Potential for Using Essential Oils to Protect Viscoelastic Thermal Compression–Treated Hybrid Poplar

Adam Scouse
Frederick A. Kamke
Jeffrey J. Morrell

Abstract
The potential for using pretreatment with cinnamon leaf oil or juniper foliage oil to improve the durability of viscoelastic thermal compression (VTC)–treated hybrid poplar was explored in a field termite test and a laboratory mold test. The addition of oils prior to VTC processing had variable effects on resistance to both mold and termite attack. Subsequent gas chromatography–mass spectroscopy assessment of residual oil components in VTC processed material suggested that the heating associated with the process was associated with substantial degradation of oil components that might help explain the lack of protective effect. The results suggest that further attempts to use essential oils to improve the durability of VTC processed materials must first explore less aggressive heating conditions to reduce the potential for thermal degradation of oil components while still achieving the attractive gains in physical properties associated with the process.

Hybrid poplar has been widely planted in temperate regions globally because of its rapid growth and uniform wood properties. Two negative characteristics of the resulting wood are its low density and minimal resistance to fungal or insect attack (Zabel and Morrell 1992, Bowyer et al. 2003).

One method for enhancing the properties of hybrid poplar is to densify the material, either through adding monomers for subsequent polymerization or by compressing the material. Polymer impregnation can be costly because of the high loadings of chemical required to produce effective decay resistance, making it uneconomical for most applications. Densification can be effective, but care must be taken to avoid damaging the material during compression. Viscoelastic thermal compression (VTC) was developed to improve the strength and stiffness properties of low density wood veneer by subjecting them to combinations of elevated temperature, steam, and compression (Kamke 2006, Kamke and Sizemore 2008, Kamke and Rathi 2011). The VTC process has been evaluated for densifying a number of materials and shows promise for increasing the value of lower density plantation materials. One property not enhanced by VTC treatment is durability, which may limit the potential applications of VTC treated material (Schwarze and Sypcher 2005, Skyba et al. 2009, Kutnar et al. 2010).

One method for improving the durability of VTC treated wood material would be to use essential oils from various plant materials as protectants. Essential oils have been examined in a variety of applications and, depending on the source, exhibit activity against a range of fungi and insects. In general; however, individual oils lack broad spectrum activity and tend to work best when used in combinations. One potential method for improving poplar durability might be to pretreat with mixtures of essential oils prior to VTC treatment to produce stronger, more durable materials.

Two possible oil candidates for this application are cinnamon leaf oil and juniper oil. Cinnamon leaf oil is a commercially available food grade extract from \textit{Cinnamomum osmophleum} that has been evaluated in a variety of tests on wood materials (Chang and Cheng 2002, Wang et al. 2005, Hsu et al. 2007, Li et al. 2008, Yen and Cheng 2008). It is relatively inexpensive and readily available. Western juniper (\textit{Juniperus occidentalis}) is an invasive species on much of the Great Basin in the Rocky Mountains of the Western United States and researchers have long
sought economic uses for its woody biomass in order to provide financial incentive for removal (Kurth and Ross 1954; Adams 1987a, 1987b). Extracts of juniper foliage and heartwood have been reported to have activity against a range of organisms (Adams et al. 1988, Sichamba 2012). The potential for using essential oils extracted from foliage as wood protectants might encourage further juniper removal efforts. One risk associated with using essential oils in VTC processing is that the elevated temperatures associated with the process will result in thermal degradation of oil components, thereby reducing treatment efficacy. However, developing such VTC treatments would provide an attractive option for protecting this material when it is exposed under adverse environmental conditions.

The objective of this research was to evaluate the effects of juniper oil or cinnamon leaf oil on the resistance of VTC treated hybrid poplar to fungal and termite attack.

Materials and Methods

Hybrid poplar

Hybrid poplar (Populus trichocarpa × Populus deltoides) was harvested in Boardman, Oregon, peeled into 3.5-mm-thick veneer by Columbia Forest Products (Klamath Falls, Oregon) and stored at 20°C and 65 percent relative humidity. For the following experiments, veneer specimens were cut to a final dimension of 3.5 by 26 by 140 mm (radial by tangential by longitudinal).

Essential oil formulations

Cinnamon leaf oil was purchased from OliveNation LLC (Charlestown, Massachusetts) and stored at −2°C until needed. Western juniper foliage (Juniperus occidentalis) was collected from the Deschutes National Forest approximately 2 km from Sisters, Oregon. Bough material, consisting of leaves, berries, and small branches (less than 15 mm in diameter) was collected from the bottom 1/3rd of the tree and stored at 20°C and 65 percent relative humidity. Batches of bough material were weighed, placed inside a distillation column and steam distilled for five hours. The resulting oil and water mixture was decanted for oil collection. The oil was then weighed to determine distillation yield on a wet weight basis and stored at −2°C. A total of 13 distillations were performed yielding approximately 200 g of western juniper oil. Oil yields averaged 0.66 percent.

Five ethanol (95%)-based treatment solutions were prepared from cinnamon leaf oil or western juniper foliage oil concentrates containing 10 percent cinnamon oil, 10 percent juniper oil, 1.25 percent each of juniper and cinnamon oil, 2.5 percent each of juniper and cinnamon oil, or 5 percent each of juniper and cinnamon oil. Control samples were treated with only 95 percent ethanol. An aqueous 5 percent boric acid equivalent solution of sodium octaborate tetrahydrate (DOT) served as a positive treatment control because it has been observed to have excellent activity against termites and limited activity against mold and stain fungi (Lloyd 1998).

Hybrid poplar treatment with essential oils

Oven-dry hybrid poplar veneer specimens were submerged in borate or essential oil solutions in a desiccator and a vacuum was drawn for 15 minutes and then released. Specimens were removed from the preservative treatment, blotted dry, weighed to determine treatment uptake, oven-dried at 50°C for 24 hours, and weighed.

VTC treatment

Half of the specimens in each treatment group were set aside, while the remaining specimens were subjected to VTC treatment (Scouse 2012). VTC processing occurred at 170°C in a sealed densification chamber that was equipped with a hot-press. Specimens were placed between the hot-press platens and steam was introduced into the densification chamber at 669 kPa (gage) for 30 seconds. Platens remained open without compression to allow the samples to be steamed for an additional 90 seconds. Steam was released from the chamber for a period of 30 seconds to reach atmospheric pressure. Specimens were then compressed radially from 3 mm to a target thickness of 1 mm at 170°C and the compression was maintained for 180 seconds, before a cooling stage reduced platen temperatures to 93°C over 120 seconds. After cooling, platen compression pressure was released and the platens opened. The densification chamber was then opened and specimens were removed.

Hybrid poplar treatment with essential oils following VTC processing

In one experiment, samples were treated with a preservative following VTC processing to determine if post-VTC application would be an effective alternative to the detrimental effects of heat and steam. In these cases, a 10 percent essential oil treatment composed of 5 percent cinnamon oil and 5 percent juniper oil in toluene (wt%) was delivered by vacuum impregnation. Toluene was used as a nonswelling delivery solvent in this particular experiment to avoid thickness recovery of the densified veneers following essential oil treatment.

Hybrid poplar bending strength

Specimens were subjected to nondestructive, three-point bending tests before and after VTC processing according to ASTM Standard D4761-05 (ASTM International 2011) using a Sintech MTS machine equipped with TestWorks II v2.11f software and an Omega LCCB-300lb load cell. Test specimens were positioned (flatwise—load applied in thickness direction) along a 100-mm span while 75 Newtons were applied at center span at a load rate of 5 mm/min. Specimen beam dimensions, load and displacement measurements were used to create individual stress/strain curves that were used to determine peak load (N), peak stress (MPa), and modulus of elasticity (MOE; MPa) for individual specimens.

Essential oil retention of specimens

The effect of VTC treatment on specimen essential oil detection was determined by extraction and analysis by gas chromatography–mass spectrometry (GC-MS). Veneer specimens were ground to pass a 30 mesh screen. One gram of the resulting dust was placed in a vial with 25 mL of methanol or hexane, sonicated for 3 hours at room temperature, and then allowed to stand for 48 hours before analysis.

The extracts were injected into a Shimadzu QP2010S GC-MS equipped with a 30-m-long 5HRX1-MS carbwax column (0.25-mm inside diameter). Injector temperature
was 275°C and oven temperature was raised from 50°C to 260°C at a rate of 2°C/min with helium as the carrier gas at 30 ml/min. Column flow was 1 ml/min with a linear velocity of 36.3 cm/s. Mass spectrometry scanned between 20 and 400 m/z. Compound identification was completed using the NIST08 Mass Spectral Library. The proportion of total peak area as a percentage of all peaks detected in a given analysis was used to assess relative abundance of an individual compound. Changes in these proportions were used to assess the effects of VTC processing on the stability of each compound.

**Termite resistance**

Resistance to Formosan termites (*Coptotermes formosanus*) was evaluated in a field test following a modification of the American Wood Protection Association (AWPA) Standard E21-06 (AWPA 2012a) at a site located near Hilo, Hawaii. The test pieces were smaller than those used for the standard, making it difficult to surrounding them with the nontreated wood typically used to encourage termite feeding. Instead, specimens were sandwiched between two sheets of 9-mm-thick, nontreated Douglas-fir plywood (600 by 600 mm) for exposure. Five samples from each treatment group were glued to the face of one Douglas-fir plywood panel using a glue stick to secure samples, and then a second sheet was laid on top of the array to sandwich specimens into place. The plywood boards were bolted together to drive into the ground to encourage termite attack.

Feeder stakes were driven into the ground in the spaces between the concrete blocks and the plywood was placed on the blocks and covered as per the AWPA E21-06 standard to minimize the risk of wetting (AWPA 2012a). Termites had access to all samples for 6 months. Termite damage was visually assessed using a rating system of 10 to 0 (no mold) to 5 (complete coverage) according to AWPA Standard E24-06. Termites had an initial density of 330 kg/m³ and an average MOE of 7.51 GPa. Density and MOE increased to 980 kg/m³ and 27.3 GPa, respectively, following VTC processing. Specimen dimensions increased 0.8 mm (3%) in the transverse direction, while radial thickness was reduced 68 percent from 3.53 to 1.14 mm. The degree of densification and increase in MOE values were similar to those reported by Kutnar et al. (2008).

**Results and Discussion**

**Bending strength**

The hybrid poplar veneers had an initial density of 330 kg/m³ and an average MOE of 7.51 GPa. Density and MOE increased to 980 kg/m³ and 27.3 GPa, respectively.

**Mold resistance**

Resistance to mold growth was evaluated following the AWPA Standard E24-06 (AWPA 2012b) mold box test. Ten specimens per treatment group were suspended over soil inoculated with a suspension containing *Trichoderma spp.* (Pers.:Fr), *Asperillus niger* (Tiegh.), *Penicillium citrinum* (Thom), and *Alternaria alternata* (Fr.:Fr.) Keissl.). Southern pine sapwood was included as a mold susceptible species as specified in the standard. The samples were incubated at 28°C in a high humidity environment that encouraged condensation. The tangential surfaces of each specimen were visually evaluated at 2-week intervals over an 8-week period for degree of mold attack on a scale from 0 (no mold) to 5 (complete coverage) according to AWPA Standard E24-06.
extract sample, and the absolute amount of extractable material may change due to volatilization or creation of thermal degradation products. However, decreases in the relative abundance of a compound of interest in comparison to other detected compounds provide evidence of the thermal or hydrolytic stability.

The results obtained using pure cinnamon oil were consistent with previous reports and indicated loss of cinnamaldehyde with VTC processing (Wang et al. 2005). Western juniper oil contained a number of components including α-pinene, sabinene, car-3-ene, p-cymene, camphor, linalool, terpinen-4-ol, and bornyl acetate that 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).

Many of the components observed in pure juniper and cinnamon oil were also observed in extracts from vacuum-impregnated hybrid poplar veneers, indicating that vacuum impregnation was successful at treating poplar with the desired components. While it was not possible to determine the absolute quantity of individual compounds lost or retained, certain general trends were observed.

Peak area abundance of selected components decreased markedly in extracts from VTC treated veneers. Cinnamaldehyde peaks in extracts from veneer specimens treated with 10 percent cinnamon oil represented 13.8 percent of the total peak area prior to treatment and only 3.0 percent of total peak area after treatment (Figure 1). All compounds observed in western juniper leaf oil decreased in abundance following VTC processing, although several decreased to a lesser extent (notably bornyl acetate and terpinen-4-ol; Fig. 2). These decreases may reflect complete or partial degradation, but indicate that the heat associated with the VTC process was detrimental to the essential oil components.

Analysis of extracts from veneers treated with 2.5, 5, or 10 percent essential oil combinations, but not subjected to VTC treatment, showed increases in peak area abundance for terpinen-4-ol, cinnamaldehyde, and bornyl acetate with increasing oil treatment concentrations (Fig. 3). This pattern was not witnessed in treated veneers that had undergone VTC processing, reinforcing the assumption that VTC processing negatively affected the essential oil components.

Instability during VTC processing sharply reduces the value of an essential oil treatment. While it might be possible to use higher concentrations of extracts to account for this loss, costs would increase while the heat degraded essential oil components may have other negative effects on the properties of the finished materials.

Termitest

Hybrid poplar veneers sandwiched between two pieces of Douglas-fir plywood and exposed to Formosan termites for 6 months in Hilo, Hawaii, experienced varying levels of termite attack, but the results still allowed for performance assessments. The upper and lower plywood layers were heavily attacked as were pine sapwood control samples placed around the edges of the panels. These results indicated that conditions were suitable for aggressive termite attack. The samples were protected from wetting, but termite workers can carry moist soil from the ground to the wood. As a result, sample masses were sometimes above their original values at the end of the test; however, this effect was very variable and did not appear to be related to the original treatment.

Non-VTC-processed veneers with no oil treatment experienced the heaviest termite attack, with a median rating of 4 (Table 2). VTC processing of hybrid poplar veneer appeared to increase resistance to termite attack although the effect was not always consistent. Median termite damage ratings ranged from 4 to 8 for VTC-treated veneer, while mass loss due to termite attack decreased from 40 to 10 percent. Density is generally not a good predictor of resistance to termite attack, suggesting that changes associated with heating during VTC processing rendered the wood less attractive to attack.

Non-VTC-treated veneer specimens pretreated with 10 percent juniper oil were most resistant to termite attack, with a median rating of 10 and little variation in attack between specimens. Mass loss due to termite attack was also low, with an average 10 percent mass loss. These results support previous research showing juniper oil to be strongly anti-termitic (Adams et al. 1988, Clark and McChesney 1990, Sichamba 2012). Other treatments that performed

![Figure 1.—Relative abundance of cinnamaldehyde in extracts of hybrid poplar veneers treated with cinnamon leaf oil and extracted before or after viscoelastic thermal compression (VTC) processing.](image1)

![Figure 2.—Relative abundance of selected western juniper leaf oil components in extracts from nontreated and viscoelastic thermal compression (VTC)–treated hybrid poplar veneers.](image2)
well against termite attack included DOT and a 10 percent combination of juniper and cinnamon oil (Table 2). DOT has well known abilities to restrict attack by Formosan termites when used at the proper level. These results affirm that effect and showed that heating had little impact on the efficacy of this inorganic compound. The remaining treatments provided more variable protection against termite attack.

Increasing concentrations of essential oil resulted in improved termite protection. Median termite rating values for non–VTC-treated veneers impregnated with 2.5, 5, or 10 percent 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 percent. Veneers treated with 10 percent essential oil in toluene following VTC processing exhibited little resistance to termite attack. This was attributed to the limited ability of the toluene-based treatment solution to move into the veneers. Post VTC processing treatment was explored as a method to avoid the detrimental effects of heating; however, the inability to deliver an adequate amount of essential oil limited this approach.

While the modification of the test method to use thin veneers surrounded by plywood in place of larger blocks surrounded by solid wood may have produced more variable termite attack, the overall results indicated that the use of juniper oil or cinnamon leaf oil improved resistance to termite attack, but the effect was reduced by VTC processing.

**Mold tests**

Exposure to mold attack for 8 weeks produced nearly complete discoloration of untreated pine sapwood, indicating conditions were suitable for fungal growth (Table 3). Most of the poplar samples experienced progressive mold attack over the 8-week period.

VTC-treated veneers were smoother and darker in appearance than their nonprocessed counterparts regardless of whether they had been pretreated with DOT or an essential oil mixture. This darker color made it more difficult to visually rate the samples for mold coverage, but the procedures were to assess mold coverage, not surface darkness.

VTC-processed samples tended to be more susceptible to mold (Table 3). The heat associated with the VTC process may have enhanced mold growth by degrading hemicelluloses, producing more free sugars that would support enhanced fungal growth (Boonstra and Tjeerdsma 2006, Zabel and Morrell 1992). The 5 percent DOT treatment served as a positive treatment and nearly completely inhibited surface mold growth. DOT is susceptible to water leaching and the steam and pressure present during VTC processing might be expected to result in some chemical loss. However, VTC processing did not affect DOT effectiveness. DOT appeared to be a likely candidate for decreasing mold susceptibility of VTC wood.

Of the essential oil treatments tested, only non–VTC-processed specimens treated with 10 percent cinnamon oil exhibited any resistance to mold growth compared with controls. Mold ratings decreased from 3.5 for nontreated veneers to 1 for the 10 percent cinnamon oil treatment. The effectiveness of cinnamon oil reinforces previous research (Chang and Cheng 2002, Wang et al. 2005, Li et al. 2008).

The 10 percent cinnamon oil treatment was less effective when specimens were subjected to VTC processing. Median surface ratings of VTC processed veneers receiving cinnamon oil treatment increased from 1 to 4. The high temperature (170°C) and steam pressure associated with VTC processing would have the dual effect of thermally degrading cinnamaldehyde and increasing the availability of carbohydrates for fungal growth (Wang et al. 2005). The steam injection step during the VTC process may have also encouraged cinnamaldehyde migration from the wood.

Regardless of essential oil composition, the remaining preservative treatments had little effect on mold surface ratings. 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 relatively high median surface ratings (rating = 4).

Combinations of essential oils did not inhibit mold growth more than any single essential oil treatment. 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 failed to produce any synergistic effects against mold fungi.

**Conclusions**

VTC treatment enhanced the physical properties of hybrid poplar veneers, but addition of cinnamon leaf oil or juniper foliage extracts prior to VTC processing had inconsistent effects on resistance to termite and fungal attack. Analysis of samples after VTC processing suggested that the lack of improvement in resistance to biological attack was related to thermal destruction of oil components. Further attempts to use natural products to enhance durability will need to consider employing less aggressive pressing conditions that limit this thermal effect and retain oil activity.

**Acknowledgments**

Funding for this study was provided by the USDA Wood Utilization Special Research Grant (2010-34158-20790) and the Oregon State University Green Building Materials Laboratory.

**Literature Cited**


**Table 3.**—Degree of surface mold on hybrid poplar specimens with or without an initial preservative treatment or viscoelastic thermal compression (VTC) processing and exposed in an AWPA E24 mold box test.

<table>
<thead>
<tr>
<th>Treatment</th>
<th>VTC processing (+/-)</th>
<th>4 wk</th>
<th>6 wk</th>
<th>8 wk</th>
<th>Mold rating*</th>
</tr>
</thead>
<tbody>
<tr>
<td>None</td>
<td>–</td>
<td>2.0 ± 1.5</td>
<td>3.0 ± 1.5</td>
<td>3.5 ± 1.4</td>
<td></td>
</tr>
<tr>
<td>5% DOT</td>
<td>–</td>
<td>2.0 ± 0.8</td>
<td>4.0 ± 0.7</td>
<td>5.0 ± 0.8</td>
<td></td>
</tr>
<tr>
<td>10% Juniper oil</td>
<td>–</td>
<td>0.0 ± 0.4</td>
<td>0.0 ± 0.6</td>
<td>0.0 ± 0.6</td>
<td></td>
</tr>
<tr>
<td>10% Juniper oil</td>
<td>+</td>
<td>0.0 ± 0.8</td>
<td>0.0 ± 0.8</td>
<td>0.0 ± 0.6</td>
<td></td>
</tr>
<tr>
<td>5% Juniper/cinnamon oils</td>
<td>–</td>
<td>1.0 ± 0.6</td>
<td>3.0 ± 0.8</td>
<td>4.0 ± 0.9</td>
<td></td>
</tr>
<tr>
<td>5% Juniper/cinnamon oils</td>
<td>+</td>
<td>1.5 ± 1.2</td>
<td>3.0 ± 1.4</td>
<td>4.5 ± 1.5</td>
<td></td>
</tr>
<tr>
<td>5% Juniper/cinnamon oils</td>
<td>+</td>
<td>2.0 ± 1.1</td>
<td>4.0 ± 1.0</td>
<td>5.0 ± 1.3</td>
<td></td>
</tr>
<tr>
<td>10% Juniper/cinnamon oils</td>
<td>–</td>
<td>2.0 ± 1.4</td>
<td>3.0 ± 1.6</td>
<td>4.0 ± 1.6</td>
<td></td>
</tr>
<tr>
<td>10% Juniper/cinnamon oils</td>
<td>+</td>
<td>2.0 ± 1.2</td>
<td>4.0 ± 1.1</td>
<td>5.0 ± 1.2</td>
<td></td>
</tr>
<tr>
<td>5% Juniper/cinnamon oils</td>
<td>–</td>
<td>1.0 ± 1.2</td>
<td>3.0 ± 1.3</td>
<td>4.0 ± 1.4</td>
<td></td>
</tr>
<tr>
<td>5% Juniper/cinnamon oils</td>
<td>+</td>
<td>2.0 ± 0.9</td>
<td>3.0 ± 0.9</td>
<td>4.0 ± 0.90</td>
<td></td>
</tr>
<tr>
<td>10% Juniper/cinnamon oils</td>
<td>–</td>
<td>1.0 ± 0.7</td>
<td>2.5 ± 0.9</td>
<td>4.0 ± 1.0</td>
<td></td>
</tr>
<tr>
<td>10% Juniper/cinnamon oils</td>
<td>+</td>
<td>1.0 ± 0.8</td>
<td>3.0 ± 1.1</td>
<td>4.0 ± 1.2</td>
<td></td>
</tr>
<tr>
<td>10% Juniper/cinnamon (post-VTC)</td>
<td>+</td>
<td>3.0 ± 1.0</td>
<td>4.0 ± 1.1</td>
<td>5.0 ± 0.8</td>
<td></td>
</tr>
<tr>
<td>Pine control</td>
<td>–</td>
<td>3.0 ± 2.1</td>
<td>3.5 ± 1.9</td>
<td>3.5 ± 1.9</td>
<td></td>
</tr>
</tbody>
</table>

* Values represent sample mean ratings ± one standard deviation on 10 replicates for all but the pine control, which had 5 replicates. Mold ratings range from 0 (no mold) to 5 (complete coverage).


