas 1973). Certain U.S. tree species like Douglas-fir, most pines, and sweetgum are especially susceptible to staining fungi.

Molds behave much like blue stain in attacking wood. Molds affect wood appearance and permeability. Molds appear to be more tolerant of unfavorable conditions and some even have the ability to degrade toxic chemicals (Nicholas 1973). Mold is particularly problematic for veneer and lumber producers in warm and humid environments (Eaton & Hale 1993).

Preventing staining fungi requires "...immediate removal of logs from the forest followed by kiln drying or rapid air drying" (Eaton & Hale 1993). Techniques for preventing mold in wood include complete submersion under water, using sprinklers to limit oxygen, and application of fungicides. Keeping stored wood dry and moving freshly harvested wood from the forest as quickly as possible are helpful (Tsoumis 1991). Careful control of timber yard hygiene can also limit fungus and insect attack (Findlay 1962). However, preventing wood from staining fungi attack is not adequate, since decay fungi also threaten wood products while in service and must be considered.

# 2.4.3 Brown Rot Fungi

Fungi are the most economically detrimental agents of deterioration, destroying wood cells and discoloring the wood surface (Nicholas 1973). Brown-rot fungi are Basidiomycetes that primarily attack carbohydrates in both hardwoods and softwoods. These fungi rapidly depolymerize cellulose using enzymes secreted by hyphae that penetrate pit membranes or bore through cell walls while metabolizing structural polysaccharides. Wood strength decreases significantly after only slight amounts of weight loss (Eaton & Hale 1993). The resulting decayed wood is a "crumbly residue that is composed primarily of lignin" (Eriksson et al. 1990).

Protecting wood from brown-rot fungi is essential to prolonging service life. This generally means limiting one of the four factors needed for successful growth - a food source, presence of free water, oxygen, and moderate temperatures. Wood acts as the food source while fungal growth thrives at moisture contents between 40% to 80% and temperatures between 21°C and 32°C (Haygreen & Bowyer 1989). Brownrotted wood is discolored and has decreased density, increased hygroscopicity, and significantly reduced bending properties (MOR and MOE) depending upon the wood species, fungal species, and environmental conditions (Tsoumis 1991). Although many methods have historically been used to quantify the degree of fungal attack, mass loss due to decay remains the principle method for qualifying fungal degradation (Hartley 1958).

A common brown-rot fungus, *Gloeophyllum trabeum*, is present in Australia, North America, New Zealand, Europe, and southern Africa. Identified by the reddish brown to dark brown color and deep cracking that appears late in the decay process, this fungus grows at a rate of 6 to 11.4 mm per day. Optimum fungal growth occurs when wood moisture content falls between 30% to 50% (although capable of living in a range of moisture contents) and temperatures around 35°C. The fungus appears in many common situations such as felled logs, domestic timbers in ground/mortar contact, window joinery, roofing timbers, bridge timbers, wooden boats, and wood composites (Eaton & Hale 1993). Ideally, wood should always be kept dry to protect against decay fungi, although this is not always possible.

# **2.5 Wood Preservatives**

In order to combat biological degredation on wood, humans have developed preservatives that prolong product service life. Treated wood products can last anywhere from 20 to 40 times longer than their untreated counterparts (Townsend & Solo-Gabriele 2006). Historically, wood preservation was a critical component of country trade and warfare. Even Noah was instructed in Genesis 6:14 to "pitch the ark within and without" (Zinkel & Russell 1989). The current preservative industry bloomed in the 1830s when creosote and the full cell (Bethel) treatment process were patented. Creosote, which contains more than 200 chemical compounds, has remained an effective treatment that controls fungi, termites, and marine borers. Preservatives have continued to evolve based on end use requirements and include first generation systems (creosote, chromated copper arsenate, ammoniacal copper zinc arsenate), non-arsenical copper-based systems (alkaline copper quaternary, ciooer azole), copper azole), borates (disodium octaborate tetrahydrate, zinc borate), organic biocides, and non-biocidal additives (water repellants, waxes) (Morrell et al. 2011).

Over the past fifteen years, many changes have taken place in the preservative industry. Public perception about bioactive chemical use is changing and these perceptions influence government policy and consumer acceptance. Some European countries have gone as far to encourage organic only preservative systems for residential applications (Townsend & Solo-Gabriele 2006). Alternatives to traditional wood preservatives have included chemical modification, thermal modification, and essential oils with biological activity (Hill 2006, Yang & Clausen 2007). New preservative systems must be evaluated on the basis of cost, performance, and environmental impacts during and at the end of service life (Hill 2006). One approach to wood protection incorporates essential oils as wood preservatives. These oils have been studied extensively for pharmaceutical, agricultural, and food preservative applications.

## 2.5.1 Essential Oils

Essential oils, also known as volatile oils, are defined as "...aromatic oil liquids obtained from plant material (flowers, buds, seeds, leaves, twigs, bark, herbs,

wood, fruits and roots)" derived by fermentation, expression, enfleurage, or steam distillation methods (Van de Braak & Leijten 1999). Essential oils from plants like rosemary, eucalyptus, and lemongrass have been used to create insecticides, food preservatives, flavorings, and pharmaceuticals (Isman 2006, Burt 2004, Yang & Clausen 2007). Extraction processes using steam distillation originated in Egypt, India, and Persia. The Greek historians Herodotus, Pliny, and Dioscorides were among the first to reference distillation as a process (although vaguely), mentioning turpentine over 2000 years ago (Guenther 1948). Some essential oils contain more than 60 chemical components that vary in composition depending upon oil type, harvest location, and growing season (Burt 2004). Chemical components are often identified using gas chromatography and mass spectrometry (GCMS) and commercially interesting essential oils have been studied extensively for their major active components (Burt 2004).

Essential oils can also be derived from trees. The heartwoods of decay resistant species like white oak, black locust, osage orange, teak, and baldcypress contain phenolic compounds that can act in a synergistic manner as natural preservatives (Nicholas 1973). Eastern redcedar (*Juniperus virginiana*), incense cedar (*Calocedrus decurrens*), Port Orford cedar (*Chamaecyparis lawsoniana*), and many tropical hardwoods have all been investigated for their essential oil compositions or antifungal activities (Mun & Prewitt 2011, Veluthoor et al. 2011, Wang et al. 2012, Kawamura et al. 2011). Two plants that have potentially antitermitic and antifungal properties are western juniper and the cinnamon tree.

#### 2.5.2 Juniper Oil

Western juniper (*Juniperus occidentalis*) is a fire intolerant species that occupies 3.7 million acres in the eastern and central regions of Oregon. The tree is an encroaching species capable of altering hydrologic cycles and reducing biological diversity. Management practices promoting juniper removal encourage the return of native vegetation, increased soil water holding capacity, and increased plant diversity. Two principle methods are used to control invasive western juniper stands: prescribed burning and mechanical removal (Barrett 2007). Woody material left over from mechanical removal can potentially be utilized for value-added products like essential oils extracted from juniper leaves and heartwood.

Investigations into value-added uses for western juniper have been carried out since the 1950s. Steam distillation of juniper chips yielded 0.9% - 1.25% juniper oil (dry-weight basis) with the oil being comprised mostly of cedrol and cedrene (Kurth & Ross 1954). Adams (1987a) used GCMS analysis to identify additional components including  $\alpha$ -cedrene (8.8%),  $\beta$ -cedrene (2.6%), thujopsene (18.9%), cuparene (1.5%), cedrol (38.9%), and widdrol (1.6%). Adams (1987b) also experimented with hexane and methanol extraction techniques, and found that juniper foliage was rich in phytochemicals. He found that oil yields peaked in late fall and early winter but phytochemical variations due to seasonality were not significant.

The compounds extracted from juniper oil have been identified as having both antitermitic and antifungal properties. Essential oil extracted from *Juniperus occidentalis* heartwood/sapwood resulted in 100% termite mortality within

approximately one week (Adams et al. 1988). Further studies into fungal and bacterial resistance indicate that the essential juniper oil inhibits some, but not all species. Antifungal and antitermitic activity varied with the extraction technique as well as from where the oil was extracted (i.e. bark, sapwood, or foliage). Hexane extracted oil performed well against the fungus species *Cryptococcus neoformans* but was ineffective against *Trichophyton mentagrophytes* (Clark & McChesney 1990).

In order to be used as a wood preservative, juniper oil must have no adverse effects on human health. While animal testing of undiluted western juniper oil has found it to be a moderate irritant, rabbits displayed barely perceptible or no irritation at concentrations of 5% and 0.5%, respectively (Craig et al. 2004). Pilot studies using western juniper shavings as animal bedding found no evidence of clinical or biochemical abnormalities on animal subjects, indicating that shavings did not result in hypersensitive responses (Blythe et al. 2001).

#### 2.5.3 Cinnamon Oil

Cinnamon (*Cinnamomum osmophloeum*) is a durable hardwood that is native to Taiwan (Wang et al. 2005). Essential oils extracted by steam distilling foliage or heartwood chips have both fungal and termite inhibitory effects. The principle active oil ingredients are cinnamaldehyde and eugenol. Antifungal assays indicate that these compounds have an antifungal index of 100% against brown-rot (*Laetiporus sulphureus*) and between 70% - 100% for white rot (*Coriolus versicolor*) (Wang et al. 2005). Cinnamaldehyde, eugenol, and  $\alpha$ -terpineol produced 100% termite mortality against *C. formosanus* after 14 days in a no-choice bioassay (Chang & Cheng 2002). These laboratory results suggest that cinnamon leaf oil could be employed to prevent biodegradation.

While cinnamon oil has been shown to protect against some brown-rot and white rot fungi, it must protect wood against the variety of biological agents it will encounter during its service life. Cinnamon oil could be combined with other ingredients such as additional essential oils, antioxidants, or metal chelators to create a synergistic effect (Green & Shultz 2003). Synergistic effects between cinnamaldehyde and eugenol or octyl gallate produced complete fungal inhibition of *G. trabeum, Lenzites betulina*, and *Laetiporus sulphureus* (Yen et. al. 2008, Hsu et al. 2007). Therefore, combining juniper and cinnamon essential oils may create opportunities for their use as a novel preservative system.

# 2.6 Conclusion

Diminishing timber supplies and changing attitudes towards wood preservation will challenge producers to make wood products last longer, boast better performance attributes, and reduce environmental impacts during and after product service life. Wood modification could help answer this challenge. VTC processing offers unique opportunities to utilize low density species for new applications. One challenge for VTC wood is decay resistance. Essential oil preservative systems that do not cause significant swelling could be used to treat VTC wood. Preliminary laboratory tests suggest that some oils have the ability to protect against fungi and insects. However, it is unclear whether these materials will perform against a broad range of biological agents on a material with such inherently low durability. The following research examined the use of natural plant extracts in conjunction with VTC processing to create a stronger, more durable wood product while utilizing plantation grown timber.

### **CHAPTER 3 – MATERIALS AND METHODS**

#### 3.1 Materials

Hybrid poplar (*Populus trichocapra* x *Populus deltoides*) was harvested in Boardman, OR and peeled into veneer by Columbia Forest Products (Klamath Falls, OR). Veneer sheets approximately 3.5 mm thick were stored at 20°C and 65% relative humidity until needed.

Timbor $(Na_2B_8O_{13}\cdot 4H_2O)$ , Rio Tinto Minerals, CA), an industrial borate wood treatment powder was used to formulate a 5% (boric acid equivalent) by weight solution in water.

Cinnamon leaf oil was purchased from OliveNation LLC (Charlestown, MA) and stored at 2°C until needed.

Western juniper foliage (*Juniperus occidentalis*) was collected from two sites within the Deschutes National Forest approximately one mile from Sisters, OR. Bough material consisting of leaves, berries, and small branches (less than 15 mm in diameter) was collected from the bottom 1/3<sup>rd</sup> of the tree using loppers, stored in a burlap sack, and transported to Corvallis, OR. Foliage was stored at 20°C and 65% relative humidity until steam distillation.

## **3.2. Experimental Design**

Hybrid poplar veneers were allocated to one of seven preservative treatments. Half of specimens receiving preservative treatments, approximately 380, were subjected to viscoelastic thermal compression (VTC) following preservative application. Samples were then assigned to one of three durability tests; a termite resistance test, a mold box test, and a brown-rot exposure bending test. The project's experimental design, along with the number of specimens (n) assigned to each test, is show in Table 1.

|  |                         | Termite<br>Test | Mold<br>Box | Decay<br>Bending |
|--|-------------------------|-----------------|-------------|------------------|
|  |                         | n               | n           | n                |
| No Preservative                        | Hybrid Poplar Veneer    | 5               | 10          | 20               |
|  | VTC Pressed H.P. Veneer | 5               | 10          | 20               |
| Timbor® Preservative                   | Hybrid Poplar Veneer    | 5               | 10          | 20               |
|  | VTC Pressed H.P. Veneer | 5               | 10          | 20               |
| 10% Cinnamon Oil                       | Hybrid Poplar Veneer    | 5               | 10          | 20               |
| Preservative                           | VTC Pressed H.P. Veneer | 5               | 10          | 20               |
| 10% Juniper Oil Preservative           | Hybrid Poplar Veneer    | 5               | 10          | 20               |
|  | VTC Pressed H.P. Veneer | 5               | 10          | 20               |
| 2.5% Juniper Oil/Cinnamon              | Hybrid Poplar Veneer    | 5               | 10          | 20               |
| Oil Preservative                       | VTC Pressed H.P. Veneer | 5               | 10          | 20               |
| 5% Juniper Oil/Cinnamon Oil            | Hybrid Poplar Veneer    | 5               | 10          | 20               |
| Preservative                           | VTC Pressed H.P. Veneer | 5               | 10          | 20               |
| 10% Juniper Oil/Cinnamon               | Hybrid Poplar Veneer    | 5               | 10          | 20               |
| Oil Preservative                       | VTC Pressed H.P. Veneer | 5               | 10          | 20               |
| 10% Juniper Oil/Cinnamon               | Hybrid Poplar Veneer    | 5               | 10          | 20               |
| Oil Preservative (post VTC processing) | VTC Pressed H.P. Veneer | 5               | 10          | 20               |

Table 1 - Experimental design with treatment group replication

# **3.3 Sample Fabrication and Testing**

Hybrid poplar veneer sheets were machined to 3.5 mm x 26 mm x 140 mm (radial x tangential x longitudinal). Specimens containing noticeable defects such as knots, splits, or extreme angle grain deviation were removed. Following fabrication,

specimens were oven dried at  $103^{\circ}$ C for 24 hours, weighed, and their dimensions were measured (nearest – 0.01 mm).

Specimens were subjected to non-destructive, three point bending tests according to ASTM Standard D4761-05 (ASTM 2010) using a Sintech MTS machine equipped with TestWorks II v2.11f software and an Omega LCCB-300lb load cell. Specimens were positioned (flatwise) along a 100mm span and center-span loaded perpendicular to grain up to 75 Newtons at a load rate of 5 mm/minute (Figure 2). Specimen beam dimensions, load and displacement measurements were used to create individual stress/strain curves. Following bending, the TestWorks software reported peak load (N), peak stress (MPa), and MOE (MPa) for individual specimens.

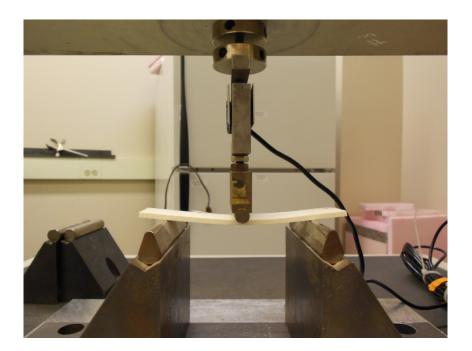


Figure 2 – Three-point non-destructive bending test used to evaluate hybrid poplar veneer modulus of elasticity

## **3.4 Preservative Formulation and Application**

#### 3.4.1 Western Juniper Oil Formulation

Recently cut western juniper bough material was weighed and steam distilled shortly after collection. Approximately 2,500 g was placed inside a distillation column and steam distilled for five hours (Figure 3). The resulting oil and water mixture was collected from the distillation column condenser and drained into a beaker. Water was removed from the resulting water/oil mixture by decanting. The resulting oil was weighed to determine distillation yield on a wet weight basis and stored at -2°C. A total of thirteen distillations were performed yielding approximately 200 g of western juniper oil. Seventy grams of oil were obtained from boughs collected in January; the remaining 130 g was obtained from boughs collected in June. Oils from separate distillations were mixed together for use.



Figure 3 - Steam distillation device used to collect western juniper oil

### 3.4.2 Timbor® Treatment Formulation

A boron preservative treatment was prepared by mixing Timbor® Industrial powder with warm water by weight to create a 5% (boric acid equivalent) concentration of Timbor® preservative.

# 3.4.3 Essential Oil Formulation

Preservative treatments were prepared by mixing essential oils with 95% ethanol by weight. Ethanol was chosen as the oil solvent due to its ability to rapidly evaporate. Solutions were prepared containing 10% cinnamon oil, 10% juniper oil, 1.25% each of juniper and cinnamon oil, 2.5% each of juniper and cinnamon oil, and 5% each of juniper and cinnamon oil.

### 3.4.4 Preservative Application

Oven-dry hybrid poplar veneers were submerged in one of the above mentioned borate or essential oil treatments within a desiccator. A vacuum was drawn for fifteen minutes, than released. Following treatment, specimens were removed from the preservative treatment, blotted dry, weighed to determine treatment uptake, and oven-dried at 50°C for 24 hours. The samples were then weighed.

# 3.5 Viscoelastic Thermal Compression and Bending Tests

Following preservative application, veneer samples were placed between two stainless-steel platens heated to 170°C inside the VTC processing machine's

pressurized chamber (Table 2). Specimens from multiple treatment groups were processed together in an effort to eliminate any batch effect that might result from VTC pressing.

Steam was introduced into the densification chamber at 669 kPa (gage) for 30 seconds. The platens were then separated to allow steam exposure to specimens for an additional 90 seconds. Steam was released from the chamber for a period of 30 seconds. Specimens were then compressed radially to platen position 2.03 mm at 170°C. Specimen compression took place for 180 seconds; a cooling stage then reduced platen temperatures to 93°C over 120 seconds. After cooling, platen compression pressure was released and platens opened. The densification chamber was then opened and specimens were removed. The dimensions and weight of each specimen were measured before and after VTC treatment.

| Step # | Step Name                 | Step Duration Internal Platen Temperature (C |        |     |  |  |  |
|--------|---------------------------|--|--------|-----|--|--|--|
|        |                           | (seconds)                                    | Bottom | Тор |  |  |  |
| 1      | Positioning               | 10   | 170    | 170 |  |  |  |
| 2      | Steam Inlet               | 30   | 170    | 170 |  |  |  |
| 3      | Steaming (no compression) | 90   | 170    | 170 |  |  |  |
| 4      | Steam Release             | 30   | 170    | 170 |  |  |  |
| 5      | Compression               | 180  | 170    | 170 |  |  |  |
| 6      | Cooling                   | 120  | 93     | 93  |  |  |  |
| 7      | Compression Release       | 5  | 93     | 93  |  |  |  |
| 8      | Open Chamber              | 5  | 170    | 170 |  |  |  |

Table 2 - Viscoelastic thermal compression press schedule

The VTC schedule was chosen because it used lower processing temperatures but still produced materials with suitable properties. Following VTC treatment, veneers were oven-dried at 50°C for 24 hours, reweighed, and dimensions recorded.

The bending properties of VTC processed hybrid poplar veneer were assessed by subjecting each veneer to the non-destructive bending tests previously mentioned using the same span to depth ratios. Test span was shortened from 100 mm to 33 mm to keep a consistent span to depth ratio for VTC samples. Maximum load, maximum stress, and MOE were recorded. Increases in specimen density as a result of VTC treatment were calculated using oven-dry weights and volumes.

## **3.6 Post Treatment Preservative Application**

In one experiment, samples were treated with a preservative following VTC processing. In these cases, a 10% essential oil treatment composed of 5% cinnamon oil, 5% juniper oil, and 90% toluene was formulated by weight basis. This preservative system was applied using previously described methods. Veneers were oven-dried for 24 hours at 50°C, submerged in the preservative system, and placed inside a desiccator where a vacuum was drawn for 15 minutes. The vacuum was released and the samples were weighed to determine treatment uptake. The specimens were oven-dried at 50°C for 24 hours and weighed.

## **3.7 GC-MS Analysis**

The retention of essential oil in specimens was determined by extraction and analysis by gas chromatography-mass spectrometry. Veneer specimens were reduced to particles using a Wiley mill until they passed through a screen with 1 mm openings. One gram of the resulting dust was placed in a vial with 25 ml of methanol, sonicated for three hours in a Fischer Scientific Solid State/Ultrasonic FS-14, and then allowed to stand for 48 hours. Additional wood samples were extracted in the same manner using hexane as a nonpolar solvent. After extraction, the samples were allowed to settle before analysis.

The extracts were injected in a Shimadzu GCMS-QP2010S with a 30 m long 5HRXI-MS carbowax column (0.25 mm ID diameter). Injector temperature was 275°C. Oven temperature was raised from 50°C to 260°C at a rate of 2°C per minute with helium as the carrier gas at 30 ml/minute. Column flow was 1 ml/min with a linear velocity of 36.3 cm/sec. Mass spectrometry scanned between 20 and 400 m/z. Compound identification was completed using the NIST08 Mass Spectral Library. Peak integration was used to compare compound abundance across multiple treatment groups.

### **3.8 Decay Assessments**

The durability of VTC hybrid poplar, with and without essential oils, was assessed against Formosan termites, mold fungi, and a brown-rot decay fungus.

## 3.8.1 Formosan Termite Test

Resistance to termites was evaluated following a modification of AWPA Standard E21-06 (AWPA 2010). The standard surrounds test blocks with untreated pure sapwood as feeder material on concrete blocks just above the soil. The assembly is covered to prevent wetting of the specimens. Since test specimens were smaller and thinner than the standard specified, they were instead sandwiched between two sheets of 9 mm thick Douglas-fir plywood at a test site in Hilo, Hawaii. Five samples from each treatment group were glued to the face of one of the Douglas-fir plywood panels (0.6 m x 0.6 m) using an Elmer's glue stick to secure samples in the array during shipping. Numbered metal tags were also attached to each specimen for identification (Figure 4). The second sheet of plywood was laid on top of the array, which sandwiched specimens into place, and the plywood boards were secured together using nylon nuts and bolts.



Figure 4 - Formosan termite test array with VTC and non-VTC processed veneers attached

Feeder stakes were driven into the ground in the spaces between the concrete blocks. The board was placed on the blocks and covered as per the AWPA E21-06 standard. Termites had access to all samples. Following six months exposure, plywood boards were opened up and Formosan termite damage was assessed. Specimens were examined and visually rated using the following rating system: 10 - sound surface nibbles permitted, 9 - light attack, 7 - moderate attack and penetration, 4 - heavy, 0 - failure. Samples were also oven-dried and weighed to determine mass loss.

| 5   | 54         | 60  | 24  | control     |    |          |
|-----|------------|-----|-----|-------------|----|----------|
| 96  | 38         | 81  | 148 | VTC control |    |          |
| 64  | 48         | 62  | 90  | boron       |    |          |
| 77  | 50         | 147 | 74  | boron VTC   |    |          |
| 7   | 882        | 76  | 87  | C.O.        |    |          |
| 878 | 875        | 69  | 880 | C.O. VTC    |    |          |
| 95  | 149        | 82  | 59  | J.O.        |    |          |
| 93  | 92         | 88  | 61  | J.O. VTC    |    |          |
| 56  | 42         | 85  |     | 2.50%       |    |          |
| 7   | 78         |     | 78  |             | .3 | 2.5% VTC |
| 52  | 36         | 63  | 97  | 5%          |    |          |
| 14  | 100        | 80  | 79  | 5% VTC      |    |          |
| 26  | 9          | 877 | 68  | 10%         |    |          |
| 57  | 53         | 66  | 873 | 10% VTC     |    |          |
| 40  | 73         | 72  | 879 | 10% Post    |    |          |
| 70  | 47         | 87  | 86  |             |    |          |
| 84  | 65         | 83  | 876 |             |    |          |
| 874 | 34         | 55  | 58  |             |    |          |
| 13  | 49         | 67  | 71  |             |    |          |
| 7   | <b>′</b> 5 | 881 | 91  |             |    |          |

Figure 5 - Formosan termite test array specimen map

### 3.8.2 Mold Box Test

Sample resistance to mold growth was evaluated following AWPA Standard E24-06 (AWPA 2010). A mold box environmental chamber allowing for temperature and relative humidity regulation was constructed to hold ten randomly assigned hybrid poplar samples from each experimental group. A layer of soil approximately 50 mm thick was evenly distributed over a mesh screen which rested over 90 mm of circulating water heated to 25<sup>o</sup>C. After soil addition, the box was conditioned for 48 hours before inoculating with a water/mold-spore suspension.

The mold spore suspension was created using pure cultures of *Trichoderma spp.* (Pers.:Fr), *Aspergillus niger* (Tiegh.), *Penicillium citrinum* (Thom), and *Alternaria alternata* ((Fr.:Fr.) Keissl.). The strains were inoculated onto 1.5% malt extract agar in petri plates and incubated at 28<sup>o</sup>C until spores formed. A minimum of four plates were prepared for each fungus for inoculation suspension preparation.

After approximately two weeks, sterile deionized water was added to each plate and the surface was rubbed to dislodge spores and hyphae fragments. The water from the petri dishes was decanted into a container to produce approximately 1,800 ml of solution. Three drops of Tween 80® was added to reduce water surface tension and help disperse spores. Then the solution was diluted to 2,800 ml with sterile distilled water.

The total amount of water added to the soil was determined using the soil water holding capacity equation found in AWPA Standard E10-06. The soil required approximately 2,800 ml of water to reach the required moisture content and water

holding capacity. The spore/hyphae suspension was evenly poured over the soil surface. After soil inoculation, two petri dishes containing 1.5% malt extract agar were left in the box for 24 hours. The petri dishes were then removed and incubated for two weeks to determine if viable fungal spores from the introduced species were present.

Eight holding bars were used to hang samples 8 cm above the soil surface. A total of 166 specimens were placed in the box with at least 2 cm between faces and 4 cm from the box walls (Figure 5). The tangential surfaces of each specimen were visually evaluated for degree of mold attack according to AWPA E24-06 grading procedures every two weeks over eight weeks. Rating descriptions are given in Table 3.



Figure 6 - Hybrid poplar veneer specimens placement within a mold box

Table 3 - AWPA Standard E24-06 mold growth assessment rating system

| Rating | Description  |
|--------|--|
| 0      | No visible mold growth   |
| 1      | Mold covering up to 10% of surfaces providing growth is not so intense   |
|        | or colored as to obscure the sample color over more than 5% of surfaces  |
| 2      | Mold covering between 10% and 30% of surfaces providing growth is not    |
|        | so intense or colored as to obscure the sample color on more than 10% of |
|        | surfaces   |
| 3      | Mold covering between 30% and 70% of surfaces providing growth is not    |
|        | so intense or colored as to obscure the sample color on more than 30% of |
|        | surfaces   |
| 4      | Mold on greater than 70% of surfaces providing growth is not so intense  |
|        | or colored as to obscure the sample color on more than 70% of surfaces   |
| 5      | Mold on 100% of surfaces or with less than 100% coverage and with        |
|        | intense or colored growth obscuring greater than 70% of the sample color |

# 3.8.3 Bending Test

The effects of VTC treatment, with and without essential oils, on resistance to fungal decay were assessed by measuring changes in flexural properties during exposure to a brown rot fungus. A brown rot fungal suspension was prepared. Hybrid poplar veneers were inoculated with the suspension in specially prepared and sealed environmental chambers. Finally, bending tests were performed every two weeks to asses specimen bending properties.

Fungal inoculum was prepared by adding 4mm disks, cut from a culture of *Gloeophyllum trabeum* (Pers.:Fr.) Murrill (Isolate: MAD617), into a flask containing 100 ml of 1.5% malt extract. Flasks were incubated for twenty days in stationary culture at 28°C and mycelium was collected by filtration, rinsed with 300 ml of sterile distilled water, and macerated by blending to break up individual hyphae. The

resulting suspension was refrigerated at 5°C until needed. All glassware was sterilized prior to use.

Autoclavable bags with breathable patches were filled with 100 g of vermiculite and 100 ml of deionized water and then autoclaved for 45 minutes at 121°C.

Following sterilization, 10 hybrid poplar veneer specimens from a given treatment were added to a given bag. The bags were closed using rubber-bands and incubated at 20°C and 65% relative humidity for two weeks to equilibrate. Time to equilibrate was required to allow specimens to reach a common moisture content and allow for specimen swelling before the bending tests.

Following the conditioning period, the bags were unsealed and samples were removed for inoculation. Specimens were inoculated by dripping 1 ml of the *G*. *trabeum* hyphae suspension onto the endgrain surface. The bags were resealed with the rubber bands and incubated at 20°C and 65% relative humidity.

Changes in sample bending properties were assessed every two weeks using the methods previously described. However, some specific modifications were made for this test. Bending tests were performed inside of environmental bags that were 0.1 mm thick. Using calipers, specimen measurements were made from outside the bag to account for bag thickness (nearest – 0.01 mm). Span-to-depth ratios between VTC processed and non-VTC processed samples were not the same because the wetted specimens had expanded. Instead, bending properties were compared using bending stiffness (EI) values as opposed to MOE values. This allowed for stiffness comparisons among samples of different sizes. EI is defined as the MOE multiplied by specimen moment of inertia (I). I, for a rectangular cross-section, is defined by:

$$EI = MOE \ x \ \frac{width \ x \ depth^3}{12}$$

Bending properties were assessed over eight weeks following sample conditioning and inoculation with *G. trabeum*. Comparisons of EI data between treatment groups were made using Microsoft Excel's single factor ANOVA and twosample t-tests assuming unequal variances.

## **CHAPTER 4 – RESULTS AND DISCUSSION**

## 4.1 Hybrid Poplar Veneer Properties

Hybrid poplar veneer specimens used in this experiment had weight, density,

and MOE values that were consistent with previous studies (Figures 6-8, Table 4).

Kutnar et al. (2008a) reported oven-dry hybrid poplar density as 330 kg/m<sup>3</sup> with a

MOE of 8,200 MPa. Average test specimen density in this experiment was 330 kg/m<sup>3</sup>

with a MOE of 7,561.3 MPa.

Table 4 - Properties of hybrid poplar veneer specimens used to evaluate the effects of VTC treatment and natural oil extracts on resistance to biological attack

| Property            | Mean   | <b>Standard Deviation</b> |
|---------------------|--------|---------------------------|
| Width (mm)          | 26.34  | 0.23                      |
| Length (mm)         | 140.88 | 1.54                      |
| Depth (mm)          | 3.55   | 0.15                      |
| Oven-dry Weight (g) | 4.30   | 0.39                      |
| Density $(g/cm^3)$  | 0.33   | 0.03                      |
| MOE (MPa)           | 7561.3 | 1204.7                    |
| $EI (Pa \cdot m^4)$ | 0.7204 | 0.0801                    |

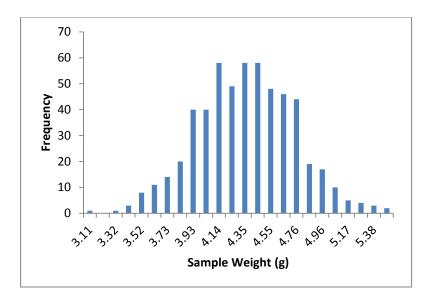


Figure 7 - Distribution of sample weight in hybrid poplar veneers used to evaluate the effects of VTC treatment and natural oil extracts on resistance to biological attack

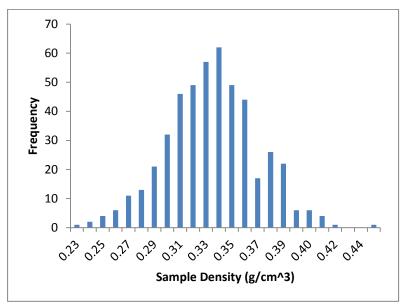


Figure 8 - Distribution of density in hybrid poplar veneers used to evaluate the effects of VTC treatment and natural oil extracts on resistance to biological attack

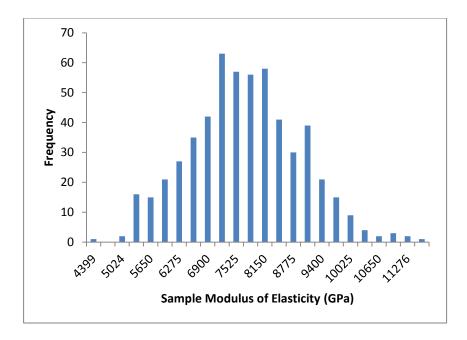


Figure 9 - Distribution of modulus of elasticity in hybrid poplar veneers used to evaluate the effects of VTC treatment and natural oil extracts on resistance to biological attack

# 4.2 Juniper Oil Distillations

Juniper oil steam distillations were performed in January and June of 2011.

Moisture content of juniper foliage at time of collection averaged 45.7%.

Approximately 2,500 grams of bough materials was distilled for five hours during

each run. Each distillation produced about 16.2 grams of oil for a total of 194.7

grams. Wet weight foliage yields for materials collected in January and June were

0.79% and 0.64%, respectively (Figure 9).

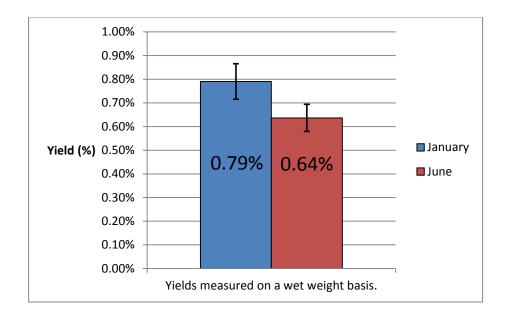


Figure 10 - Western juniper foliage oil yields (wet weight basis)

The low yields observed in steam distillations were expected. Adams (1987b) reported steam distillation yields from juniper heartwood chips for two varieties of *J. occidentalis* of 1.43% and 1.86%. There was also suggestive, yet inconclusive evidence that yields from juniper foliage, collected in January and June, differed statistically (two-sample t-test assuming unequal variances, p = 0.049). Although time of foliage collection may play a role in distillation yields, Adams (1987b) concluded that "…seasonal variation in the yields of phytochemicals from the leaves of *Juniperus* would not appear to present a major problem in their utilization."

#### 4.3 Preservative Retention

A total of 560 specimens were assigned to the seven preservative treatments, with 80 specimens allocated to each particular treatment. Preservative retentions were noticeably larger for specimens treated with cinnamon oil (Table 5). The higher retentions associated with cinnamon oil treatment may correspond to the strong performance of these specimens during mold box testing, but did not appear to affect resistance to termite attack.

| Treatment:   | Weight Gain (g):    |
|--|---------------------|
| Timbor   | 7.30 <u>+</u> 1.62  |
| 10% cinnamon oil                                     | 18.42 <u>+</u> 2.27 |
| 10% juniper oil                                      | 6.46 <u>+</u> 1.07  |
| 2.5% essential oil combination                       | 3.33 <u>+</u> 0.67  |
| 5% essential oil combination                         | 4.72 <u>+</u> 0.68  |
| 10% essential oil combination                        | 8.30 <u>+</u> 1.10  |
| 10% essential oil combination<br>post VTC processing | no retention        |

Table 5 - Sample weight gains of hybrid poplar veneers due to preservative treatment

Preservative uptakes for post VTC treated specimens tended to be low. VTC processed wood undergoes a heat treatment that tends to "seal" the cell lumens, limiting flow of free water through cells (Skyba & Spycher 2005). Despite vacuum treatment, the near elimination of cell voids by VTC treatment may reduce wood hygroscopicity and limit the ability of a preservative system to penetrate into wood cells (O'Connor 2007). The poor performance of specimens in this treatment group during durability tests likely reflected the limited uptake.

### 4.4. VTC Processing

Hybrid poplar veneers used in this experiment went from an initial density of  $330 \text{ kg/m}^3$  and an average MOE of 7,512.3 MPa to  $980 \text{ kg/m}^3$  and a MOE increase of 260% to 27,303.9 MPa following VTC processing. Specimen dimensions increased 0.8 mm (3%) in the transverse direction while radial thickness was reduced 68% from 3.53 mm to 1.14 mm. Differences in the longitudinal direction were negligible (Table 6).

| Property            | Hybrid poplar veneer |           | VTC Processed hybrid poplar |                    |  |
|---------------------|----------------------|-----------|-----------------------------|--------------------|--|
|                     |                      |           | veneer                      |                    |  |
|                     | Mean                 | Standard  | Mean                        | Standard Deviation |  |
|                     |                      | Deviation |                             |                    |  |
| Width (mm)          | 26.34                | 0.23      | 27.16                       | 0.24               |  |
| Length (mm)         | 140.88               | 1.54      | 140.82                      | 0.25               |  |
| Depth (mm)          | 3.55                 | 0.15      | 1.14                        | 0.12               |  |
| Oven-dry Weight (g) | 4.30                 | 0.39      | 4.24                        | 0.33               |  |
| Density $(g/cm^3)$  | 0.33                 | 0.03      | 0.98                        | 0.11               |  |
| MOE (MPa)           | 7561.3               | 1204.7    | 27,303.9                    | 6,381.4            |  |
| $EI (Pa \cdot m^4)$ | 0.7204               | 0.0801    | 0.0898                      | 0.0185             |  |

Table 6 - Properties of hybrid poplar veneers with and without VTC treatment

The degree of densification and increase in MOE values were similar to those reported by Kutnar et al. (2009) who found increases up to 132% in hybrid poplar veneer density from VTC processing. However, degree of densification can be varied during VTC processing to produce materials with the desired final properties. Increases in MOE were similar to those reported by Kamke (2006) who found MOE's of densified radiata pine increased from 9,370 MPa to 20,200 MPa. Overall, VTC processing of specimens yielded uniform results that were similar to those reported previously.

Specimens from different treatment groups were densified together in batches to allow for proper statistical analysis. This minimized the risk of a batch effect, while randomizing VTC processing effects over all treatment groups. However, this also meant that specimens receiving different treatments were exposed to steam and pressure while together within the environmental chamber. Processing specimens from different treatment groups in one batch could result in volatilization of preservative components that could possibly move into other specimens during the steaming stage. Cinnamon leaf oil appeared to be particularly sensitive to this issue. Even a slight contamination could affect other properties, particularly those related to fungal or insect resistance.

#### 4.5 GC-MS Analysis

Randomly selected specimens from each preservative treatment group were extracted and this extract was analyzed by gas chromatography – mass spectrometry (CG-MS) analysis. Samples of pure juniper leaf oil and cinnamon oil were diluted in methanol or hexane and analyzed using GC-MS for comparison. Chemical components of juniper and cinnamon oil were identified and compared. Following peak integration, compound peak area relative abundance was compared across treatment groups. While relative abundance results could not be used to make comparisons with past research findings, relative abundance could be compared across treatment groups within this particular data set (Tables 7 and 8).

| Chemical               | Tatro et | Rudlo   | off et | Duringer | Juniper       | Hybrid  | VTC       |  |  |
|------------------------|----------|---------|--------|----------|---------------|---------|-----------|--|--|
|                        | al.      | al. (19 | 980)   | et al.   | oil           | poplar  | processed |  |  |
|                        | (1973)   |         |        | (2010)   | diluted       | treated | hybrid    |  |  |
|                        |          |         |        |          | to            | with    | poplar    |  |  |
|                        |          |         |        |          | $1/1000^{th}$ | 10%     | treated   |  |  |
|                        |          |         |        |          | in            | juniper | with 10%  |  |  |
|                        |          |         |        |          | MeOH          | oil     | juniper   |  |  |
|                        |          |         | • •    |          | 0.04          |         | oil       |  |  |
| Tricyclene             |          | 0.7     | 2.3    |          | 0.94          | -       | -         |  |  |
| α-Thujene              |          | 1.2     | 0.9    |          | 1.10          | 0.62    | 0.14      |  |  |
| α-Pinene               | 7.9      | 2.9     | 1.2    |          | 3.49          | 0.52    | 0.17      |  |  |
| Camphene               |          | 0.2     | 1.0    |          | 0.71          | 0.11    | 0.08      |  |  |
| Sabinene               | 37.9     | 14.2    | 8.4    | 13.1     | 10.43         | 1.59    | -         |  |  |
| Myrcene                | 2.1      | 1.8     | 1.1    |          | 1.65          | 0.39    | 0.5       |  |  |
| $\alpha$ -Phellandrene |          | 1.1     | 0.5    |          | 0.36          | 0.24    | 0.46      |  |  |
| Car-3-ene              | 7.8      | 0.3     | 0.4    |          | 0.81          | 0.20    | 0.19      |  |  |
| α-Terpinene            | 3.1      | 2.7     | 1.2    |          | 1.15          | 1.39    | -         |  |  |
| ho-Cymene              | 7.7      | 11.6    | 6.8    |          | 10.77         | 2.63    | 1.48      |  |  |
| β-Phellandrene         |          | 2.3     | 1.4    |          | 3.941         | -       | 0.81      |  |  |
| $\gamma$ -Terpinene    |          | 4.4     | 2.4    |          | -             | -       | 0.67      |  |  |
| Terpinolene            |          | 1.1     | 0.7    |          | 0.92          | 0.8     | 0.63      |  |  |
| Linalool               |          | 0.5     | 1.0    | 3.7      | 0.80          | 0.53    | 0.38      |  |  |
| Camphor                | Х        | 0.4     | 0.8    | 3.8      | 1.24          | 1.42    | 1.39      |  |  |
| Borneol                |          | 1.8     | 3.6    | 4.3      | 1.40          |         | 0.86      |  |  |
| Terpinen-4-ol          |          | 9.5     | 6.5    | 19.7     | 10.32         | 13.78   | 13.87     |  |  |
| $\alpha$ -Terpineol    |          | 0.4     | 0.9    |          | 1.03          | 2.28    | -         |  |  |
| $\rho$ -Cymenol        |          | 1.1     | 0.3    |          | 1.06          | -       | -         |  |  |
| Bornyl acetate         |          | 20.6    | 38.9   | 24.1     | 12.89         | 13.77   | 10.15     |  |  |
| Elemol                 |          | 1.5     | 2.8    |          | -             | -       | -         |  |  |
| m-cymene               |          |         |        | 9.2      | -             | -       | -         |  |  |
| Cinnamaldehyde         |          |         |        |          | -             | 13.77   | 3.02      |  |  |
| °(-) not detected      |          |         |        |          |               |         |           |  |  |

Table 7 – Relative amounts of compounds in western juniper leaf oils°

| Chemical          | Pure cinnamon<br>oil diluted to<br>1/1000 <sup>th</sup> in<br>MeOH | Hybrid poplar<br>treated with 10%<br>cinnamon oil | VTC processed hybrid<br>poplar treated with 10%<br>cinnamon oil |
|-------------------|--|---|---|
| Cinnamaldehyde    | 98.01  | 82.20   | 64.64   |
| Benzoic acid      | -  | 14.80   | 26.71   |
| °(-) not detected |  |   |   |

Pure cinnamon oil (OliveNation LLC, city, state) contained 98% cinnamaldehyde and was used as a standard. These values are consistent with previous research by Wang et al. (2005), who reported that cinnamaldehyde constituted anywhere from 8% to 81% of *Cinnamonmum osmophloeum* leaf oil. The high amount of cinnamaldehyde observed in cinnamon oil used in this experiment most likely reflects refining during manufacturing.

Western juniper contained a number of components including alpha-pinene, sabinene, car-3-ene, p-cymene, camphor, linalool, terpinen-4-ol, and bornyl acetate. These compounds were similar to those reported previously in western juniper leaf and heartwood oils (Tatro et al. 1973, Rudloff et al. 1980, Duringer et al. 2010). While relative percentages of these components varied depending upon harvest season, tree location, and extraction technique, the western juniper leaf oil formulated in this experiment appeared to be similar to oils studied in past research. Many of the components observed in pure juniper and cinnamon oil were also observed in extracts from treated hybrid poplar veneers. While it was not possible to quantify a given component, certain general trends were observed.

Peak area abundance decreased remarkedly when hybrid poplar veneers were VTC processed. Veneer specimens treated with 10% cinnamon oil experienced a 71% drop in cinnamaldehyde abundance when veneers were VTC processed (Figure 11). In addition, all compounds observed in western juniper leaf oil decreased in abundance following VTC processing (Figure 10). This decrease suggests that VTC processing may have decomposed compounds or made the extracts more susceptible to leaching from veneers during processing.

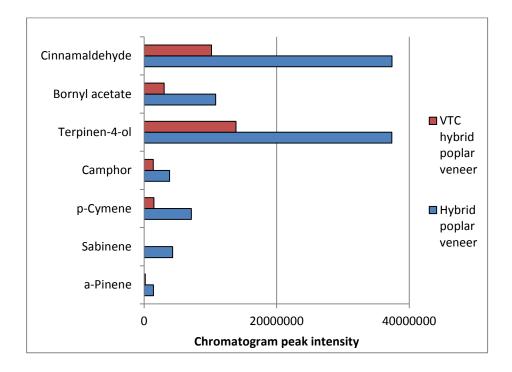


Figure 11 - Abundance of selected components in extracts from hybrid poplar veneers treated with western juniper leaf oils

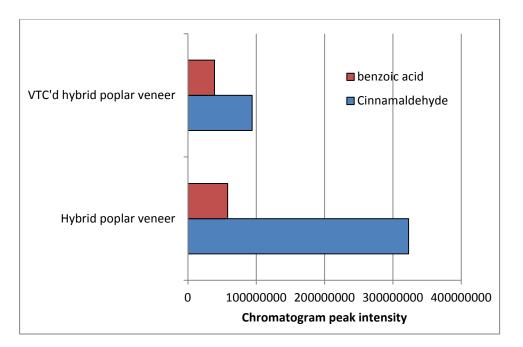


Figure 12 - Abundance of cinnamaldehyde and benzoic acid in extracts from hybrid poplar veneers

Increasing essential oil concentrations in preservative treatments was associated with increased compound peak area abundance. Analysis of extracts from hybrid poplar veneers treated with 2.5%, 5%, and 10% essential oil combinations experienced peak area abundance increases for terpinen-4-ol, cinnamaldehyde, and bornyl acetate (Figure 12). This pattern was not witnessed in treated veneers that had undergone VTC processing, reinforcing the assumption that VTC processing affected residual extraction.

Instability during VTC processing would sharply reduce the value of a treatment. While it might be possible to use higher concentrations of the extract to account for the loss, product cost would increase. Furthermore, there was no way to

determine whether degraded products might have other negative effects on the properties of the finished veneer.

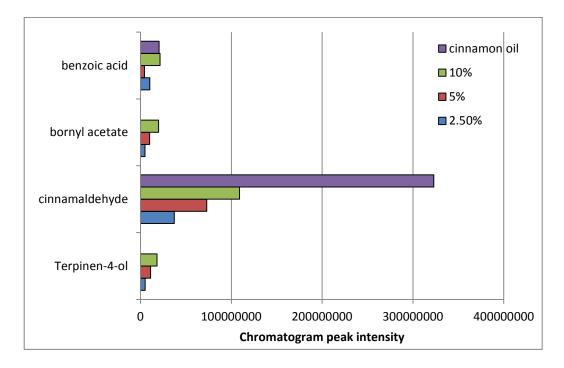


Figure 13 - Abundance of selected components in extracts from hybrid poplar veneers treated with essential oil formulations

# 4.6 Durability Assessments

### 4.6.1 Termite Test

Hybrid poplar veneers were sandwiched between two pieces of Douglas-fir plywood and exposed to Formosan termites for 6 months in Hilo, Hawaii following a modification of AWPA Standard E21-06. The low sample replication for each treatment (n = 5), coupled with the variability, tended to limit the ability to compare treatments. Medians are recommended for comparing nonparametric data sets while Mann-Whitney tests are recommended for making comparisons amongst treatment groups (Link & De Groot 1989). However, Mann-Whitney tests are inappropriate for data sets where values do not differ between many treatments and the sample size is small (Ramsey & Schafer 2002). Because of these limitations, the results will be discussed qualitatively.

In addition to data variability, non-uniform termite attack within the plywood array created positional effects. Although treatment group specimens were evenly distributed across the plywood array, specimens receiving a rating of 10 were clustered in a particular zone within the array. Specimens on the edge of the array were subjected to higher levels of attack, with some samples being destroyed (Figure 13). This further complicated the assessment, but it reflected the inherent variability associated with termite tests. Methods for reducing this problem in future tests would be to add more replicates, expose in several termite arrays, and to allow the test to run longer.

Hybrid poplar veneer specimens receiving neither VTC processing nor extract treatment experienced the heaviest termite attack, with a median rating of 4 (Figure 14). VTC processing of hybrid polar veneer appeared to increase resistance to termite attack although the effect was not always consistent. Median termite damage ratings increased to 8 for VTC treated veneer, while mass loss due to termite attack decreased from 40% to 10% (Figures 14 and 15). Density is not generally a predictor of resistance to termite attack, suggesting that either the heating associated with VTC processing or the potential for volatile contamination during the simultaneous processing of treated and non-treated veneers influenced attack.

| 5   | 54        | 60  |                   | 1 | Rat       | ing |
|-----|-----------|-----|-------------------|---|-----------|-----|
| 96  | 38        | 81  | 148               |   |           | 10  |
| 64  | 48        | 62  | 90                |   |           | 9   |
| 77  | 50        | 147 | 74                |   |           | 7   |
| 7   | 882       | 76  | 87                |   |           | 4   |
| 878 | 875       | 69  | 880               |   |           | 0   |
| 95  | 149       | 82  | 59                |   |           |     |
| 93  | 92        | 88  | 61                |   | 24 inches |     |
| 56  | 42        | 85  |                   |   |           |     |
| 7   | 78        | 2   | 23                |   |           |     |
| 52  | 36        | 63  | 97                |   |           |     |
| 14  | 100       | 80  | 79                |   |           |     |
| 26  | 9         | 877 | 68                |   |           |     |
| 57  | 53        | 66  | 873               |   |           |     |
| 40  | 73        | 72  | 879               |   |           |     |
| 70  | 47        | 87  | 86                |   |           |     |
| 84  | 65        | 83  | 876               |   |           |     |
| 874 | 34        | 55  | 58                |   |           |     |
| 13  | 49        | 67  | 71                |   |           |     |
| 7   | 75        | 881 | 91                | ↓ |           |     |
|     |           |     |                   |   |           |     |
| ←   |           |     | $\longrightarrow$ |   |           |     |
|     | 24 inches |     |                   |   |           |     |

Figure 14 - Distribution of termite ratings in specimens in an array exposed to Formosan termite attack for six months

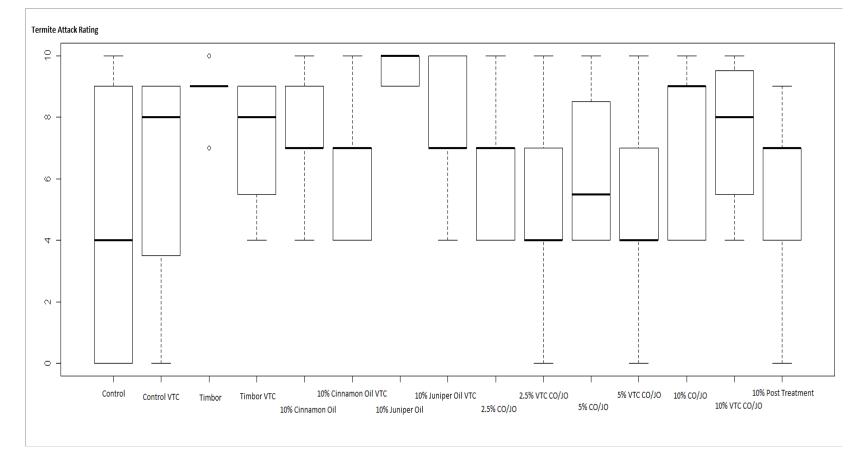


Figure 15 – Box plots reporting median Formosan termite damage ratings in hybrid poplar veneers subjected to variable treatments and exposed for six months in Hilo, HI.

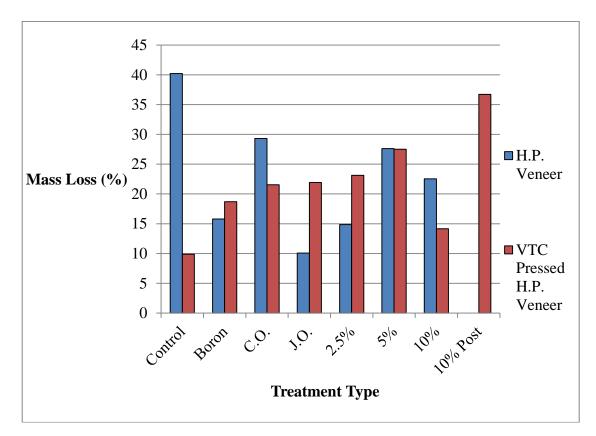


Figure 16 – Median mass loss in hybrid poplar veneers subjected to variable treatments and exposed to Formosan termites for six months in Hilo, HI.

|              |             | n | Average<br>Rating | Median<br>Rating | SD       | Mass<br>Loss<br>(%) | SD   |
|--------------|-------------|---|-------------------|------------------|----------|---------------------|------|
| No           | H.P. Veneer | 5 | 4.6               | 4.0              | 4.8      | 40.2                | 32.5 |
| Preservative | VTC Pressed | 5 | 6.3               | 8.0              | 4.3      | 9.9                 | 10.2 |
|              | H.P. Veneer |   |                   |                  |          |                     |      |
| Timbor®      | H.P. Veneer | 5 | 8.8               | 9.0              | 1.1      | 15.8                | 2.5  |
| Preservative | VTC Pressed | 5 | 7.8               | 9.0              | 2.4      | 18.7                | 11.0 |
|              | H.P. Veneer |   |                   |                  |          |                     |      |
| 10%          | H.P. Veneer | 5 | 7.4               | 7.0              | 2.3      | 29.3                | 12.0 |
| Cinnamon Oil | VTC Pressed | 5 | 6.4               | 7.0              | 2.5      | 21.6                | 9.6  |
| Preservative | H.P. Veneer |   |                   |                  |          |                     |      |
| 10% Juniper  | H.P. Veneer | 5 | 9.6               | 10.0             | 0.5      | 10.1                | 1.1  |
| Oil          | VTC Pressed | 5 | 7.0               | 7.0              | 3.0      | 21.9                | 16.1 |
| Preservative | H.P. Veneer |   |                   |                  |          |                     |      |
| 2.5% Juniper | H.P. Veneer | 5 | 5.8               | 4.0              | 2.7      | 14.9                | 7.5  |
| Oil/Cinnamon | VTC Pressed | 5 | 5.0               | 4.0              | 3.7      | 23.1                | 16.6 |
| Oil          | H.P. Veneer |   |                   |                  |          |                     |      |
| Preservative |             |   |                   |                  |          |                     |      |
| 5% Juniper   | H.P. Veneer | 5 | 6.3               | 5.5              | 2.9      | 27.6                | 18.6 |
| Oil/Cinnamon | VTC Pressed | 5 | 5.0               | 4.0              | 3.7      | 27.5                | 18.7 |
| Oil          | H.P. Veneer |   |                   |                  |          |                     |      |
| Preservative |             |   |                   |                  | •        |                     |      |
| 10% Juniper  | H.P. Veneer | 5 | 7.2               | 9.0              | 2.9      | 22.5                | 14.2 |
| Oil/Cinnamon | VTC Pressed | 5 | 7.5               | 8.0              | 2.6      | 14.2                | 7.6  |
| Oil          | H.P. Veneer |   |                   |                  |          |                     |      |
| Preservative |             |   |                   |                  |          |                     |      |
| 10% Juniper  | VTC Pressed | Ē |                   | <b>–</b> ^       | <u> </u> | 2.5-                |      |
| Oil/Cinnamon | H.P. Veneer | 5 | 5.4               | 7.0              | 3.5      | 36.7                | 35.8 |
| Oil Post VTC |             |   |                   |                  |          |                     |      |
| Processing   |             |   |                   |                  |          |                     |      |

Table 9 - Degree of termite damage on hybrid poplar specimens exposed without treatment or subjected to combinations of VTC processing and natural oil extracts

Hybrid poplar veneer specimens treated with 10% juniper oil were most resistant to termite attack, with a median rating of 10 and little variation between specimens. Mass loss due to termite attack was also low, with an average 10% mass loss. This observation reinforces previous research showing juniper oil to be strongly anti-termitic (Adams et al. 1988, Clark & McChesney 1990, Sichamba 2012). Other treatments that performed well against termite attack included a disodium octaborate tetrahydrate (DOT) powder (Timbor®) and a 10% combination of juniper and cinnamon oil (Table 9).

Increasing concentrations of essential oil resulted in improved termite protection. Median termite rating values for non-VTC treated veneers impregnated with 2.5%, 5%, and 10% concentrations of cinnamon and juniper oil increased from 4 to 5.5 to 9, respectively. A similar pattern was observed in VTC processed specimens as termite ratings increased from 4 to 8 as the treatment concentration rose from 2.5% to 10%. Treating VTC processed veneers with 10% essential oil treatment following processing (post treatment) did little to protect against termite attack.



Figure 17 - Hybrid poplar veneer specimens with five degrees of termite attack rating of 10,9,7,4,and 0 (left to right)

Chemical analyses indicated that VTC processing tended to reduce the activity of the extracts added to the material, likely reflecting thermal degradation of compounds during steaming. However, post VTC treatment with oils was largely ineffectual because the material was not receptive to fluid uptake. Improving the resistance of VTC treated wood to termite attack may require much higher levels of treatment with an understanding that some of this material may thermally degrade.

### 4.6.2. Mold Box Test

Exposure to *Trichoderma*, *Aspergillus niger*, *Penicillium citrinum*, and *Alternaria alternata* for eight weeks produced nearly complete discoloration of untreated pine sapwood, indicating that conditions were suitable for aggressive fungal attack (Table 10). Most samples experienced progressive mold attack over the eight week period.

Comparing between treatment groups was difficult due to the non-linear mold rating system. Lebow et al. (2008) described the difficulty in evaluating durability tests mentioning "...a return to more prescriptive data presentation may be warranted, as average ratings do not always adequately characterize the performance of a durable product." Often times, durability data are skewed by the occurrence of early failures, which are important in preservative evaluations (Lebow et al. 2008). Because mold ratings are based on an ordinal scale, Link and DeGroot (1989) suggested that it was inappropriate to compare group means.

VTC 2 weeks Treatment n 4 weeks 6 weeks 8 weeks 2.0 +3.5 <u>+</u> 1.4 10 2.0 + 1.5Poplar control receiving 3.0 +no preservative 1.5 1.5 10 2.0 +2.0 + 0.84.0 +5.0 + 0.8+1.0 0.7 Timbor<sup>®</sup> preservative \_ 10  $0.0 \pm$  $0.0 \pm 0.4$  $0.0 \pm$  $0.0 \pm 0.6$ 0.3 0.6 0.0 + 0.80.0 + 10 0.0 +0.0 + 0.6+0.8 0.8 10% cinnamon oil 10 0.0 + 0.0 + 0.00.0 +1.0 + 0.9\_ 0.8 preservative 0.0 10 0.0 +1.0 + 0.63.0 +4.0 + 0.9+0.4 0.8 3.0 + 10% juniper oil 10 1.0 + 1.5 + 1.24.5 + 1.5preservative 1.4 1.4 10 1.5 +2.0 + 1.14.0 +5.0 + 1.3 +1.4 1.0 2.5% juniper 10 0.5 + 2.0 <u>+</u> 1.4 3.0 +4.0 <u>+</u> 1.6 \_ oil/cinnamon oil 1.4 1.6 preservative 2.0 <u>+</u> 1.2 5.0 <u>+</u> 1.2 10 1.0 + 4.0 <u>+</u> +1.5 1.1 0.0 + 3.0 + 5% juniper oil/cinnamon 10 1.0 <u>+</u> 1.2 4.0 <u>+</u> 1.4 \_ oil preservative 1.1 1.3 10  $0.0 \pm$  $2.0 \pm 0.9$ 3.0 <u>+</u>  $4.0 \pm 0.9$ +0.9 0.9 10% juniper 10 0.0 +1.0 + 0.72.5 +4.0 + 1.0oil/cinnamon oil 0.2 0.9 preservative 10 0.0 +1.0 + 0.83.0 +4.0 <u>+</u> 1.2 +0.4 1.1 10% juniper 10 2.0 <u>+</u> 3.0 + 1.04.0 +5.0 + 0.8+oil/cinnamon oil post 1.2 1.1 VTC processing Pine control blocks \_ 5 2.5 +3.0 + 2.13.5 <u>+</u> 3.5 + 1.92.4 1.9 Values represent sample mean rating  $\pm$  one standard deviation. Mold rating ranged from 0 (no mold) to 5 (complete coverage)

Table 10 - Median hybrid poplar veneers specimens surface mold growth rating ( $\pm 1$  standard deviation) in an AWPA E24 mold box test

The Mann-Whitney test is recommended for comparing medians for nonparametric data (Link & DeGroot 1989). While the Mann-Whitney test was used in this instance to compare mold ratings, "...troublesome situations arise where sample sizes are small or when there are large number of ties" (Ramsey & Schafer 2002). Our mold test did experience a large number of ties within treatment groups. This must be acknowledged when considering the strength of the statistical analyses presented.

Hybrid poplar veneers receiving VTC processing were smoother and darker in appearance than their unprocessed counterparts. Because these specimens were visually rated for mold presence, it is possible that a darker contrast on the specimen surface influenced mold ratings. However, the rating of specimens was predicated upon the percentage of mold coverage on the specimen rather than the mold color intensity. It is believed that mold surface growth ratings were assigned fairly in accordance with the standard mold rating system.

Surface mold growth on hybrid poplar veneer controls had a median rating of 3.5 which did not differ statistically from pine control blocks (Mann-Whitney test with continuity correction, p = 0.82). VTC processing was associated with significantly more mold growth on specimens (one sided Mann-Whitney test with continuity correction, p = 0.00084), with median surface ratings increasing from 3.5 to 5 following VTC treatment (Figure 17).

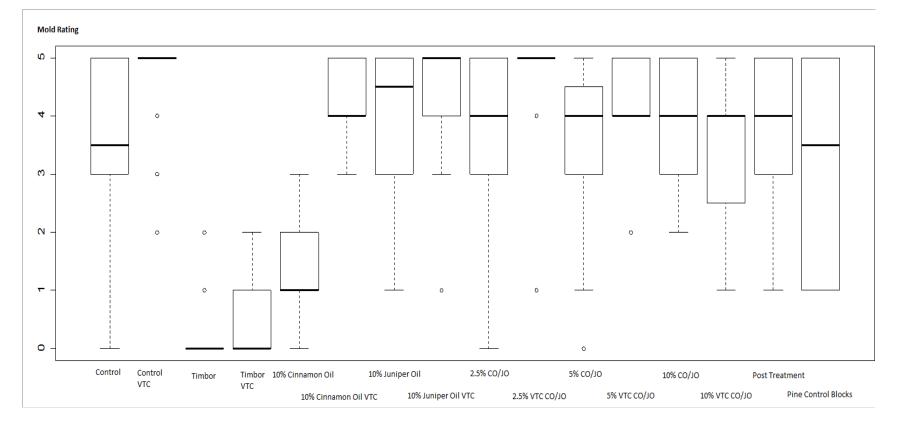


Figure 18 – Box plots reporting median mold rating for hybrid poplar veneer specimens subjected to various treatments and exposed to fungal attack in an AWPA mold box test

The increase in mold susceptibility due to VTC processing was not surprising. Molds can not degrade wood cell wall polymers, but instead utilize accessible sugars, starches, and proteins stored within ray parenchyma cells (Eriksson et al. 1990, Rayner & Boddy 1988). The heat associated with the VTC process may enhance mold growth by degrading hemicellulose due to acetyl group cleavage (Tjeerdsma & Militz 2005). Therefore, VTC processing may improve fungal access to these reserves.

The 5% DOT treatment served as a positive treatment and nearly completely inhibited surface mold growth. VTC processing did not affect DOT effectiveness (Mann-Whitney test with continuity correction, p = 0.51). DOT is susceptible to water leaching and the steam and pressure during VTC processing might be expected to result in some chemical loss. DOT appeared to be a likely candidate for decreasing mold susceptibility of VTC wood.

Of the essential oil treatments tested, only specimens treated with 10% cinnamon oil exhibited mold ratings that were significantly lower than controls (one sided Mann-Whitney test with continuity correction,  $p = 1.98 \times 10^{-5}$ ). The mold ratings decreased from 3.5 to 1 for the 10% cinnamon oil treatment. The effectiveness of cinnamon oil reinforces previous research (Wang et al. 2005, Chang & Cheng 2002, Wang et al. 2011).

The 10% cinnamon oil treatment was less effective when specimens were subjected to VTC processing. Median surface ratings of VTC processed veneers receiving cinnamon oil treatment rose from 1 to 4. The high temperature (170°C) and steam pressure (97 psi) created during VTC processing could thermally degrade cinnamaldehyde, the principle chemical compound found in cinnamon oil (Wang et al. 2005). The steam injection step during the VTC process may also produce cinnamaldehyde leaching. This hypothesis is supported by the fact that small traces of cinnamaldehyde were detected in extracts from multiple treatment groups during GC-MS analysis, indicating that cinnamaldehyde contamination was an issue.

Regardless of essential oil composition, the remaining preservative treatments had little effect on mold surface ratings (Figure 17). Mold performance was poorly correlated with preservative treatment concentration. Specimens receiving multiple treatment concentrations (2.5%, 5%, and 10%) without receiving VTC processing still had high median surface ratings (rating = 4).

Combinations of essential oils did not perform better than single essential oil treatments. Combinations of cinnamaldehyde and eugenol have been shown to perform better than either chemical alone (Yen and Chang 2008). However, combining cinnamaldehyde with juniper oil did not produce any synergistic effect. This test could be improved by using fewer treatment groups with more replicates with combinations of other natural products such as clove oil, mint oil, or cedar heartwood oil.

## 4.6.3 Decay Bending Test

While some tests resulted in significant changes in mass and bending strength, overall attempts at establishing the brown rot fungus met with limited success. In the first bending decay experiment, specimens receiving particular preservative treatments were randomly allocated to environmental bags in an attempt to minimize environmental chamber effects. Each bag contained ten specimens from multiple treatment groups. The volatile nature of the essential oils in some treatments appeared to inhibit the ability of the brown-rot fungus to become established within bags. The only evidence of fungal growth following four weeks of incubation were in bags containing only non-treated control specimens. Fungal hyphae were visible and veneers felt soft and spongy to the touch. Both VTC and non-VTC processed specimens experienced statistical similar mass losses of 22.2% (two-sample t-test assuming unequal variances, p = 0.09). Average MOE for non-VTC processed hybrid poplar veneers decreased from 5,026.1 MPa to 1,194.1 MPa at the end of the eight week exposure.

In the second test, the original test specimens were segregated by treatment group and incubated in new sterilized environmental bags. Specimens were then reinoculated with *G. trabeum* and incubated at 23°C and 65% relative humidity. No signs of decay were present after four weeks and the experiment was terminated. It is believed that volatiles from one or more treatments diffused into all specimens, making it difficult to for the test fungus to become established.

In the third test new VTC and non-VTC processed veneers were fabricated, treated with their respective preservative treatment as previously described, and placed in bags and inoculated with *G. trabeum* as previously described. Treatment groups were reduced from the original eight down to four and consisted of a set of controls, a 10% juniper oil treatment, a 10% cinnamon oil treatment, and a 10% juniper/cinnamon oil combination treatment. No fungal growth was observed following four weeks of incubation so the bags were re-inoculated. The second inoculation involved dripping fungal suspension into specimen end grain instead of inoculating vermiculite.

Fungal growth four weeks after the inoculation was only observed on one treatment group, hybrid poplar veneers that received VTC treatment and a 10% juniper oil preservative. Average mass loss due to decay for these specimens was 10.7% ( $\pm$  4.9%). Once again, it was difficult to create conditions where decay fungi could become established on test specimens.

While limited statements about the influence of brown rot decay on bending properties can be made, the decay bending tests did provide valuable information on the effects of moisture exposure on VTC wood behavior. VTC processed specimens were exposed to moisture conditions that increased moisture contents to well above fiber saturation point. While moisture content information during the eight week exposure is unknown, Final moisture contents ranged from 82% to 169%. Specimens also experienced 78% to 195% radial thickness swell over the eight week exposure. These values were substantially higher than the 19% to 38% thickness swell values observed by Kamke et al. (2009). The larger thickness swells in this experiment were not surprising due to the proportional relationship between degree of densification and springback. Veneer density increased as much as 197% during VTC processing. VTC processing normally includes an extra process step with heat treatment at 200°C. Heat treatment was omitted in the current study.

Veneer specimens were subjected to three point non-destructive bending tests as many as six times during decay bending experiments. Both Gonzalez (2010) and Li et al. (2007) observed 10% to 20% reductions in control specimen MOE as a result of repeated bending. A similar behavior was observed for hybrid poplar veneer specimens used in this experiment (Figure 18). Average MOE for control specimens decreased 34% after six bending tests. Decreases in MOE could also be attributed to specimen thickness swell and increases in specimen moisture content as veneers acclimated over the eight week period. However, these values were determined when specimens were subjected to bending within environmental bags. Following removal from bags and return to the oven dry state, MOE returned to the same levels observed at the initial bending test.

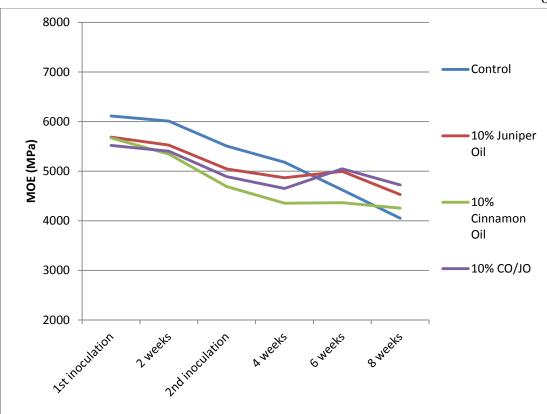


Figure 19 - MOE's of hybrid poplar veneer subjected to various treatments and exposed to wet conditions for eight weeks

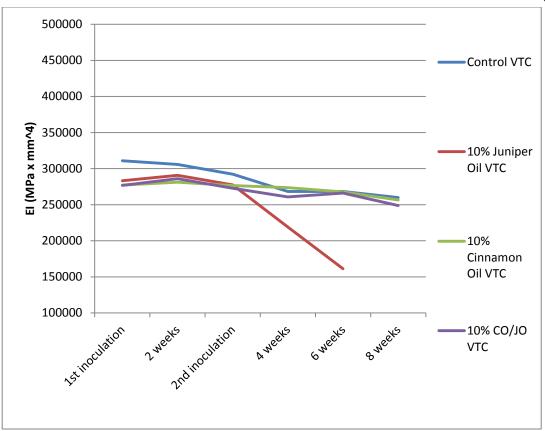


Figure 20 - VTC treated hybrid poplar veneer EI's following exposure to brown-rot fungi *G. trabeum* 

## **CHAPTER 5 - CONCLUSIONS**

All conclusions pertinent to wood durability and thickness recovery in these experiments are limited to hybrid poplar veneers receiving the particular VTC processing schedule used in their densification. It is important to note that VTC processed veneers did not receive a heat treatment.

The VTC process has the potential to give low density species the mechanical properties required for use in structural wood composites. Hybrid poplar veneers in this experiment experienced increases in density of approximately 260%. Densified veneers could be incorporated into laminated veneered lumber, plywood, oriented strand board, or used to increase surface hardness for products like flooring or cabinetry. However, VTC treated wood is limited by its susceptibility to biological attack and its thickness swelling response to moisture. VTC treatment improved the median termite attack rating from 4 to 8. However, VTC processing also increased mold susceptibility, raising the median mold coverage rating from 3.5 to 5.

Protecting VTC treated wood from moisture is of utmost importance if it is to be incorporated into structural use. When exposed to water, the swelling response of VTC wood reduces the overall veneer density, thus decreasing the mechanical properties for which it would be utilized. VTC specimens recovered to 83% of their original thickness following eight week exposure inside environmental bags. Future research should continue to explore ways to reduce thickness springback resulting from moisture exposure. One potential to control thickness recovery is by adding a heat treatment step to the VTC densification process. Including a heat treatment of 180°C following VTC processing has been shown to help reduce set recovery (Kutnar & Kamke 2012).

Essential oils proved effective at enhancing durability when used in high concentrations. Compared to controls, a 10% juniper oil treatment increased the median termite attack rating from 4 to 10 and reduced mass loss from 40% to 10%. A 10% cinnamon oil treatment dropped the median mold coverage rating from 3.5 to 1. However, the abundance of chemical compounds inherent within these oils decreased remarkedly when treated samples were subjected to VTC processing. The concentrations of essential oils required to increase hybrid poplar veneer durability most likely exclude them commercial feasibility.

Additional opportunities for increasing wood durability exist. Industrial borate powder treatments applied prior to VTC processing could represent one approach. The industrial preservative Timbor® proved effective at surviving VTC treatment at 170°C and protecting veneers specimens from termites and staining fungi with respective median attack ratings of 8 and 0. However, alternative pretreatments must withstand the high temperature and pressures that take place during processing. Treating wood following VTC processing will prove difficult, as preservative uptake and retention is far lower for VTC wood than unprocessed veneers.

Opportunities to address both durability and set recovery exist. Combining a DOT pretreatment with a wax coating following VTC processing may better protect VTC wood from biological decay and moisture. This approach may make VTC wood

suitable for outdoor environments. However it is unlikely that VTC wood will be utilized in outdoor applications where repeated exposure to liquid water takes place.

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## APPENDICES

|     | Time    | Input Material, | Oil Weight |       | Distillation |
|-----|---------|-----------------|------------|-------|--------------|
| Run | (Hours) | wet weight (g)  | (g)        | Yield | Date         |
| 2   | 5       | 2363.71         | 17.48      | 0.74% | 1/17/11      |
| 3   | 5       | 2055.58         | 15.52      | 0.76% | 1/18/11      |
| 4   | 5       | 2755.75         | 24.15      | 0.88% | 1/19/11      |
| 5   | 5       | 2803.31         | 12.87      | 0.46% | 1/20/11      |
| 6   | 5       | 1878.15         | 10.45      | 0.56% | 6/4/11       |
| 7   | 5       | 2384.78         | 14.37      | 0.60% | 6/5/11       |
| 8   | 5       | 2422.34         | 16.85      | 0.70% | 6/6/11       |
| 9   | 5       | 2668.73         | 16.69      | 0.63% | 6/7/11       |
| 10  | 5       | 2655.63         | 15.1       | 0.57% | 6/11/11      |
| 11  | 5       | 2426.25         | 17.06      | 0.70% | 6/12/11      |
| 12  | 5       | 2683.87         | 17.58      | 0.66% | 6/13/11      |
| 13  | 5       | 2426.48         | 16.62      | 0.68% | 6/14/11      |

Table 1 – Western juniper steam distillation data

| Timbor | 10%<br>Cinnamon<br>oil | 10%<br>Juniper oil | 2.5%<br>Cinnamon<br>/juniper<br>oil | 5%<br>Cinnamon<br>/juniper<br>oil | 10%<br>Cinnamon<br>/juniper<br>oil | 10%<br>Cinnamo<br>n/junipe<br>r oil -<br>post VTC<br>treatme<br>nt |
|--------|------------------------|--------------------|-------------------------------------|-----------------------------------|------------------------------------|--|
| 6.72   | 18.44                  | 7.04               | 2.62                                | 4.27                              | 8.25                               | -1.09  |
| 7.11   | 20.05                  | 7.13               | 2.76                                | 2.94                              | 8.13                               | 0.39   |
| 7.40   | 21.53                  | 5.15               | 3.20                                | 4.65                              | 8.24                               | -1.06  |
| 7.51   | 18.79                  | 5.34               | 2.86                                | 4.08                              | 8.30                               | 0.23   |
| 6.77   | 20.35                  | 4.74               | 3.09                                | 3.79                              | 7.60                               | -1.17  |
| 6.98   | 19.44                  | 6.24               | 3.21                                | 4.92                              | 8.47                               | 0.08   |
| 7.42   | 19.87                  | 5.88               | 2.96                                | 5.31                              | 8.09                               | -0.72  |
| 8.30   | 17.22                  | 7.84               | 3.12                                | 4.42                              | 8.55                               | -1.73  |
| 7.83   | 12.23                  | 6.86               | 3.61                                | 5.00                              | 6.72                               | -1.10  |
| 7.41   | 17.59                  | 6.58               | 3.18                                | 4.85                              | 8.44                               | -1.28  |
| 10.26  | 19.63                  | 4.67               | 3.28                                | 4.74                              | 8.00                               | -1.14  |
| 6.42   | 21.33                  | 6.57               | 2.85                                | 3.54                              | 6.73                               | -0.54  |
| 7.29   | 18.39                  | 4.75               | 3.26                                | 5.04                              | 7.63                               | -1.17  |
| 6.67   | 19.07                  | 5.63               | 3.10                                | 4.42                              | 7.11                               | -1.04  |
| 5.88   | 18.09                  | 6.67               | 2.59                                | 2.75                              | 7.82                               | -1.05  |
| 6.46   | 17.79                  | 5.71               | 2.91                                | 3.89                              | 7.51                               | -0.80  |
| 5.63   | 18.56                  | 6.90               | 2.94                                | 5.49                              | 7.19                               | -1.04  |
| 7.49   | 18.18                  | 5.87               | 3.56                                | 4.55                              | 6.81                               | -0.88  |
| 6.57   | 18.08                  | 5.93               | 2.36                                | 4.56                              | 7.63                               | 0.15   |
| 5.56   | 13.71                  | 7.26               | 3.14                                | 5.62                              | 7.87                               | 0.21   |
| 7.44   | 18.96                  | 7.16               | 2.73                                | 4.51                              | 8.03                               | -0.94  |
| 6.12   | 17.91                  | 5.89               | 3.59                                | 5.31                              | 8.83                               | -0.48  |
| 6.98   | 17.72                  | 6.50               | 3.11                                | 6.36                              | 8.71                               | -2.69  |
| 7.73   | 13.87                  | 6.52               | 3.95                                | 5.56                              | 8.25                               | -3.48  |
| 8.15   | 19.59                  | 5.68               | 3.21                                | 4.23                              | 9.46                               | 0.02   |
| 6.50   | 19.33                  | 4.92               | 3.05                                | 5.68                              | 9.07                               | -1.17  |
| 7.92   | 18.07                  | 7.93               | 3.38                                | 4.54                              | 8.82                               | -1.40  |
| 6.61   | 20.08                  | 7.96               | 2.73                                | 4.77                              | 8.10                               | -1.50  |
| 7.51   | 19.11                  | 6.80               | 2.85                                | 4.44                              | 7.71                               | -1.12  |
| 7.06   | 19.15                  | 5.63               | 2.43                                | 4.94                              | 8.91                               | -2.49  |
| 6.42   | 19.32                  | 6.39               | 3.06                                | 4.94                              | 7.95                               | -2.48  |

Table 2 – Hybrid poplar veneer weight gain (%) following essential oil treatment application

|       |       |      |      |      |       | 81    |
|-------|-------|------|------|------|-------|-------|
| 6.75  | 18.02 | 5.78 | 3.72 | 5.39 | 7.61  | -2.91 |
| 7.72  | 18.31 | 6.81 | 3.03 | 4.94 | 6.10  | -2.92 |
| 8.34  | 19.35 | 5.42 | 2.86 | 6.16 | 8.85  | -3.46 |
| 6.40  | 16.81 | 6.78 | 3.11 | 4.43 | 6.78  | 0.11  |
| 7.26  | 19.23 | 6.45 | 3.33 | 4.47 | 8.22  | -2.74 |
| 7.18  | 19.36 | 5.99 | 3.26 | 4.58 | 7.30  | -3.36 |
| 8.37  | 20.24 | 9.15 | 2.73 | 4.28 | 5.41  | -2.27 |
| 7.10  | 18.70 | 6.13 | 2.88 | 5.01 | 8.92  | 0.02  |
| 7.17  |       | 5.40 | 2.26 | 4.14 | 8.31  | -3.15 |
| 8.04  | 8.95  | 4.10 | 3.14 | 5.15 | 9.11  | -1.99 |
| 6.41  | 18.49 | 6.80 | 2.90 | 5.10 | 8.66  | -1.49 |
| 9.27  | 19.17 | 2.42 | 2.69 | 4.19 | 7.87  | -1.71 |
| 7.51  | 19.55 | 6.18 | 3.61 | 4.31 | 8.96  | -1.76 |
| 8.50  | 19.93 | 7.12 | 3.24 | 4.60 | 8.20  | -1.65 |
| 6.80  | 19.10 | 5.95 | 3.53 | 4.73 | 9.38  | -1.64 |
| 7.21  | 19.97 | 6.40 | 4.42 | 4.91 | 7.64  | -2.31 |
| 7.75  | 20.71 | 6.37 | 4.29 | 5.29 | 8.29  | -1.70 |
| 7.45  | 20.20 | 5.56 | 7.08 | 4.17 | 8.31  | -1.67 |
| 8.17  | 19.16 | 5.20 | 3.66 | 4.60 | 8.39  | 0.03  |
| 6.99  | 14.40 | 5.54 | 3.82 | 4.30 | 6.45  | -2.04 |
| 5.80  |       | 6.35 | 3.18 | 4.45 | 8.86  | -3.17 |
| 6.14  |       | 7.34 | 3.31 | 4.62 | 8.14  | 0.31  |
| 6.90  |       | 6.59 | 3.30 | 4.60 | 8.32  | -2.24 |
| 7.21  |       | 7.48 | 3.58 | 5.90 | 7.69  | -1.83 |
| 7.51  |       | 6.63 | 4.41 | 4.39 | 8.43  | -2.28 |
| 7.85  |       | 7.27 | 3.83 | 5.15 | 9.95  | -2.73 |
| 6.02  |       | 6.70 | 3.92 | 5.45 | 10.55 | -2.68 |
| 6.67  |       | 5.34 | 3.86 | 4.46 | 9.31  | -3.24 |
| 7.32  |       | 6.31 | 3.80 | 4.58 | 8.86  | -1.90 |
| 8.10  |       | 5.53 | 3.88 | 4.07 | 8.80  | -1.74 |
| 8.08  |       | 5.95 | 3.61 | 4.34 | 10.71 | -0.46 |
| 7.96  |       | 7.27 | 4.25 | 5.34 | 10.10 | 0.11  |
| 9.92  |       | 7.84 | 3.70 | 4.01 | 7.86  | -0.84 |
| 7.77  |       | 7.18 | 3.68 | 4.39 | 7.89  | -1.09 |
| 8.78  |       | 9.71 | 3.29 | 6.14 | 7.97  | -1.62 |
| 6.53  |       | 6.47 |      | 4.47 | 9.27  | -2.13 |
| 6.78  |       | 6.15 |      | 4.20 | 9.88  | -2.05 |
| -4.06 |       | 6.48 |      | 4.88 | 8.07  | -0.34 |
| 7.90  |       | 6.45 |      | 4.50 | 10.50 | -2.31 |

| 0 | 2 |
|---|---|
| 0 | 2 |

|       |      |      |       | 82    |
|-------|------|------|-------|-------|
| 7.92  | 6.61 | 4.74 | 9.48  | -2.31 |
| 8.40  | 6.35 | 3.98 | 5.26  | -3.23 |
| 10.35 | 7.81 | 6.19 | 9.48  | -2.63 |
| 10.11 | 7.09 | 4.22 | 11.40 | 0.40  |
| 7.84  | 6.46 | 4.17 | 8.82  | -2.06 |
| 8.03  | 6.00 | 5.16 | 9.79  | 0.05  |
| 8.52  | 7.43 | 5.60 | 8.67  | -2.42 |
| 8.20  | 7.74 | 6.32 | 8.26  | -0.05 |
| 7.52  | 7.19 | 3.96 | 6.98  | -2.78 |
| 7.10  | 7.94 | 4.85 | 7.24  | -1.27 |
|       |      |      |       | 0.35  |
|       |      |      |       | -2.80 |
|       |      |      |       | -2.51 |
|       |      |      |       | -1.14 |
|       |      |      |       | -3.45 |
|       |      |      |       | -1.73 |
|       |      |      |       | -1.63 |
|       |      |      |       | -0.24 |
|       |      |      |       | -2.66 |
|       |      |      |       | -1.55 |
|       |      |      |       | -1.49 |
|       |      |      |       | -1.60 |
|       |      |      |       | -0.03 |

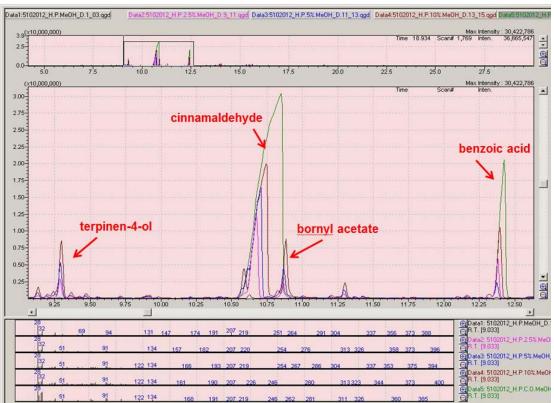


Figure 1 – Abundance of selected components in extracts from hybrid poplar veneers treated with essential oil formulations (black = control, pink = 2.5% cinnamon/juniper oil, blue = 5% cinnamon/juniper oil, brown = 10% cinnamon/juniper oil, green = 10% cinnamon oil)

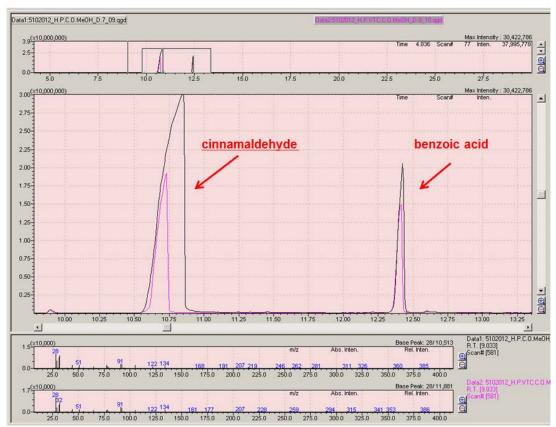


Figure 2 – Abundance of cinnamaldehyde and benzoic acid in extracts from hybrid poplar veneers and VTC treated hybrid poplar veneers (black = 10% cinnamon oil, pink = 10% cinnamon oil post VTC processing)

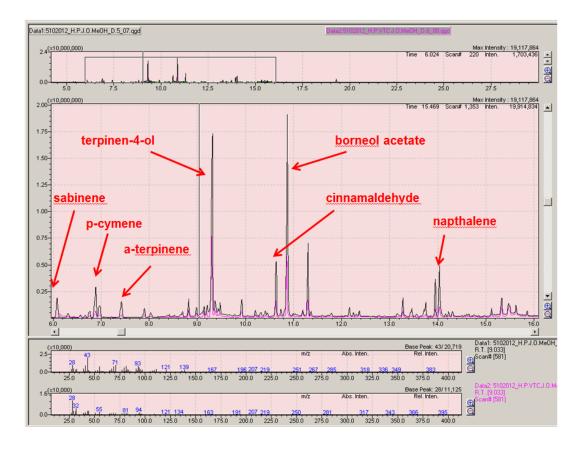


Figure 3 – Abundance of selected components in extracts from hybrid poplar veneers treated with western juniper leaf oils (black = 10% juniper oil, pink = 10% juniper oil post VTC processing)

|                |         | Initial<br>Weight |              | Final Wet<br>Weight | Final Dry<br>Weight |
|----------------|---------|-------------------|--------------|---------------------|---------------------|
| Classification | Sample# | (g)               | Final Rating | (g)                 | (g)                 |
| True Control   | 24      | 4.27              | 4            | 4.286               | 2.323               |
|                | 148     | 4.55              | 10           | 8.024               | 4.33                |
|                | 323     | 4.677             | 0            | 2.792               | 1.659               |
|                | 428     | 4.316             | 9            | 8.43                | 3.929               |
|                | 548     | 4.728             | 0            | 1.33                | 1.079               |
| VTC Control    | 34      | 4.922             |              |                     |                     |
|                | 142     | 4.079             | 7            | 5.204               | 3.056               |
|                | 433     | 4.052             | 0            | 7.633               | 3.915               |
|                | 505     | 4.171             | 9            | 6.205               | 3.974               |
|                | 517     | 4.085             | 9            | 7.75                | 3.827               |
| Boron Control  | 32      | 5.454             | 7            | 10.07               | 4.386               |
|                | 70      | 4.782             | 9            | 10.007              | 4.08                |
|                | 163     | 4.704             | 10           | 11.37               | 4.107               |
|                | 232     | 4.495             | 9            | 10.084              | 3.799               |
|                | 345     | 4.582             | 9            | 9.635               | 3.824               |
| Boron VTC      | 48      | 4.647             | 9            | 9.844               | 3.988               |
|                | 63      | 4.505             | 7            | 8.893               | 3.598               |
|                | 150     | 4.925             | 10           | 10.584              | 4.447               |
|                | 328     | 4.707             | 4            | 6.119               | 2.961               |
|                | 539     | 4.802             | 9            | 10.684              | 4.21                |
| Cinnamon Oil   |         |                   |              |                     |                     |
| Control        | C.O.A   | 5.133             | 7            | 7.86                | 3.706               |
|                | C.O.B   | 4.451             | 10           | 7.915               | 3.651               |
|                | C.O.C   | 5.58              | 9            | 6.763               | 4.072               |
|                | C.O.D   | 5.445             | 4            | 4.683               | 2.741               |
|                | C.O.E   | 4.672             | 7            | 8.111               | 3.545               |
| Cinnamon Oil   |         |                   |              |                     |                     |
| VTC            | 703     | 4.342             | 4            | 5.445               | 2.97                |
|                | 721     | 3.56              | 7            | 5.556               | 2.766               |
|                | 730     | 4.962             | 4            | 5.798               | 3.501               |
|                | 736     | 3.516             | 7            | 5.278               | 2.942               |
|                | 739     | 4.129             | 10           | 6.564               | 3.795               |
| Juniper Oil    |         |                   |              |                     |                     |
| Control        | 79      | 4.942             | 10           | 9.777               | 4.437               |
|                | 103     | 4.049             | 10           | 8.878               | 3.641               |
|                | 308     | 4.841             | 9            | 9.081               | 4.321               |
|                | 383     | 4.687             | 9            | 9.445               | 4.165               |

Table 3 – Hybrid poplar veneer termite attack ratings and specimen weight following six months of exposure to Formosan termites

|                 | 437   | 4.508 | 10  | 8.372 | 4.139 |
|-----------------|-------|-------|-----|-------|-------|
| Juniper Oil VTC | 222   | 5.297 | 4   | 6.725 | 3.586 |
|                 | 276   | 4.788 | 10  | 8.118 | 4.564 |
|                 | 313   | 4.288 | 10  | 8.03  | 4.095 |
|                 | 337   | 4.858 | 4   | 5.158 | 3.002 |
|                 | 410   | 4.234 | 7   | 6.156 | 2.969 |
| 2.5% Control    | 12    | 5.663 | 4   | 9.241 | 4.843 |
|                 | 208   | 4.874 | 4   | 7.47  | 4.094 |
|                 | 224   | 3.844 | 10  | 8.078 | 3.533 |
|                 | 254   | 4.456 | 4   | 6.684 | 3.261 |
|                 | 406   | 5.161 | 7   | 7.902 | 4.701 |
| 2.5% VTC        | 372   | 3.902 | 0   | 3.316 | 2.032 |
|                 | 414   | 4.266 | 4   | 6.24  | 3.708 |
|                 | 481   | 3.739 | 10  | 7.431 | 3.557 |
|                 | 2.5.A | 4.408 | 7   | 6.186 | 3.521 |
|                 | 2.5.B | 4.072 | 4   | 5.876 | 2.864 |
| 5% Control      | 207   | 3.596 | 7   | 7.179 | 2.892 |
|                 | 369   | 4.374 | 10  | 8.023 | 4.006 |
|                 | 370   | 5.081 | 4   | 6.285 | 3.534 |
|                 | 494   | 5.307 | n/a |       |       |
|                 | 502   | 4.195 | 4   | 4.343 | 2.014 |
| 5% VTC          | 367   | 4.462 | 7   | 6.99  | 3.802 |
|                 | 429   | 4.5   | 10  | 8.079 | 4.307 |
|                 | 440   | 4.675 | 0   | 3.115 | 2.3   |
|                 | 514   | 4.175 | 4   | 5.595 | 3.024 |
|                 | 533   | 3.857 | 4   | 3.993 | 2.309 |
| 10% Control     | 134   | 5.026 | 4   | 5.38  | 3.228 |
|                 | 348   | 4.455 | 10  | 8.967 | 3.974 |
|                 | 360   | 4.639 | 9   | 7.469 | 4.157 |
|                 | 392   | 4.588 | 4   | 5.796 | 2.756 |
|                 | 455   | 4.653 | 9   | 8.643 | 3.92  |
| 10% VTC         | 214   | 5.093 | 7   | 8.444 | 4.311 |
|                 | 344   | 4.908 | 4   | 6.659 | 3.622 |
|                 | 472   | 4.303 | 9   | 7.71  | 4.025 |
|                 | 495   | 4.47  |     | 8.263 | 3.85  |
|                 | 509   | 3.661 | 10  | 7.779 | 3.333 |
| 10% Post VTC    |       |       |     |       |       |
| Treatment       | 623   | 4.15  | 4   | 4.483 | 2.62  |
|                 | 671   | 4.05  | 7   | 4.571 | 2.918 |
|                 | 697   | 4.403 | 0   | 0.183 | 0.153 |
|                 | 808   | 4.801 | 7   | 7.546 | 3.828 |
|                 | 842   | 3.733 | 9   | 6.249 | 3.659 |

|              |        |       | eeks | -     | eeks |       | eeks | 8 W   | eeks |
|--------------|--------|-------|------|-------|------|-------|------|-------|------|
| Classificati | Sample | Front | Back | Front | Back | Front | Back | Front | Back |
| on           | #      | Side  | Side | Side  | Side | Side  | Side | Side  | Side |
|              | 27     | 2     | 0    | 2     | 0    | 3     | 1    | 3     | 2    |
|              | 46     | 0     | 0    | 1     | 0    | 2     | 2    | 3     | 2    |
|              | 97     | 2     | 2    | 2     | 2    | 3     | 4    | 3     | 4    |
|              | 126    | 0     | 0    | 0     | 0    | 2     | 2    | 3     | 3    |
|              | 251    | 3     | 2    | 3     | 3    | 4     | 4    | 4     | 5    |
|              | 274    | 2     | 2    | 2     | 2    | 3     | 4    | 3     | 4    |
|              | 297    | 4     | 3    | 4     | 4    | 5     | 5    | 5     | 5    |
|              | 338    | 4     | 3    | 4     | 3    | 5     | 3    | 5     | 4    |
| True         | 482    | 4     | 2    | 4     | 2    | 4     | 4    | 5     | 5    |
| Control      | 493    | 0     | 0    | 0     | 0    | 0     | 0    | 1     | 0    |
|              | 39     | 3     | 4    | 3     | 4    | 5     | 5    | 5     | 5    |
|              | 57     | 2     | 0    | 2     | 1    | 4     | 2    | 4     | 2    |
|              | 88     | 2     | 2    | 2     | 3    | 4     | 4    | 5     | 5    |
|              | 121    | 3     | 1    | 3     | 2    | 4     | 3    | 5     | 5    |
|              | 202    | 1     | 1    | 2     | 2    | 4     | 4    | 5     | 5    |
|              | 238    | 2     | 2    | 2     | 2    | 3     | 3    | 4     | 3    |
|              | 267    | 2     | 1    | 2     | 2    | 4     | 4    | 5     | 5    |
|              | 283    | 4     | 3    | 4     | 3    | 5     | 4    | 5     | 5    |
| VTC          | 287    | 2     | 3    | 2     | 4    | 4     | 4    | 5     | 5    |
| Control      | 382    | 3     | 2    | 3     | 3    | 4     | 4    | 5     | 5    |
|              | 18     | 0     | 0    | 0     | 0    | 0     | 0    | 0     | 0    |
|              | 101    | 0     | 0    | 0     | 0    | 0     | 0    | 0     | 0    |
|              | 170    | 0     | 0    | 0     | 0    | 0     | 0    | 0     | 0    |
|              | 195    | 0     | 0    | 0     | 0    | 0     | 0    | 0     | 0    |
|              | 302    | 0     | 0    | 0     | 0    | 0     | 0    | 0     | 0    |
|              | 353    | 0     | 0    | 0     | 0    | 0     | 0    | 0     | 0    |
|              | 404    | 0     | 0    | 1     | 0    | 2     | 0    | 2     | 0    |
|              | 443    | 0     | 0    | 0     | 0    | 0     | 0    | 0     | 0    |
| Boron        | 478    | 1     | 1    | 1     | 1    | 1     | 1    | 1     | 1    |
| Control      | 547    | 0     | 0    | 1     | 0    | 1     | 0    | 1     | 0    |
|              | 3      | 0     | 0    | 0     | 0    | 0     | 0    | 0     | 0    |
|              | 85     | 0     | 0    | 0     | 0    | 0     | 0    | 0     | 0    |
|              | 178    | 1     | 2    | 1     | 2    | 1     | 2    | 1     | 2    |
| Boron VTC    | 320    | 0     | 0    | 0     | 0    | 0     | 0    | 0     | 0    |

Table 4 - Mold coverage ratings for hybrid poplar veneer specimen tangential faces at two week intervals during eight weeks exposure to mold fungi

|             |      |   |   |   |   |   |   |   | 8 | 39 |
|-------------|------|---|---|---|---|---|---|---|---|----|
|             | 438  | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |    |
|             | 445  | 0 | 0 | 0 | 1 | 0 | 1 | 0 | 1 |    |
|             | 474  | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 |    |
|             | 519  | 0 | 0 | 0 | 0 | 1 | 0 | 0 | 0 |    |
|             | 522  | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |    |
|             | 523  | 0 | 3 | 0 | 3 | 0 | 3 | 0 | 1 |    |
|             | 35   | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 |    |
|             | CO.F | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 |    |
|             | CO.G | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |    |
|             | CO.H | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 |    |
|             | CO.I | 0 | 0 | 0 | 0 | 0 | 3 | 0 | 3 |    |
|             | CO.J | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 1 |    |
|             | CO.K | 0 | 0 | 0 | 0 | 0 | 0 | 2 | 2 |    |
|             | CO.L | 0 | 0 | 0 | 0 | 0 | 0 | 2 | 2 |    |
| Cinnamon    | CO.M | 0 | 0 | 0 | 0 | 0 | 1 | 2 | 3 |    |
| Oil Control | CO.N | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 |    |
|             | 701  | 0 | 0 | 1 | 1 | 3 | 3 | 5 | 5 |    |
|             | 710  | 0 | 0 | 1 | 1 | 3 | 2 | 4 | 4 |    |
|             | 712  | 0 | 1 | 1 | 1 | 2 | 2 | 3 | 3 |    |
|             | 719  | 0 | 0 | 1 | 1 | 3 | 3 | 4 | 4 |    |
|             | 723  | 0 | 0 | 1 | 1 | 3 | 2 | 3 | 4 |    |
|             | 724  | 0 | 0 | 1 | 1 | 2 | 3 | 3 | 4 |    |
|             | 731  | 0 | 1 | 1 | 1 | 3 | 3 | 4 | 4 |    |
|             | 733  | 0 | 0 | 3 | 1 | 5 | 3 | 5 | 5 |    |
| Cinnamon    | 734  | 0 | 0 | 2 | 2 | 5 | 4 | 5 | 5 |    |
| Oil VTC     | 741  | 0 | 1 | 2 | 1 | 4 | 3 | 5 | 5 |    |
|             | 33   | 0 | 1 | 1 | 1 | 1 | 1 | 1 | 1 |    |
|             | 96   | 1 | 2 | 2 | 1 | 2 | 2 | 3 | 3 |    |
|             | 133  | 0 | 0 | 0 | 1 | 5 | 3 | 5 | 5 |    |
|             | 231  | 4 | 4 | 4 | 4 | 5 | 5 | 5 | 5 |    |
|             | 256  | 3 | 1 | 3 | 1 | 4 | 3 | 5 | 5 |    |
|             | 268  | 0 | 0 | 1 | 0 | 3 | 1 | 3 | 1 |    |
|             | 373  | 3 | 1 | 3 | 2 | 5 | 3 | 5 | 5 |    |
|             | 409  | 0 | 0 | 1 | 1 | 1 | 1 | 2 | 3 |    |
| Juniper Oil | 411  | 2 | 2 | 2 | 2 | 3 | 3 | 4 | 4 |    |
| Control     | 538  | 3 | 2 | 3 | 3 | 3 | 3 | 5 | 5 |    |
|             | 29   | 1 | 0 | 2 | 2 | 4 | 3 | 5 | 5 |    |
| Juniper Oil | 111  | 3 | 2 | 3 | 2 | 5 | 3 | 5 | 5 |    |
| VTC         | 173  | 1 | 1 | 2 | 2 | 3 | 3 | 4 | 4 |    |

|            | 262   | 1 | 1 | 2 | 1 | 4 | 3 | 5 | 5 |
|------------|-------|---|---|---|---|---|---|---|---|
|            | 272   | 3 | 2 | 3 | 2 | 4 | 4 | 5 | 5 |
|            | 378   | 4 | 3 | 4 | 3 | 4 | 4 | 5 | 5 |
|            | 384   | 3 | 3 | 3 | 3 | 4 | 4 | 5 | 5 |
|            | 393   | 0 | 0 | 2 | 1 | 3 | 3 | 3 | 3 |
|            | 490   | 1 | 0 | 1 | 0 | 1 | 1 | 1 | 1 |
|            | 510   | 4 | 4 | 4 | 4 | 4 | 4 | 5 | 5 |
|            | 93    | 1 | 0 | 3 | 3 | 4 | 4 | 4 | 4 |
|            | 183   | 1 | 0 | 3 | 1 | 4 | 3 | 5 | 5 |
|            | 220   | 0 | 0 | 0 | 1 | 1 | 1 | 3 | 3 |
|            | 243   | 3 | 0 | 4 | 3 | 5 | 5 | 5 | 5 |
|            | 249   | 1 | 1 | 2 | 2 | 2 | 4 | 3 | 4 |
|            | 296   | 0 | 0 | 1 | 0 | 1 | 2 | 1 | 2 |
|            | 470   | 3 | 1 | 3 | 1 | 4 | 3 | 5 | 3 |
|            | 496   | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 0 |
| 2.5%       | 524   | 4 | 1 | 3 | 2 | 4 | 3 | 5 | 5 |
| Control    | 550   | 4 | 0 | 4 | 0 | 4 | 3 | 5 | 3 |
|            | 86    | 3 | 1 | 3 | 2 | 4 | 4 | 5 | 5 |
|            | 152   | 0 | 0 | 2 | 0 | 3 | 3 | 5 | 5 |
|            | 412   | 0 | 0 | 3 | 2 | 4 | 4 | 5 | 5 |
|            | 416   | 2 | 0 | 2 | 1 | 3 | 3 | 5 | 5 |
|            | 512   | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 |
|            | 515   | 2 | 4 | 4 | 4 | 5 | 5 | 5 | 5 |
|            | 2.5.C | 1 | 0 | 2 | 2 | 3 | 3 | 4 | 4 |
|            | 2.5.D | 1 | 1 | 2 | 2 | 4 | 4 | 5 | 5 |
|            | 2.5.E | 4 | 3 | 4 | 3 | 5 | 4 | 5 | 5 |
| 2.5% VTC   | 2.5.F | 3 | 3 | 3 | 3 | 4 | 4 | 5 | 5 |
|            | 56    | 0 | 0 | 0 | 0 | 3 | 3 | 3 | 3 |
|            | 68    | 0 | 0 | 1 | 0 | 3 | 1 | 4 | 3 |
|            | 210   | 1 | 0 | 2 | 1 | 3 | 3 | 4 | 4 |
|            | 217   | 4 | 1 | 4 | 3 | 4 | 5 | 5 | 5 |
|            | 279   | 1 | 0 | 2 | 2 | 3 | 3 | 4 | 4 |
|            | 291   | 0 | 0 | 1 | 1 | 2 | 3 | 4 | 4 |
|            | 317   | 0 | 0 | 2 | 2 | 4 | 3 | 5 | 5 |
|            | 397   | 0 | 1 | 0 | 2 | 0 | 3 | 1 | 4 |
|            | 424   | 3 | 0 | 3 | 0 | 4 | 1 | 5 | 3 |
| 5% Control | 544   | 0 | 0 | 1 | 0 | 2 | 0 | 2 | 0 |
|            | 89    | 2 | 0 | 3 | 2 | 3 | 3 | 4 | 5 |
| 5% VTC     | 124   | 0 | 0 | 1 | 0 | 2 | 1 | 2 | 2 |

|           |     |   |   |   |   |   |   |   | 9 |
|-----------|-----|---|---|---|---|---|---|---|---|
|           | 193 | 0 | 0 | 2 | 2 | 4 | 4 | 4 | 4 |
|           | 213 | 1 | 0 | 2 | 1 | 3 | 3 | 5 | 4 |
|           | 290 | 3 | 2 | 3 | 3 | 5 | 5 | 5 | 5 |
|           | 325 | 0 | 0 | 2 | 1 | 3 | 3 | 4 | 4 |
|           | 368 | 1 | 0 | 2 | 2 | 4 | 4 | 5 | 5 |
|           | 423 | 0 | 0 | 1 | 1 | 3 | 3 | 5 | 5 |
|           | 432 | 2 | 0 | 3 | 2 | 4 | 3 | 4 | 4 |
|           | 435 | 0 | 0 | 1 | 1 | 3 | 3 | 4 | 4 |
|           | 5   | 0 | 0 | 2 | 1 | 3 | 2 | 5 | 5 |
|           | 21  | 0 | 0 | 0 | 3 | 3 | 4 | 5 | 5 |
|           | 253 | 0 | 0 | 2 | 1 | 3 | 1 | 3 | 3 |
|           | 264 | 0 | 0 | 1 | 0 | 3 | 1 | 4 | 4 |
|           | 324 | 1 | 0 | 1 | 1 | 3 | 1 | 3 | 3 |
|           | 327 | 0 | 0 | 1 | 1 | 3 | 3 | 4 | 5 |
|           | 343 | 0 | 0 | 1 | 1 | 2 | 2 | 4 | 4 |
|           | 349 | 0 | 0 | 1 | 0 | 2 | 1 | 4 | 4 |
| 10%       | 532 | 0 | 0 | 1 | 1 | 3 | 3 | 5 | 3 |
| Control   | 535 | 0 | 0 | 0 | 1 | 2 | 1 | 2 | 2 |
|           | 13  | 0 | 1 | 3 | 3 | 4 | 4 | 5 | 5 |
|           | 72  | 0 | 0 | 2 | 2 | 4 | 3 | 4 | 4 |
|           | 181 | 0 | 0 | 0 | 0 | 2 | 0 | 3 | 2 |
|           | 301 | 0 | 0 | 1 | 1 | 3 | 4 | 4 | 4 |
|           | 309 | 1 | 0 | 1 | 1 | 2 | 3 | 2 | 3 |
|           | 376 | 1 | 1 | 1 | 1 | 1 | 1 | 2 | 1 |
|           | 387 | 0 | 0 | 2 | 1 | 4 | 3 | 5 | 5 |
|           | 388 | 0 | 0 | 1 | 0 | 3 | 3 | 4 | 4 |
|           | 418 | 0 | 0 | 1 | 1 | 3 | 3 | 4 | 4 |
| 10% VTC   | 511 | 0 | 0 | 1 | 1 | 2 | 3 | 2 | 3 |
|           | 561 | 0 | 0 | 2 | 0 | 3 | 1 | 4 | 4 |
|           | 562 | 0 | 0 | 1 | 0 | 3 | 3 | 5 | 5 |
|           | 564 | 0 | 0 | 0 | 0 | 3 | 1 | 3 | 3 |
|           | 568 | 1 | 0 | 2 | 1 | 4 | 3 | 5 | 5 |
|           | 574 | 2 | 2 | 2 | 2 | 4 | 3 | 4 | 4 |
|           | 577 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 1 |
|           | 578 | 0 | 0 | 0 | 0 | 3 | 3 | 5 | 5 |
|           | 922 | 0 | 1 | 2 | 2 | 3 | 3 | 4 | 4 |
| 10% Post  | 931 | 1 | 2 | 1 | 3 | 3 | 3 | 3 | 4 |
| Treatment | 935 | 0 | 0 | 1 | 0 | 3 | 1 | 4 | 3 |
| 10% Post  | 606 | 1 | 1 | 3 | 2 | 4 | 4 | 5 | 5 |

| VTC       | 654 | 2 | 2 | 3 | 3 | 4 | 4 | 5 | 5 |
|-----------|-----|---|---|---|---|---|---|---|---|
| Treatment | 660 | 2 | 0 | 2 | 1 | 4 | 2 | 5 | 5 |
|           | 664 | 0 | 1 | 1 | 2 | 1 | 3 | 2 | 3 |
|           | 667 | 3 | 1 | 3 | 3 | 5 | 5 | 5 | 5 |
|           | 677 | 2 | 2 | 3 | 3 | 4 | 4 | 5 | 5 |
|           | 189 | 2 | 2 | 3 | 2 | 4 | 3 | 5 | 4 |
|           | 692 | 4 | 3 | 4 | 4 | 5 | 4 | 5 | 5 |
|           | 804 | 4 | 0 | 4 | 2 | 5 | 4 | 5 | 5 |
|           | 811 | 3 | 3 | 4 | 4 | 5 | 5 | 5 | 5 |
| Pine      |     |   |   |   |   |   |   |   |   |
| control 1 |     | 4 | 4 | 5 | 5 | 5 | 5 | 5 | 5 |
| Pine      |     |   |   |   |   |   |   |   |   |
| control 2 |     | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 |
| Pine      |     |   |   |   |   |   |   |   |   |
| control 3 |     | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 |
| Pine      |     |   |   |   |   |   |   |   |   |
| control 4 |     | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 |
| Pine      |     |   |   |   |   |   |   |   |   |
| control 5 |     | 0 | 0 | 1 | 1 | 1 | 1 | 1 | 1 |
| Pine      |     |   |   |   |   |   |   |   |   |
| control 6 |     | 1 | 0 | 1 | 1 | 2 | 2 | 2 | 2 |

| weeks   |      |             |         |               |        |         |         |         |
|---------|------|-------------|---------|---------------|--------|---------|---------|---------|
|         | Tag  |             |         |               | 2      | 4       | 6       |         |
|         | #    | inoculation | 2 weeks | reinoculation | weeks  | weeks   | weeks   | 8 weeks |
| True    |      |             |         |               |        |         |         |         |
| Control | 000  | 0000 0      | 0002 7  | 6066 A        | 4024.0 | F 420 C | F 40C 4 |         |
| VTC     | 820  | 9899.9      | 9903.7  | 6066.4        | 4924.6 | 5438.6  | 5406.4  |         |
|         | 3001 | 10939.5     | 9858.2  | 9459          | 8676.8 | 6832.3  | 5262.6  | 5551.3  |
|         | 3002 | 8381.6      | 8189.3  | 6061.7        | 6265.5 | 3644.2  | 4153.7  | 12553.6 |
|         | 3003 | 6855        | 6736.1  | 5981.6        | 5988.6 | 5896.2  | 4578.6  | 9446.2  |
|         | 3004 | 8403.9      | 7288.6  | 6051          | 5766.5 | 4651.9  | 5522.5  | 6661.1  |
|         | 668  | 7358.4      | 7440.2  | 6327.3        | 6001.2 | 5957.9  | 3988.5  | 6280    |
|         | 3005 | 10469.8     | 10577.5 | 9894.2        | 7761.7 | 7487.8  | 7275.5  | 4756.2  |
|         | 854  | 9059.9      | 9733.2  | 6549.1        | 5288.6 | 3766.5  | 4252.8  | 4092    |
|         | 3006 | 6491.3      | 6998.4  | 5503.2        | 4012.5 | 4349.5  | 3726.2  | 5793.9  |
|         | 3007 | 6839.1      | 6618    | 5770.7        | 5071.1 | 4725.4  | 5022.8  | 6350.7  |
| True    |      |             |         |               |        |         |         |         |
| Control | 3008 | 7239.1      | 7514.7  | 6531.8        | 6572   | 5357.1  | 4851.8  | 8003.7  |
|         | 3009 | 7173.4      | 6616.8  | 6020.4        | 5483.8 | 4969.1  | 4387    | 6893.9  |
|         | 3010 | 6247.7      | 6107.8  | 6086.6        | 5280.8 | 4741.7  | 4274.3  | 5992.2  |
|         | 3011 | 5211.4      | 5165.8  | 4508.8        | 4577   | 3656.4  | 3474.2  | 5537.1  |
|         | 3012 | 4910.4      | 5071.1  | 4176.1        | 3820.2 | 3931.4  | 2817.6  | 5147.1  |
|         | 3013 | 7012.5      | 6507.2  | 6687.2        | 6612.6 | 5321    | 4751.7  | 7040.2  |
|         | 3014 | 5443        | 5539.8  | 4911.2        | 4835.8 | 4462.1  | 4177    | 5915.8  |
|         | 3015 | 5909.1      | 5999.4  | 5526.1        | 5247.3 | 5287.4  | 4087.4  | 6217.4  |
|         | 3016 | 5622.1      | 5363.7  | 4561.2        | 3780.4 | 3749.3  | 3156.6  | 5446.6  |
|         | 3017 | 6340.7      | 6219    | 6057.1        | 5589.7 | 4732.9  | 4528.7  | 6472.3  |
| J.O.    |      |             |         |               |        |         |         |         |
| VTC     | 3018 | 6123.2      | 6190.1  | 6261.9        | 4183.5 | 2314    |         |         |
|         | 3019 | 4667        | 5142.3  | 4776.8        | 3915   | -       |         |         |
|         | 3020 | 5321.3      | 5492.8  | 3928          | 3279.2 | 2131.7  |         |         |
|         | 3021 | 8110.3      | 8500    | 6065.8        | 5749   | -       |         |         |
|         | 803  | 7814.2      | 8283.8  | 5672.1        | 4401.1 | 3160.4  |         |         |
|         | 832  |             | 4238.7  | 4276.2        | 4111.3 | 3129    |         |         |
|         | 851  | 5856.2      | 5689.7  | 5407.5        | 2418.9 | -       |         |         |
|         | 3022 | 3524.9      | 3611.8  | 3035.3        | 1024.7 | -       |         |         |
|         | 672  | 8853.4      | 8629    | 6942.5        | 6036.3 | 3691.7  |         |         |
|         | 694  | 7515.6      | 7833.1  | 4956.6        | 5654   | 4480.1  |         |         |
| J.O.    |      |             |         |               |        |         |         |         |
| Control | 3023 | 4387.6      | 4493.9  | 3596.8        | 3588.5 | 3626.8  | 3161.2  | 5743    |
|         |      |             |         |               |        |         |         |         |

Table 5 – Modulus of elasticity values (MPa) for hybrid poplar veneers treated with essential oil formulations during exposure to brown rot fungi *G. trabeum* for eight weeks

|         |      |         |         |         |        |        |        | 94     |
|---------|------|---------|---------|---------|--------|--------|--------|--------|
|         | 3024 | 5724.5  | 5261.4  | 5186.8  | 4632.8 | 5189.2 | 4764.5 | 6428.8 |
|         | 3025 | 6939.8  | 6889.4  | 5958.7  | 6213.8 | 6068.3 | 5499.1 | 7898   |
|         | 3026 | 5651.3  | 5628.3  | 5104    | 4829   | 5081   | 4322.2 | 5585.9 |
|         | 3027 | 6199.2  | 6025.5  | 4808.1  | 5171.7 | 5276.9 | 4951   | 5922.7 |
|         | 3028 | 3366.8  | 3478.6  | 3198.5  | 3100.8 | 3141.1 | 3369.3 | 4120   |
|         | 3029 | 7062.1  | 6976.5  | 6134.4  | 6608   | 6661.8 | 5818.5 | 8146   |
|         | 3030 | 5490.4  | 5553    | 4647.9  | 4529.4 | 5179.2 | 4845.1 | 6201.6 |
|         | 3031 | 5614.9  | 4973.1  | 6381.7  | 4345.4 | 4491.4 | 3825.4 | 5717.4 |
|         | 3032 | 6444.6  | 5968.7  | 5424.1  | 5643.2 | 5239.3 | 4735.3 | 6599.3 |
| C.O.    |      |         |         |         |        |        |        |        |
| VTC     | 846  | 6924.3  | 7009    | 5293.4  | 5803.3 | 5994.5 | 5433.2 | 4374.9 |
|         | 843  | 8253.6  | 8666.5  | 6849.5  | 6241.8 | 4343.5 | 3766.5 | 5460.4 |
|         | 3033 | 7251.5  | 7161    | 5117.3  | 5732.5 | 5335.6 | 4961.9 | 5752   |
|         | 3034 | 6697.1  | 6656.5  | 4142.7  | 4880.8 | 4809   | 4615.2 | 3594.2 |
|         | 3035 | 5517.1  | 5510.7  | 4997.6  | 4662.8 | 4646.3 | 4049.7 | 4237.3 |
|         | 3036 | 9563.2  | 9463.4  | 6781.2  | 6668.4 | 5897.2 | 4456.3 | 4969.7 |
|         | 3037 | 6567.8  | 6249.8  | 5215.3  | 5735.4 | 5377.9 | 4176.3 | 4784.5 |
|         | 3038 | 7436.1  | 7379.4  | 5621.2  | 5094.2 | 5252.5 | 4864.8 | 3821.2 |
|         | 3039 | 13333.3 | 15230.1 | 10622.6 | 11198  | 9562.6 | 7848.2 | 4343.4 |
|         | 678  | 7656    | 8374.6  | 5158    | 5515.2 | 5724.9 | 4870.2 | 3674.8 |
| C.O.    |      |         |         |         |        |        |        |        |
| Control | 3040 | 2920.4  | 2895    | 2468.7  | 2469   | 2507.9 | 2297.6 | 1940.5 |
|         | 3041 | 3967.4  | 4154    | 3460.4  | 3220.5 | 3233.1 | 3162.1 | 2665.6 |
|         | 3042 | 7159.2  | 6366.9  | 6006.6  | 5188.6 | 4785   | 5083   | 3845.4 |
|         | 3043 | 6698.4  | 6384.2  | 5235.2  | 4569.1 | 4507.2 | 4216   | 3893.8 |
|         | 3044 | 5243    | 4545.5  | 3772.2  | 3684.6 | 3578.3 | 3388.6 | 2853.4 |
|         | 3045 | 6567    | 6080.1  | 5459.1  | 4676.6 | 5201.5 | 5389.6 | 3756.7 |
|         | 3046 | 6157.12 | 5675.7  | 5418.2  | 5066.2 | 5190.5 | 4436.2 | 3480.8 |
|         | 3047 | 5866.8  | 5319.4  | 4543.8  | 4381.3 | 4258.3 | 4176.1 | 3412   |
|         | 3048 | 7262.6  | 6996.4  | 6341.7  | 6087.4 | 6030.3 | 6493.5 | 4329.7 |
|         | 3049 | 4863.5  | 5035.8  | 4194.3  | 4206.7 | 4352.7 | 3902.2 | 3172.5 |
| 10%     | 2050 | 6504    | 600 A 0 | 5250.0  | 4654   |        |        |        |
| VTC     | 3050 | 6504    | 6994.9  | 5359.8  | 4651   | 5729.4 | 4814.7 | 443.4  |
|         | 3051 | 10207.1 | 10129.4 | 9110.6  | 4660   | 7417.4 | 6666.8 | 8409   |
|         | 841  | 4059.7  | 4511.8  | 4015.9  | 4161.6 | 3669.5 | 3891.5 | 4713.6 |
|         | 3052 | 4172.4  | 4250.6  | 3985.1  | 3905.6 | 3403   | 3431.9 | 4825.4 |
|         | 651  | 5192.6  | 5356.2  | 4774.9  | 3913   | 4512.9 | 3626   | 4524.1 |
|         | 649  | 5387.7  | 5702.6  | 4927.3  | 4184.5 | 4442.3 | 4001.5 | 4407.2 |
|         | 852  | 6967.4  | 6757.1  | 5935.2  | 5459.1 | 6814   | 3560.4 | 5159.8 |
|         | 830  | 6633.2  | 6808.1  | 4936.9  | 4466.6 | 5398.4 | 4280.3 | 4596.4 |

|         |      |        |        |        |        |        |        | 95     |
|---------|------|--------|--------|--------|--------|--------|--------|--------|
|         | 3053 | 8537.1 | 8774.8 | 6873   | 7101   | 6799.5 | 6772   | 7575.8 |
|         | 814  | 8438.2 | 8770.1 | 5203.4 | 5282   | 5393.2 | 3561   | 5985.9 |
| 10%     |      |        |        |        |        |        |        |        |
| Control | 3054 | 2924.3 | 3075.8 | 3001.7 | 2842.4 | 3057   | 2897   | 2738.3 |
|         | 3055 | 5976.1 | 5862.2 | 5371   | 5149.8 | 5430.4 | 5681.3 | 4448.3 |
|         | 3056 | 6294.7 | 6029.6 | 5055.8 | 4579.8 | 6079.5 | 4985.7 | 4991.9 |
|         | 3057 | 4831.1 | 4773.2 | 4129.5 | 4032.7 | 4150.4 | 3815.1 | 3479.5 |
|         | 3058 | 5711.6 | 5409.7 | 4564.3 | 4761.8 | 4738.9 | 4241.2 | 3959.4 |
|         | 3059 | 4032.4 | 4092.4 | 4063.2 | 3813.1 | 4228   | 4140.7 | 3542.6 |
|         | 3060 | 6147.1 | 6004.3 | 5867.5 | 5558   | 5853.2 | 5738.6 | 5137.6 |
|         | 3061 | 8424.4 | 8012.3 | 6681.5 | 6665.5 | 6979.7 | 6638.6 | 5997.4 |
|         | 3062 | 5995.4 | 5751.6 | 5366.1 | 4803.9 | 5284.3 | 4840.8 | 4577.3 |
|         | 3063 | 4848.6 | 5028   | 4802.9 | 4318.5 | 4670.9 | 4230.6 | 4181.8 |

reinocula Tag # inoculation weeks tion weeks weeks weeks weeks True Control VTC True Control J.O. VTC \_ \_ -J.O. Control 

Table 6 – Bending stiffness values (MPa x  $mm^4$ ) for hybrid poplar veneers treated with essential oil formulations during exposure to brown rot fungi *G. trabeum* for eight weeks

|         |      |        |        |                  |        |        |        | 97     |
|---------|------|--------|--------|------------------|--------|--------|--------|--------|
|         | 3024 | 591242 | 543411 | 559817           | 486572 | 524231 | 508126 | 499815 |
|         | 3025 | 704235 | 699121 | 684488           | 665806 | 609610 | 605426 | 602739 |
|         | 3026 | 562420 | 560131 | 532526           | 512937 | 508271 | 474469 | 461632 |
|         | 3027 | 636276 | 618448 | 583170           | 551354 | 553846 | 532248 | 502594 |
|         | 3028 | 394861 | 407973 | 410938           | 384542 | 373243 | 429224 | 351940 |
|         | 3029 | 733950 | 725053 | 705551           | 673618 | 690303 | 613309 | 607734 |
|         | 3030 | 561274 | 567674 | 530246           | 503826 | 520752 | 483912 | 464437 |
|         | 3031 | 608644 | 539074 | 744877           | 506672 | 507742 | 481319 | 480202 |
|         | 3032 | 625591 | 579394 | 550148           | 533173 | 492142 | 477248 | 495676 |
| C.O.    |      |        |        |                  |        |        |        |        |
| VTC     | 846  | 304584 | 308309 | 298583           | 302190 | 306467 | 314572 | 302462 |
|         | 843  | 212921 | 223573 | 212311           | 201981 | 198924 | 195529 | 191018 |
|         | 3033 | 379109 | 374377 | 353299           | 356662 | 337982 | 330508 | 375849 |
|         | 3034 | 232192 | 230785 | 217954           | 224618 | 214510 | 213101 | 213996 |
|         | 3035 | 392932 | 392476 | 397752           | 390075 | 368336 | 335476 | 386783 |
|         | 3036 | 166834 | 165093 | 166405           | 161872 | 169863 | 150521 | 147286 |
|         | 3037 | 415255 | 395149 | 390406           | 403443 | 380802 | 365771 | 384950 |
|         | 3038 | 209762 | 208163 | 222222           | 208944 | 202515 | 202146 | 194308 |
|         | 3039 | 274439 | 313481 | 302971           | 286167 | 301559 | 271773 | 306854 |
|         | 678  | 184400 | 201708 | 203352           | 199948 | 195200 | 187635 | 198474 |
| C.O.    |      |        |        |                  |        |        |        |        |
| Control | 3040 | 334302 | 331394 | 312096           | 312731 | 317084 | 314333 | 314801 |
|         | 3041 | 431161 | 451440 | 419377           | 388434 | 420046 | 409934 | 434899 |
|         | 3042 | 760451 | 676293 | 684253           | 614337 | 595270 | 628245 | 605260 |
|         | 3043 | 606241 | 577804 | 559739           | 489401 | 507210 | 465829 | 508802 |
|         | 3044 | 520441 | 451204 | 450094           | 421268 | 422601 | 424909 | 391411 |
|         | 3045 | 685735 | 634893 | 620201           | 538764 | 574311 | 602349 | 567035 |
|         | 3046 | 628680 | 579524 | 552330           | 544013 | 536809 | 506095 | 497915 |
|         | 3047 | 594722 | 539232 | 529218           | 492801 | 475093 | 472621 | 487900 |
|         | 3048 | 768923 | 740739 | 698694           | 682099 |        | 660915 | 657712 |
| 10%     | 3049 | 551333 | 570866 | 525176           | 502951 | 544429 | 490891 | 507187 |
| VTC     | 3050 | 354895 | 381682 | 359615           | 322585 | 337712 | 310199 | 31421  |
| VIC     | 3051 | 327270 | 324779 | 295183           | 288815 | 291010 | 288061 | 303171 |
|         | 841  | 136702 | 151926 | 145382           | 146400 | 146136 | 138015 | 133461 |
|         | 3052 | 259517 | 264381 | 277508           | 255001 | 259353 | 261374 | 256432 |
|         | 651  | 351008 | 362067 | 328473           | 289657 | 294388 | 267735 | 295845 |
|         | 649  | 341577 | 361541 | 328473<br>328545 | 320417 | 320888 | 302584 | 312742 |
|         | 852  | 192125 | 186326 | 185555           | 171661 | 188145 | 167336 | 186451 |
|         | 830  | 317282 | 325648 | 315842           | 323901 | 326043 | 280253 | 307416 |
| I       | 050  | 31/202 | 525040 | 513042           | 323901 | 320043 | 200203 | 307410 |

|         |      |        |        |        |        |        |        | 98     |
|---------|------|--------|--------|--------|--------|--------|--------|--------|
|         | 3053 | 306650 | 315188 | 303822 | 305258 | 302223 | 305485 | 306638 |
|         | 814  | 180848 | 187961 | 188116 | 184993 | 194648 | 167878 | 191324 |
| 10%     |      |        |        |        |        |        |        |        |
| Control | 3054 | 347812 | 365831 | 357559 | 360001 | 352213 | 347483 | 355017 |
|         | 3055 | 617227 | 605464 | 593240 | 554020 | 544541 | 580936 | 509787 |
|         | 3056 | 528079 | 505839 | 471456 | 419339 | 469710 | 416424 | 431512 |
|         | 3057 | 547461 | 540900 | 520247 | 512077 | 485106 | 473410 | 473261 |
|         | 3058 | 583036 | 552218 | 528405 | 512826 | 492297 | 468451 | 469308 |
|         | 3059 | 449079 | 455761 | 458390 | 451902 | 448768 | 443935 | 426135 |
|         | 3060 | 653208 | 638034 | 614615 | 593125 | 571677 | 565736 | 551284 |
|         | 3061 | 901328 | 857237 | 787946 | 742514 | 763467 | 735324 | 713066 |
|         | 3062 | 681732 | 654010 | 625161 | 588185 | 600995 | 541995 | 550696 |
|         | 3063 | 602703 | 625003 | 571607 | 526267 | 503037 | 493587 | 522865 |