# MOISTURE PROPERTIES AND BIOLOGICAL RESISTANCE OF HOT OIL TREATED WOOD

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### Abstract

Thermal modification of wood is one approach to improving the dimensional stability and the biological resistance of wood, without serious reduction of the mechanical properties. Use of a heat transfer fluid ('oil") provides the potential to enhance the quality of the treatment by controlled absorption of the heat transfer fluid and potentially by the supplementation of the oil treatment with wood protective chemicals dissolved or suspended in the fluid. This report first presents the results of moisture properties of hot oil treated wood. The main results are: Slack wax and palm oil are better than soy oil or other heating media used in this investigation in improving the moisture performance of thermally treated wood; 220°C is superior to 200°C, with 4 h being generally better than 2 h treatment. This report also gives the results of fungi resistance from soil block test on six oil treated spruce against Gloeophyllum trabeu (GT) and Postia placenta (PP), as well as the hot oil treated red pine and aspen against three species of mould: Trichoderma pseudokoningii (TP), Gliocladium virens (GV) and Aspergillus niger (AN). The results are: Hot oil treated wood has much higher decay and mould resistance than control samples; The treatment at 220°C for 4 hours appears to be the best condition in this investigation. And among all the oils used, soy oil and slack wax seem to be the best candidates for the heating media for improving biological resistance of treated wood. Concerning increasing the treatment efficiency, treatment at 220°C for 2 hours can be considered as an effective alternative, capable of improving the moisture and water excluding efficiencies and the anti-swell efficiencies by 40 -50% and reducing the weight loss during the decay test greatly.

**KEYWORDS** Wood, thermal treatments, hot oil treatment, slack wax, palm oil, soy oil, moisture properties, biological resistance, soil block test, mould resistance test

# 1. Introduction

Chemical wood preservatives such as creosote and CCA, are pesticides and raise some concerns about their environmental and health safety. Their use in some jurisdictions and for some exterior products is becoming more and more restricted. Other materials such as wood fibre/plastic composites and naturally durable wood species are often preferentially specified where there are concerns with wood preservative use. Another pesticide-free alternative capable of providing some protection to wood in exterior applications is wood modification to render wood more dimensionally stable and decay resistant. Thermal treatment of wood is known to improve wood properties by reducing hygroscopicity, improving dimensional stability and enhancing the resistance against biological attack, especially in an oxygen-free environment (Tiemann 1920, 1951; Stamm and Hansen 1937; Seborg et al. 1953; Stamm and Harris 1953; Stamm 1964; Burmester 1973; Giebeler 1983; Feist and Sell 1987; Wang and Cooper 2003). In recent years, thermal modifications of wood have been carried out successfully on an industrial scale in Europe, using steam, nitrogen or oil as the heat transfer and oxygen excluding medium. They are the oil-heat treatment in Germany (OHT-Process), Thermo Wood (or Premium wood) in Finland, Retification process (New Option Wood) and Bois Pserdure in France, Plato process in Netherlands (Kamdem 2002a; Sailor et al. 2000a, 2000b; Rapp and Sailer 2001; Militz 2002a, 2002b; Welzbacher and Rapp 2002; Syrjänen and Kangas 2000; Syrjänen 2001; Jämsä 2001; Dirol and Guyonnet 1993; Vernois 2001; Jermannaud et al. 2002; Tjeerdsma et al. 1998a, 1998b, 2000; Militz and Tjeerdsma 2001; Boonstra et al. 1998).

Concerning the hygroscopicity reduction and dimensional stability improvements of thermally treated wood, anti-shrink efficiencies of 40% to 50% can be obtained for Staybwood, by either heating at 320°C for 1 minute or at 150°C for 1 week (Stamm 1964). In Europe, wood heat treated in oil at 220°C for 4 hours had a fibre saturation point of 14%, compared with 29% for the untreated samples (Rapp and Sailer 2001). Thermo Wood and other thermal treatment processes can also reduce half of the original hygroscopicity and hydro-swelling (Syrjänen and Kangas 2000). Meanwhile, the treated wood may have higher (Vernois 2001) or lower water absorption (Welzbacher and Rapp 2004) depending on the treatment process, and higher diffusion coefficient of water along the tracheid axis (Hietala et al. 2002). In terms of the biological resistance of thermally treated wood, it was reported early that Staybwood had considerable decay resistance compared with the untreated wood (Stamm and Harris 1953; Stamm 1964). The heat-treated wood by the above industrial processes also has improved resistance against fungi (Militz 2002a, 2002b; Welzbacher and Rapp 2002). For the oil-heat treatment, at the highest temperature of 220°C, with the minimum absorption of oil, the wood gains the highest resistance against fungi, especially against brown rot fungi. In general, the biological resistance improvement of heat-treated wood depends on wood species and heat treatment conditions. It is believed to be closely associated with the EMC (equilibrium moisture content) reduction since certain MC (moisture content) of wood is necessary for the growth of fungi (Stamm and Harris 1953; Stamm 1964), but the chemical changes of the wood components during the treatment must be one of the main reasons. And for the oil-heat treated wood, the oil absorbed by wood during treatment may also have some effects on the biological resistance of wood.

This project deals with the hot oil treatment since it is believed that oil can be a good heating media as well as a carrier for other substances to further enhance the benefits of the treatment. Based on some research done in Europe (Rapp and Sailer 2001), more oils including vegetable oils, animal fats and mineral oils have been used to treat wood. And the research also pays more attention to the properties of treated wood. This paper mainly focuses on the moisture properties, including the adsorption at high humidity, and water absorption and hydro-swelling during water submersion. Meanwhile, soil block tests and mould resistance tests were carried out to study the biological resistance of hot oil treated wood. It aims to giving more information on the selection of oils and the determination of treatment temperature and time from the aspects of water related properties and biological resistance of treated wood.

#### 2. Materials and Methods

#### Moisture uptake, water absorption and dimensional stability

Samples with the dimension of  $25mm \times 25mm \times 10mm$  and with true radial/tangential orientation were cut from the spruce boards and treated in soybean oil, palm oil, hydrogenated

canola oil, tallow, lard, slack wax at 200 and 220°C for 2 and 4 hours. Then together with the control untreated samples, they were exposed to high humidity conditions (82 % RH, 26 °C) for over 4 weeks. The above samples were then submerged in water at 21°C and the water absorption, radial and tangential swelling of treated samples were determined periodically over the 28 day immersion period and compared to that of the controls. The moisture excluding efficiencies (MEE), water repellence efficiencies (WRE) and anti-swelling efficiencies (ASE) were estimated based on the moisture adsorption (MC<sub>t</sub>), water absorption (WA<sub>t</sub>) and swelling (S<sub>t</sub>) of treated samples relative to those of the controls (MC<sub>c</sub>), (WA<sub>c</sub>) and (S<sub>c</sub>): MEE (%) = 100 × (MC<sub>c</sub> - MC<sub>t</sub>) / MC<sub>c</sub>, WRE (%) = 100 × (WA<sub>c</sub> - WA<sub>t</sub>) / WA<sub>c</sub> and ASE (%) = 100 × (S<sub>c</sub> - S<sub>t</sub>) / S<sub>c</sub>, respectively. The replicates were 6 per treatment in this investigation.

# Soil block test of hot oil treated spruce

The wood species was spruce. All the samples, with the dimension of 25 mm  $\times$  25 mm  $\times$  9 mm, were from the same board. They were treated in the following hot oils: Slack wax, soy oil, palm oil, hydrogenated canola oil, lard and tallow at 200°C and 220°C for 2 hours and 4 hours, respectively. After conditioning in the lab for weeks, they were put into plastic bags, with certain distilled water in and conditioned for another 8 weeks' to increase the moisture content to 40-60%. Except for the treated spruce samples, spruce control samples, fir samples as the reference, spruce samples thermally treated in oven at 200°C for 2 hours and 4 hours, some wood plastic composite samples were also prepared for the soil block test. At the same time, two fungi: Gloeophyllum trabeum and Postia placenta, and test bottles with soil and wood feeders were prepared. The PH of soil was 5.8 and its water holding capacity was 38.8%. Certain water was added to each bottle to increase the moisture content of soil to about 130%. For the fungi resistance of thermally treated wood, the number of block replications per test variable was 6, except for the palm oil treated spruce. Because the mark of these palm treated samples was not clear any more after the water conditioning, the samples treated at 200°C and 220°C for 2 hours were used to test the resistance against Gloeophyllum trabeum, and other samples treated at 200°C and 220°C for 4 hours were used to test against Postia placenta, with the replicates of 16 or 18, respectively. After exposing the samples to the fungi for about 11 weeks, it was found that the weight loss of the reference fir samples exposed to Gloeophyllum trabeum was over 60%, so all the samples for this fungus were collected to measure the weight loss, as well as the moisture content after the exposure. But it was found that the Postia placenta used was not aggressive and the weight loss of all samples were relatively low, so the weight loss and the corresponding MC were not collected until after 20 weeks' exposure. (The main reference standard used in this investigation was ASTM D 2017-81: Standard Method of Accelerated Laboratory Test of Natural Decay Resistance of Woods.)

# Mould resistance test of hot oil treated wood

In order to investigate the mould resistance of hot oil treated wood, red pine and aspen samples were cut into the dimension of 7 mm by 20 mm in cross and 7 cm long. They were treated in soy oil, palm oil and slack wax at 220°C for 2 hours. Then they were conditioned in plastic bags with certain distilled water to increase the MC to above 40 %. Three species of mould were used, including: *Trichoderma pseudokoningii* (T.P.), *Gliocladium virens* (G.V.) and *Aspergillus niger* (A.N.). Every two samples were put into a sterilized petri dish with filter paper, wood sticks, water and certain mould culture. Replicates of 8 to 12 were used for each condition. Then all the

dishes were put in a high humidity conditioning chamber for 4 weeks. The mould growth of each dish was visually assessed, with the score of 5 being the maximum intensity and 0 being the lowest score. (The main reference standard used was D 4445-91 (Reapproved 1996): Standard test method for fungicides for controlling sapstain and mould on unseasoned lumber (laboratory method)).

### **3.** Results and Discussions

### Moisture uptake, water absorption and dimensional stability

Moisture uptake under high humidity conditions was reduced (increased moisture exclusion efficiencies) with increased oil temperature and time of treatment (Figure 1). Wood treated with palm oil, tallow and slack wax had the highest moisture excluding properties after 5 weeks and lard, soybean oil and hydrogenated canola oil had the highest moisture uptake although all provided moisture exclusion efficiencies in the 30 to 50 % range.



Figure 1 Moisture excluding efficiency (MEE) of spruce with different thermal treatments (82% RH and 26°C)

The water absorption after 28 days (Figure 2) was not greatly affected by higher treatment time and temperature except for the slack wax treatment. In fact some treatments (e.g. tallow) appeared to have worse water excluding efficiencies under more severe treating conditions. This is likely related to the lower oil retention noted below for these conditions and perhaps, to increased porosity and permeability in the wood with more severe treatment as was noted by others, and also to the increased leaching with the increase of soaking time (especially tallow). However, the slack wax stands out for its ability to retard water absorption and the effectiveness does increase with time and temperature of treatment, achieving 65% reduction in water absorption after 28 days soaking compared to control samples. Palm oil was the next most effective oil for inhibiting water uptake. These effects can also be seen in the plots of moisture content vs time (Figure 3-5). Both palm oil and slack wax had approximately linear water absorptions with time over the 28 day period while the other oils (soybean as an example in Figure 5) had rapid uptake initially followed by reduced rate of adsorption, somewhat parallel to the effect in control samples. For palm oil and slack wax, the water excluding efficiency is even

higher at short soaking times suggesting that their performance in intermittent wetting conditions would be even better compared to the other oils.



Figure 2 Water excluding efficiency (WEE) of different thermally treated spruce (28 day water soaking)



Figure 3 Water absorption rate in slack wax treated spruce compared to untreated reference samples



Figure 4 Water absorption rate in palm oil treated spruce compared to untreated reference samples



Figure 5 Water absorption rate in soybean oil treated spruce compared to untreated reference samples

Both radial (Figure 6) and tangential (Figure 7) anti-swell efficiencies increased with time and temperature of the thermal treatment. Performance was much better at 220°C than at 200°C. Radial ASE values were in the range of 50-55% while tangential values were 40-45% for the 4-hour treatment at 220°C. This difference between radial and tangential ASE's means that the differential tangential to radial shrinkage will be higher after thermal treatment not lower as had been found (Tjeerdsma et al. 1998b; Militz 2002a, 2002b) for Plato treated wood. There were slight differences in radial ASE among the different heat transfer compounds, with slack



wax, hydrogenated canola oil and soybean oil performing slightly better than the others. The slack wax treatment provided the highest tangential ASE values.

Figure 6 Effects of treatment on radial anti-swelling efficiency (ASE) of spruce



Figure 7 Effects of treatment on tangential anti-swelling efficiency (ASE) of spruce

From the swelling curves used to determine the ASE values (Figures 8 as an examples), it is evident that the swelling did not increase after 2-3 days and that the 8 day results should be indicative of actual long term ASE values.



Figure 8 Tangential swelling of slack wax treated spruce vs untreated reference



# Fungi resistance results of different oil treated wood

Fig. 9 Weight loss of spruce treated at 220°C for 2 and 4 hours in different oils during exposure to *Gloeophyllum trabeum* for 11 weeks

Fig. 9 shows the weight loss of thermally treated spruce in different oils during exposure to Gloeophyllum trabeum for 11 weeks. Both untreated spruce and fir, with the weight loss of over 60%, were non-resistant to Gloeophyllum trabeum according to this investigation. Compared with the vegetable oil and fats treated spruce, slack wax treated wood appears to be relatively resistant to this kind of brown fungi, and soy oil shows the best performance among all the vegetable oils and fats to improve the fungi resistance of spruce, especially under the thermal treatment at 220 °C for 4 hours. From the previous high humidity exposure and water soaking experiment of the thermally treated wood, it was found that slack wax and palm oil were the better candidates for the heating media, while this soil block test came up with that soy oil and slack wax could be the best choices as the heating media for such wood modification in terms of the decay resistance improvement. According to ASTM D 2017, the decay resistance of the soy oil and wax treated spruce at 220°C for 4 hours can be classified as "Resistant" to Gloeophyllum trabeum and other treated spruce at 220°C fall into the group of "Moderately resistant". Since after the thermal treatment, wood gains reduced hygroscopicity and water absorption, which could impart the improved biological resistance to wood. Fig. 10 shows that the MC of the treated wood before fungi exposure was around 40-60%, which should be good for the growth of fungi. After the fungi exposure for 11 weeks, most of samples gained more moisture, while samples treated in soy oil at 220°C for 4 hours and samples treated in slack wax at 220°C for 2 and 4 hours seemed to have reduced MC, which can be one of the reasons for the lower weight loss and is to be explained in the future.



Fig. 10 Moisture content of spruce treated at 220°C for 2 and 4 hours before and after exposure to *Gloeophyllum trabeum* for 11 weeks

In terms of P. P. attack, the fungus used was not aggressive, with the much lower weight loss after exposure for 20 weeks (Fig.11). The weight losses of spruce samples and fir samples were from 10-17 % and 11-27 %, respectively. The weight losses of soy oil treated samples were between 5 % and 10 %, while those of slack wax treated wood were even below 5 %. And the MC of these treated wood dropped a lot after the decay test (Fig. 12).



Fig. 11 Weight loss of spruce treated at 220°C during exposure to Postia placenta for 20 weeks



Fig. 12 Moisture content of spruce treated at 220°C before and after exposure to Postia placenta

Fig. 13 shows the results of mould test. Control samples were highly susceptible to mould attack, especially by mould fungi of AN and TP. Compared with aspen, red pine is more

susceptible to mould attack. The short-term mould test indicates that the hot oil treated wood has significantly higher resistance than the control samples but the soy oil treated wood has somewhat lower resistance against TP. The long-term mould resistance of these samples will be reflected by the weathering test which has been carried out since May 2004 on the roof of the Earth Science Building of University of Toronto and the results will be reported in the near future.



Fig. 13 Mould growth scale on treated and control red pine and aspen samples

### 4. Conclusions

1. Slack wax and palm oil are better than soy oil or other oils used in improving the moisture performance of thermally treated wood, and 220°C is superior to 200°C, with 4 h being generally better than 2 h treatment;

2. Hot oil treated wood has higher decay and mould resistance than control samples. The treatment at 220°C for 4 hours appears to be the best treatment condition in this investigation. Soy oil and slack wax are good heating media with respect to improving the fungi resistance of hot oil treated wood.

3. Generally speaking, treatment at 220°C for 2 hours can be considered as an effective alternative, capable of improving the moisture and water excluding efficiencies and the antiswell efficiencies by 40 - 50% and reducing the weight loss during the decay test greatly.

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