DECAY RESISTANCE AND PHYSICAL PROPERTIES OF OIL HEAT TREATED ASPEN WOOD

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The decay resistance of oil-heat treated aspen wood (*Populus tremula* I.) against white rot fungi (*Coriolus versicolor*) and brown rot fungi (*Coniophora puteana*) was investigated. Three different temperature stages and two time levels for oil heat treatment for the selection of optimum conditions were determined. Linseed oil as a heating medium was used. The mass loss of treated samples that were exposed to both fungi was significantly lower than that of the control samples. Results also showed improvement in dimensional stability after oil heat treatment. Decay resistance and dimensional stability of aspen wood were increased significantly with temperature increasing, but time seemed to have no effect on those properties. Oil heat treatment is a suitable method to improve decay resistance of aspen wood as it reduced the mass loss by 71% and 77% against *Coriolus versicolor* and *Coniophora puteana* compared with control samples, respectively. On the other hand, oil heat treatment improved the dimensional stability by about 20.5%.

Keywords: Oil heat treatment; Coriolus versicolor; Coniophora puteana; Linseed oil; Physical properties

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INTRODUCTION

Wood as a renewable natural material has good properties as well as some limiting factors such as dimensional instability and biodeterioration. Wood thermal treatments are useful and environment friendly mechanisms that can improve some wood properties like dimensional stability, durability, and sometimes color. Several studies have reported that the equilibrium moisture, swelling, and shrinkage of wood improved/ decreased with heat treatment (Stamm et al 1946; Kollmann and Schnider 1963; Burmester 1972; Hillis 1984). Also several studies reported increasing resistance to fungal attack by heat treatment (Dirol and Guyonnet 1993; Troya and Navarrete 1994; Leithoff and Peek 2001; Mazela et al 2004). The thermal modification of wood has long been recognized as a potentially useful method to improve the dimensional stabilization of wood and increase its decay resistance (Hill 2006). Some vegetable oils can be suitable as heat transfer media for thermal modification, since the boiling point of many vegetable oils is higher than 260°C (Gunstone 2002). Therefore the use of these oils as a heating medium is conceivable. Oil heat treatment is the process commercially used in Germany. In 2007 Thermoholz and Menz holz produced 4000 and 800 m³ oil heat treated wood, respectively (Estsvens and Periera 2009). This method is considered as an efficient, environment friendly wood treatment because natural plant oils, such as linseed oil, are used as a natural heating medium. Hot linseed oil causes equal and fast heat transfer in wood and it can improve the physical properties and durability of wood. In addition, linseed oil has a suitable smoke point and the tendency to polymerization, and these are important parameters of oil for heat treatment (Rapp and Sailer 2001). Leithoff and Peek (2001) also reported that oil heat treatment at the temperature only above 170°C can enhance the durability of bamboo. Hydrophobic characteristics and resistance against biological attack of thermal-oil treatment not only benefits from the heat treatment, but also from the thin coated layer formed by water repellent natural oils (Wang 2007). The main objectives of this study were the determination of effects of oil heat treatment on some physical properties of treated aspen wood, and to investigate the decay resistance of oil heat treated fast growing aspen wood.

MATERIALS AND METHODS

To investigate the decay resistance and changes in wood's physical properties during the heat treatment, hot linseed oil was used as heating medium. Small wooden blocks were treated at three temperatures stages and two time levels according to Table 1.

Table 1. Trocess Falameters for On freat freatment Aspen wood		
Temperature	Time	
100%C	4.5 Hours	
190 C	6 Hours	
205°C	4.5 Hours	
	6 Hours	
220°C	4.5 Hours	
220 C	6 Hours	

Table 1. Process Parameters for Oil Heat Treatment Aspen Wood

Wooden Samples

In this study three aspen trees were cut, and samples were selected for tests. Twenty samples were cut from each tree for the physical test, and 6 samples from each tree were cut for biological durability measurements. Wood samples were prepared from sapwood of 20 years old aspen plantation, cut into small sizes $50 \times 20 \times 20$ cm and $20 \times 20 \times 20$ cm for WPG, decay and physical tests respectively. The samples were clear, without any cracks, decay, or knots. The moisture content of samples before the treatment was about 6%.

Oil Heat Treatment of Samples

The samples were dried in an oven before the heat treatment to ease the oil heat transfer capacity. The double-decker tank (Fig. 1) was used to treat the samples. In the first stage, raw linseed oil was heated in the upper tank to keep the desired process temperature. Wooden samples were placed in the bottom tank in nitrogen atmosphere (3-5% air). In the second stage, hot linseed oil was transmitted to the bottom tank and heated to reach the desired temperature. Heat treatment started when the oil temperature reached the target temperature. After the main stage of treatment, vacuum pressure (0.1 atmosphere) was applied to remove the surplus and remaining oil from the samples. The specimens cooled off and were kept for four weeks in room temperature; then tests were performed.



Fig.1. oil heat treatment double decker tank

Physical Tests

Selected physical properties of oil heat treated aspen wood were measured in accordance with terms and conditions of ISO standards. The tested properties were weight percent gain (WPG), moisture content (MC), water absorption (WA), and shrinkage of samples. Moisture content and shrinkage of samples were recorded according to ISO 3130 and ISO 4858 standards.

Biological Durability Tests

The decay resistance of oil heat treated aspen wood against white rot (*Coriolus versicolor*) and brown rot (*Coniophora puteana*) were measured according to DIN EN 113 standards. A pair of block control and treated samples were incubated in Kolle flasks for 16 weeks. Then wood samples were taken out from the incubator and re-dried and re-weighed. For biological durability evaluation the mass loss percentages of the samples were measured.

Statistical Analysis

At least six replicates were used for each test. All data were statistically analyzed using two-way analysis of variance (ANOVA), and the mean values of measured properties were compared using Duncan's Multiple Range Test (DMRT) to determine the differences between treatment conditions.

RESULTS AND DISCUSSION

Physical Properties

Weight percent gain

Table 2 lists the weight percentage gain (WPG) for oil heat treated samples. Values of WPG of samples did not show any significant effects of time or temperature. WPG of samples after treatment are shown in Fig. 2. The measured WPG was about 83.9% to 86.2%. This high value of WPG after oil heat treatment of wood is the major disadvantage of this method.

The increment of weight after oil heat treatment presented by Sailer and Rapp (2001) was about 42 to 51% and 10 to 18% WPG in oil heat treated pine and spruce wood, respectively. Spear et al. (2006) also reported 89.9% and 87.4% WPG percentage in pine wood after oil heat treatment with linseed oil at 190°C and 200°C, respectively. It seems a high value of WPG in this study results from the small size of samples. Jones et al. (2005) reported that longer samples of sitka spruce have lower WPGs. Also, aspen is a hardwood with wide cells and is easy to penetrate (Rowel 1984). This high level of weight percent gain could be related to anatomical changes of samples too. Hietala et al. (2002) found that the size of wood pores was increased by heat treatment, perhaps because of the removal of cell wall components. Also Boonstra et al. (2006) reported that hardwood species such as beech and poplar are sensitive to collapse of vessels and deformation on the libriform fibers near the vessels.



Fig. 2. Weight Percent Gain of wood after oil heat treatment

Equilibrium moisture content (EMC)

The equilibrium moisture content of the control and treated samples are summarized in Table 2 and Fig. 3. It can be seen that oil heated treatment resulted in improved, lower EMC. However, the highest reduction in EMC percentage was obtained in wood treated at 220°C for 4.5 hours. The results of this study suggest that EMC of heat-treated samples was significantly less when the temperature was higher than 190°C, but increasing the length of the time of treatment was not affecting EMC. The highest EMC percentage was 7.1% in the control samples, which was reduced to 4.1% in the samples that were treated at 205 °C for 6 hours and 220°C. Hence the maximum 42% reduction in EMC due to oil heat treatment was recorded. Viitaniemi and Jämsä (1996, 2001) stated that heat treatment decreased the EMC of wood significantly. Jäsmä and Viitaniemi (2001) reported that lower EMC was a result of chemical changes and decrease of hydroxyl groups of cell wall. Similar reduction in EMC percentages were reported for oil heat treated softwoods by Sailer and Rapp (2001). Wang and Cooper (2005) noted that oil heat treatment decreased the EMC of wood. Salim et al. (2010) also reported that semantan bamboos that were treated in oil at 220°C had a lower EMC. Minor reduction in EMC after oil heat treatment at 190°C was reported by Epmeier et al. (2004).

Treatments (Time/Temperature)	WPG	EMC	Vol. Shrinkage	WA
control		7.1±0.8 c	13.20±0.61 e	99.99±7.56 c
4.5/190	86.2±3.1 a	4.9±0.5 b	12.19±0.26 d	29.19±3.54 b
6/190	86.2±8.5 a	4.9±0.6 b	12.06±0.28 d	28.82±4.13 b
4.5/205	86.2±4.3 a	4.3±0.4 a	10.73±0.24 c	27.18±3.37 b
6/205	85.3±4.5 a	4.1±0.3 a	10.67±0.19 bc	27.48±3.41 b
4.5/220	83.9±3.2 a	4.1±0.2 a	10.47±0.25 a	23.46±2.81 a
6/220	85.7±2.8 a	4.1±2.8 a	10.53±0.25 ab	22.35±3.34 a

Table 2. Physical Properties of Treated and Untreated Aspen Wood

- Values within the same column followed by different letters are significantly different at P<0.05

Volumetric shrinkage

Table 2 and Fig. 4 show comparatively, the volumetric shrinkage of oil heat treated wood and the control samples. The analysis of variance showed significant influence of oil heat treatment on the shrinkage of wood. The shrinkage of heat-treated samples was significantly lower when the temperature was higher. The volumetric shrinkage of oil heat treated wood at 220°C was about 10.5%. This means more than 20% reduction was obtained.



Fig. 3. Equilibrium moisture content of the control and treated samples

Fig. 4. Volumetric shrinkage of control and heated samples

According to Stamm (1964) wood that is heated at 320°C for one minute becomes more dimensionally stable. Tjeerdsma et al. (1998) found that heat treatment of wood at over 150°C reduced the shrinkage of wood and improved the dimensional stability. Similar observations have been reported by Syrjanen (2001). Also previous studies have reported oil heat treatment of wood (Sailer and Rapp 2001; Wang and Cooper 2005) and bamboo (Salim et al. 2010) reduced the shrinkage rate. These results suggest that the hemicelluloses content and OH groups were reduced at a high temperature, which would possibly reduce the shrinkage of wood (Jamsa and Viitaniemi 2001). The improvement of the dimensional stability can be explained by high reduction of hemicellulose content during heat treatment too (Burmester 1975).

Water absorption

One of the main effects of heat treatment of wood was reduction of wood hygroscopicity and water absorption. Oil heat treated aspen wood showed decreased water absorption compared with the control samples. Figure 5 and Table 2 show the average values of water absorption of the specimens. The results depicted here indicate that lower values of the water absorption (22.9%) were reached when the treatment temperature was 220°C, and highest water absorption was measured in the control samples (99.99%); this means that about 77.1% reduction occurred. It is important to note that ANOVA showed the water absorption influence by temperature of treatment, and no significant effect of time increments on water absorption were seen. Hofland and Tjeerdsma (2005), and Hyvonen et al. (2005) stated that the heat treatment of wood with rapeseed and tall oil decreased the water absorption. It could be noted that the size of the linseed oil molecule as a non swelling chemical is too large to penetrate in the cell wall. Hence, during impregnation most of the oil remains in the cell lumens (Olsson et al. 2001; Hill 2006). Also, it was found that drying oils formed an outer shell by penetration of water-repellent oil into the wood during the thermal treatment (Wang 2007).



Fig. 5. Water absorption (WA) of wood before and after oil heat treatment

Decay resistance

Results of the laboratory malt extract agar tests of oil heat treated and control specimens are listed in Table 3. After 16 weeks of exposure to the *Coriulus versicolor* and *Coniophora puteana* mass loss of control samples was significantly higher than that of the treated samples. In relative terms, good durability increment against both selected fungi was obtained after oil heat treatment. The best results were achieved in the sample that was treated in the highest temperature. Results show that temperature increment improves the durability of wood against decay, but increasing the time of treatment had no statistically significant effects on wood resistance against fungi. According to ANOVA, no interaction effect of time-temperature was seen. The results also exhibited 71% and 77% reduction in mass loss against *C.versicolor* and *C.puteana*, respectively,

after oil heat treatment at 220°C. Figure 6 compares the mass loss of treated and untreated wood after exposure to both *Coriolus versicolor* and *Coniophora puteana*.

Table 3. Treated and Untreated Wood Durability Against Coriolus versicolor and Coniophora puteana

Treatments	Mass loss (%)	Mass loss (%)
(Time/Temperature)	Coriolus versicolor	Coniophora puteana
control	33.64±4.36 c	38.09±4.21 e
4.5/190	13.50±0.75 b	15.28±1.62 cd
6/190	14.02±1.12 b	16.09±2.23 d
4.5/205	11.24±1.15 a	13.85±2.38 bc
6/205	11.08±0.93 a	12.81±1.63 b
4.5/220	9.86±0.98 a	8.43±1.80 a
6/220	9.79±0.68 a	8.43±1.43 a

- Values within the same column followed by different letters are significantly different at P<0.05

Tjeerdsma et al. (2002) reported good correlation between increment of decay resistance and hygroscopicity of heat treated wood. Based on the findings of Nuoponen (2005) it is possible to say the reduction of hydroxyl groups in holocellulose and chemical changes and cross linking of lignin are important parameters that enhance the equilibrium moisture and biological durability of thermally treated wood.



Fig. 6. Wood durability against *Coriolus versicolor* and *Coniophora puteana* before and after oil heat treatment

Dirol and Guyonnet (1993) studied the effects of wood heat treatment at temperatures between 205°C and 260°C of three less durable species containing poplar wood on resistance to several fungi such as *Coriolus versicolor* and *Coniophora puteana*. They reported in all cases mass loss of treated wood was under 1% against 40% mass loss for untreated samples. Sailer et al. (2000) revealed that oil heat treatment at temperatures of 190°C to 220°C improved the wood resistance to brown rot *Coniophora puteana*. They reported decreasing the mass loss from 48% and 40% to about 11% and 5.5% in

pine and spruce wood respectively. Comparison between several heat treatment processes showed that oil heat treatment can improve durability of spruce and pine wood against white rot *Coriolus versicolor* and brown rot *Coniophora puteana*, too (Welzbacher and Rapp 2002). Also, Leithoff and Peek (2001) found that temperatures above 170°C were effective to enhance the durability of two bamboo species.

CONCLUSIONS

The results of this study showed that oil heat treatment of plantation aspen wood can significantly improve the physical properties, such as equilibrium moisture content and volumetric shrinkage of wood, and increase the decay resistance against wood destroying fungi; however, according to the Findlay criterion, it seems that oil-heat-treated aspen does not have enough resistance for ground-contact uses. In regard to high WPG values, oil uptake is an important disadvantage of this method. The volumetric shrinkage of treated wood was inversely proportional to the treatment temperature. This study also showed that when aspen wood was subjected to the higher temperature, it became more resistant against white rot (*Coriolus versicolor*) and brown rot (*Coniophora putea*) fungi. Results also indicated that increasing the time of the heat treatment process from 4.5 to 6 hours in small samples did not have any significant effects on measured physical parameters and durability of the aspen wood. The high temperature has significant effects not only on the physical properties of wood, but also on biological durability.

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Article Submitted: August 16, 2011; Peer review completed: September 24, 2011; Revised version received: December 17, 2011; Accepted: December 19, 2011; Published: January 8, 2012.