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Effect of oil heating age on colour and dimensional stability of heat treated *Pinus radiata*

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Abstract *Pinus radiata* specimens with moisture content of 8–11% and dimensions of $90 \times 35 \times 200\ \text{mm}^3$ were heat-treated in an oil bath of commercial grade raw linseed oil. The effect of oil aging was investigated under treatment condition of 180°C and 3 hours by using oil that had been preheated for 0, 3, 9, 15, 21, and 27 hours, respectively, before the wood treatment. After treatment using oils of varying ages, wood colour change was examined using a Minolta spectrophotometer through CIE 1976 (L^*a^*b) system. Water repellent efficiency and anti swelling efficiency in high humidity conditions were measured to determine the stability of the treated wood.

The results show that stability of the treated wood was improved significantly compared to the matched untreated wood but the treated wood tended to be darker. Oil viscosity increased with the heating age resulting in slight decrease in weight percentage gain. Water repellent efficiency was decreased with an increase in heating age of oil. However no significant difference in total colour variation and wood stability (anti swelling efficiency) was observed between specimens treated with oils of varying heating age.

Abbreviations

ASE	Anti-swelling Efficiency
CIE	Commission internationale de l'éclairage
WPC	Weight Percentage Change
WRE	Water Repellent Efficiency

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Einfluss der Gebrauchsdauer des Ölbades auf die Färbung und Dimensionsstabilität von wärmebehandelter Radiatakiefer

Zusammenfassung Pinus radiata Prüfkörper mit einer Holzfeuchte von 8–11 % und den Abmessungen $90 \times 35 \times 200\ \text{mm}^3$ wurden in einem Ölbad aus Rohleinöl wärmebehandelt. Der Einfluss der Gebrauchsdauer des Ölbades wurde bei einer Wärmebehandlung von 180°C und 3 Stunden untersucht. Das dabei verwendete Öl war vor dieser Behandlung für eine Dauer von 0, 3, 9, 15, 21 und 27 Stunden vorerhitzt worden. Nach der Wärmebehandlung im unterschiedlich lang vorerhitzten Ölbad wurde die Farbveränderung im Holz mit einem Minolta Spektralfotometer anhand des CIE 1976 (L^*a^*b) Systems untersucht. Zur Bestimmung der Stabilität des behandelten Holzes wurden das Wasserabstoßungsvermögen und das Quellresistenzvermögen bei hoher Umgebungsfeuchte ermittelt.

Die Ergebnisse zeigen, dass die Stabilität des behandelten Holzes im Vergleich zu unbehandeltem Holz signifikant besser war, sich aber tendenziell dunkel verfärbte. Die Ölviskosität nahm mit zunehmender Dauer der Vorerhitzung zu. Dies führte zu einer leicht sinkenden Massenzunahme des Holzes. Das Wasserabstoßungsvermögen nahm mit zunehmender Dauer der Vorerhitzung des Öls ab. Hinsichtlich der Farbveränderung und des Quellresistenzvermögens konnte jedoch kein signifikanter Unterschied zwischen den Prüfkörpern, die mit unterschiedlich lang vorerhitzten Ölen behandelt worden waren, festgestellt werden.

1 Introduction

Due to environmental and social concerns regarding the use of tropical hardwoods and other natural woodland species, plantation forest has become the major wood resource. However, the fast grown plantation timbers have inferior qualities, particularly low durability and dimensional stability. In order to improve the durability, wood has traditionally been treated using chemicals such as CCA and creosote. However, negative impacts of such chemical treatments on human health and the environment have triggered bans in a number of developed countries from using the chemically treated wood. Therefore, physical treatments or alternative chemical treatments have been investigated.

Thermal modification of wood is a commercially viable technology. It uses heat as a medium to alter the structure and chemistry of wood to achieve desired properties. Heat treated wood is darker in colour and has better dimensional stability (Stamm 1964; Syrjänen and Kangas 2000; Bekhta and Niemz 2003). Thermal modification of wood is achieved by heating the wood usually above 160°C in an oxygen free environment, for example, using a liquid medium such as oil (Hill 2006). Stamm et al. (1946) explored this technology, by heating wood in a molten metal bath over a range of temperature and time, creating a product called Staybwood. The degree of reduction in hygroscopicity and anti swelling efficiency (ASE) almost doubled with each 10°C increase in the operating temperature. Recently, the Menz Holz process has been developed in Germany in which wood is treated at 180–220°C in refined linseed oil in a closed process vessel (Sailer et al. 2000a, 2000b).

Linseed oil is one of the main industrial or drying vegetable oils which is obtained from an annual herb flax (*Linum usitatissimum*). Global production of linseed oil in 2006 was 665,000 tonnes and it can be produced in both tropical as well as temperate regions (FAO 2008). This oil is an excellent drying agent and is used in industrial processes in paints, resins, inks, soaps, varnishes, wood treatments and linoleum (Ulvcrona et al. 2005). The high linolenic acid (C18:3) content of the linseed makes the oil drying fast thus it has good auto oxidative properties and can harden under sunlight without any modification or addition of siccatives which are chemical compounds used to accelerate drying process and improve performance of coatings (Treu et al. 2001).

The advantages of linseed oil, such as improved wood protection, ease in drying after treatment, wide availability and high boiling point (higher than 334°C), make this oil a preferred candidate for use in the heat treatment of wood. However, any change in consistency and colour of the oil with recycling may affect some wood properties after treatment, about which there is little information available.

The objective of this work was to investigate the effect of the oil aging and to determine the maximum period for which the oil can be reused. Also, the effect of oil heating age on the dimensional stability and colour of the treated wood is experimentally studied. The results are compared with untreated specimens and specimens treated in fresh oil. The effect of oil viscosity on these properties is also examined.

2 Material and method

2.1 Specimen preparation

The specimens were prepared from 2.5–3 m long boards of *Pinus radiata* obtained from a local sawmill. All boards were of the same commercial grade, dry flatsawn sapwood (10–12% moisture content). Clear specimens with dimensions of 200 mm (long) × 90 mm (wide) × 35 mm (thick) were cut from these boards. Control specimens (no treatment) and treatment specimens were prepared from each plank with 5 mm sections being trimmed from each board end, as shown in Fig. 1. These specimens were then oven-dried for 48 hours, cooled down over silica gel in a desiccator and weighed before being placed in a conditioning chamber under 65% RH and 20°C for 2–3 weeks to attain equilibrium. Eight specimens were treated in each run. In order to minimise the wood variation, the end-matched treatment specimens from one plank were used in different runs with one specimen in each run.

2.2 Treatment

Conditioned specimens with moisture content of 10–12% were heated in an oil bath using commercial grade raw linseed oil (M/S Mainland Paint and Printing Ink Ltd.) as the heating medium. For the first run fresh raw linseed oil was used. The oil was heated to 180°C before immersing the specimens and treating them for 3 hours. The oil was recirculated continuously and no external pressure was applied.

After treatment, the heating was turned-off, and lid of the oil bath was removed. Following this, specimens were kept in the oil bath for 15 minutes then the oil was drained. Next the treated specimens were oven-dried for 48 hours, cooled down over silica gel in a desiccator and weighed. Finally, the specimens were placed in a conditioning chamber under condition of 65% RH and 20°C for 2–3 weeks until equilibrium was attained.

The above experiment was repeated by using “used” linseed oil that had been heated to a temperature of 180°C for 3, 9, 15, 21 and 27 hours, respectively.

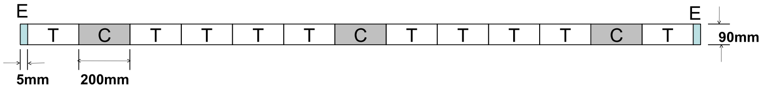


Fig. 1 Cutting pattern of the specimens where: E represents the trimmed ends; T represents the treatment specimen and C the control (untreated)
Abb. 1 Entnahmeplan der Prüfkörper: E abgeschnittene Enden des Schnittholzes, T Prüfkörper für die Wärmebehandlung, C unbehandelte Kontrollprobe

2.3 Property measurement and determination

2.3.1 *Viscosity measurement*

Viscosity of fresh linseed oil and pre-heated linseed oil were measured using a Haake viscometer with MV1 sensor with a shear rate of 1000 per second, measured at 20.2 ± 0.1°C. The mean of 5 replicates was used for each condition. In addition, oil was heated at 180°C for 3 hours with and without wood specimens to compare effect of heating in presence of wood on oil viscosity. The mean of 10 replicates was used for this examination.

2.3.2 *Weight percentage change*

The weight percentage change (WPC) was determined using (1) which gives averaged relative difference in oven-dry weight of the tested specimens through the oil heat treatment:

$$WPC = \left[\frac{(W_a - W_b)}{W_b}\right] \times 100\% \tag{1}$$

where W_b is the oven-dry weight of specimens before the treatment, and W_a is the oven-dry weight of specimens after the treatment.

2.3.3 *Colour*

Colour change was examined by visual inspection and quantified using a Minolta spectrophotometer cm2500d through CIE (Commission internationale de l'éclairage) 1976 (L^*a^*b) system which is a well established procedure for colour measurement (St-Onge et al. 2005; Esteves et al. 2008; Ahajji et al. 2009). The total colour variation was calculated from brightness (L^*) and two colour coordinates of a^* and b^*, using the following equation:

$$\Delta E = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \tag{2}$$

in which L^* is the brightness or luminance ranging from black (0) to white (100), a^* is the colour coordinate from red (positive) to green (negative) and b^* is the colour coordinate from yellow (positive) to blue (negative) (BS3900 1986a, 1986b; ADOBE 2008).

The diameter of the colour measuring circle on the specimen surface was 10 mm with the corresponding area of 0.78 cm^2. The light source was the Standard Illuminant D 65 which represents the average day light including UV region and 10° standard observer. For the colour measurements the specimens were first stabilised at 20°C and 65% RH before taking the measurements.

2.3.4 *Dimensional stability and water repellency*

Dimensional stability and water repellency were measured at 20°C by immersing the test specimens in a water bath in which the water was re-circulated continuously. During the tests, the specimens were weighed every 24 hours. Once the weight changes of the test specimens were less than 0.1%, it was assumed that the specimens had reached equilibrium and their dimensions in longitudinal, width (tangential) and thickness (radial) directions were measured. The weight was measured with an accuracy of ±0.001 g and the accuracy for the dimension measurement was ±0.01 mm.

Water repellent efficiency (WRE) was determined using (3):

$$WRE = \left(\frac{W_c - W_t}{W_c}\right) \times 100\% \tag{3}$$

where W_c is the water uptake of untreated specimens and W_t is the water uptake of treated specimens. Therefore, a positive and high value of *WRE* indicates more improvement in terms of reduction in the water uptake through the heat treatment.

In a similar way, the anti-swelling efficiency (ASE) was determined from the following equation:

$$ASE = \left(\frac{S_c - S_t}{S_c}\right) \times 100\% \tag{4}$$

in which S_c is the volumetric swelling of untreated specimens and S_t is the volumetric swelling of the treated specimens. Once again, the positive and high value for *ASE* means that the treatment reduces the volumetric swelling more significantly.

3 Results and discussion

3.1 Viscosity of oil

Linseed oil when heated for a prolonged time at higher temperature converts to "stand oil", which is a thickened heat polymerised oil (Chemwatch 2007; Tollenaar and Bolthof

Table 1 Viscosity of linseed oil after different heating times ($n = 5$). SD is standard deviation and $Y+$ and $Y-$ are respectively, upper and lower range from the mean

Tab. 1 Viskosität von unterschiedlich lang erhitztem Leinöl ($n = 5$). SD Standardabweichung; $Y+$ und $Y-$ oberer bzw. unterer Streubereich des Mittelwerts

Heating time (hours)	No.1	No.2	No.3	No.4	No.5	Mean	SD	$Y+$	$Y-$
0	48.68	48.9	48.78	48.79	48.75	*48.78*	*0.08*	0.12	0.10
6	50.86	51.41	51.58	51.28	51.45	*51.32*	*0.28*	0.26	0.46
9	51.97	52.4	52.77	52.38	52.45	*52.39*	*0.28*	0.38	0.42
15	55.91	55.99	56.04	55.98	55.96	*55.98*	*0.05*	0.06	0.07
21	58.09	57.98	58.11	58.06	58	*58.05*	*0.06*	0.06	0.07
27	58.88	59.05	60.01	59.88	59.45	*59.45*	*0.50*	0.56	0.57

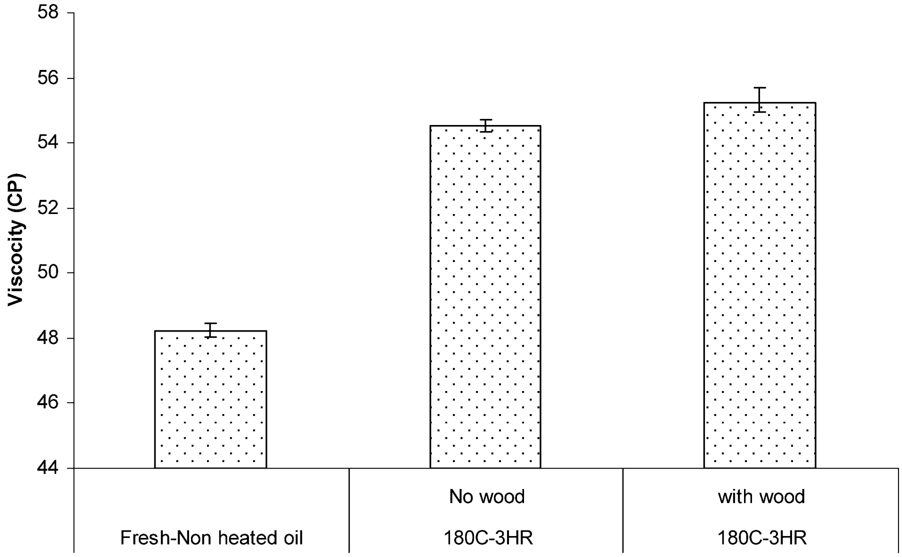


Fig. 2 Mean viscosity of fresh oil and oil heated with and without wood ($n = 10$)

Abb. 2 Mittlere Viskosität von frischem Öl und von Öl, das mit und ohne Holz erhitzt wurde ($n = 10$)

1946). With increased heating time the colour of oil was changed from pale yellow to dark brown. The mean viscosity of the oil increased with the heating duration, changing from 48.78 cp for the control to 59.45 cp after 27 hour heating (Table 1). The viscosity of the linseed oil increased on heat treatment due to evaporation of volatile components and heat polymerisation of the oil (Mabery 1923; Powers 1950). Figure 2 shows the difference in the oil viscosity between heating with wood and heating without wood, and it is found that the viscosity of the oil heated with wood was slightly higher than that without wood. This indicates that release of wood extractives and wood degradation products also contributes, to some extent, to the increase in viscosity of the linseed oil during the wood heat treatment (Sailer et al. 2000a).

3.2 Weight percentage change

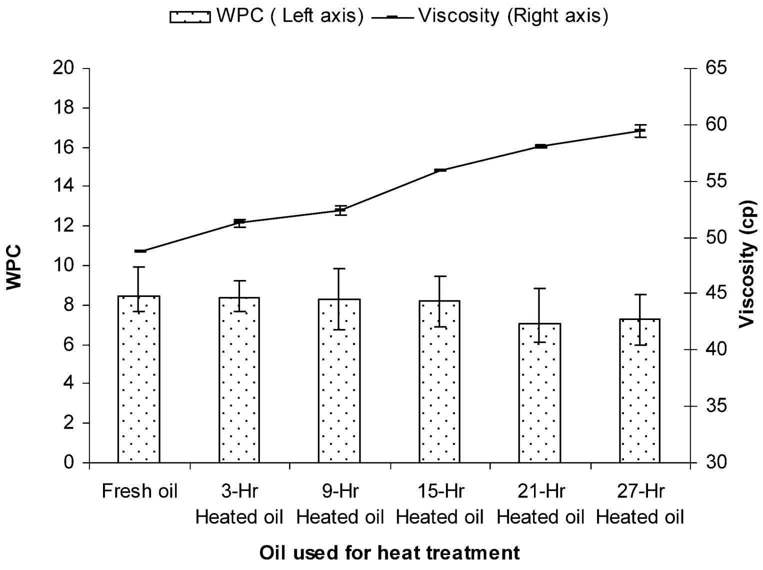
Table 2 shows the percentage change in weight after the treatments using linseed oil that had been preheated for different periods. The results show that the mean weight of the treated specimens increased by 7 to 8.4%, with the greatest increase occurring with the fresh oil. The weight change of the heated wood is believed to be attributed to a number of counteracting factors: (1) loss of wood mass due to changes in chemical composition of the wood at high

Table 2 Mean change in oven dry weight of wood after treatment $n = 8$. SD is standard deviation and $Y+$ and $Y-$ are respectively, upper and lower range from the mean

Tab. 2 Mittlere Änderung der Trockenmasse von Holz nach der Behandlung ($n = 8$). SD Standardabweichung; $Y+$ und $Y-$ oberer bzw. unterer Streubereich des Mittelwerts

Treatment medium	WPC%	SD	$Y+$	$Y-$
Fresh oil	8.44	0.74	1.52	0.75
3-Hours Heated oil	8.37	0.44	0.82	0.67
9-Hours Heated oil	8.31	0.86	1.57	1.55
15-Hours Heated oil	8.24	0.78	1.21	1.37
21-Hours Heated oil	7.07	0.89	1.79	0.95
27-Hours Heated oil	7.30	0.79	1.26	1.35

Fig. 3 Effect of heating age of oil used for wood treatment on the specimen weight percentage change and oil viscosity
Abb. 3 Einfluss der Vorerhitzungsdauer des für die Holzbehandlung verwendeten Öls auf die Massenänderung der Prüfkörper und die Ölviskosität



temperatures (Stamm 1956; Syrjänen and Kangas 2000; Esteves et al. 2008), (2) removal of wood extractives (Esteves and Pereira 2008); and (3) oil uptake by the specimens. The first two factors contribute towards a loss in the specimen weight while the third one results in a weight gain. The net result was a small increase in weight after the treatment.

Also, the weight gain of the treated specimen was affected by the cumulative oil heating time as shown in Fig. 3. The wood treated in fresh oil gained 16.23% more weight than those treated in preheated oil. The lower weight gains when treated in oil for 21 and 27 hours may be due to poorer penetration of oil resulting from the increase in oil viscosity when the oil was preheated.

3.3 Colour

Wood colour changed significantly after thermal treating with the specimens turning brownish yellow. This was consistent with previous studies (Syrjänen and Kangas 2000; Sundqvist 2002; Hapla and Militz 2004; Esteves et al. 2008). The total colour variation between the untreated control specimens and the heat treated specimens was more than 15 units (Table 3). Among the colour coordinates, the lightness of the treated wood decreased remarkably whereas the reddish and the yellowness increased to some extent (Table 3). The greater value in the changes of Δb^* compared to Δa^* indicates more yellowing of the treated wood than the changes in reddish. A similar trend was noted by Tjeerdsma et al. (2005).

However, the colour change was broadly similar for the oils being heated for different durations and was difficult to differentiate by the naked eye. This can further be supported by the data presented in Table 3 which shows that the difference in the total colour variation between wood specimens treated using preheated oils and wood specimens using fresh oil was less than 1.2 units. Generally, the human eyes are unable to differentiate colour change if less than 2–3 units (Sundqvist and Morén 2002). This suggests no effect of oil heating age on wood colour for heating period up to 27 hours on visual inspection to naked eye.

The change in wood colour during heat treatment may be due to a combination of the following factors:

- Formation of oxidative products like quinonnes (Bekhta and Niemz 2003; Tjeerdsma et al. 1998) and other coloured products from degradation of hemicelluloses and lignin (Sehlstedt-Persson 2003; Kamke 2006; Esteves et al. 2008) which migrate towards the surface during the heat treatment;
- Removal or migration of extractives (Sundqvist and Morén 2002) and nutritive compounds such as low molecular sugars and amino acids (Theander et al. 1993) towards the surface.

In addition, the oil uptake may affect the wood colour change to some extent (Sailer et al. 2000a). Figure 4 shows the relationships among the total colour variation and the weight percentage change (WPC) of the treated specimens using both fresh oil and preheated oils for the treatment. A trend was observed that the specimens with higher WPC also had greater colour variation. In addition, the lightness coordinate L^* was found to be higher in specimens treated using 21 h and 27 h heated oils than those treated in fresh oil (Table 3). This indicates that the treated wood with greater oil uptake tended to be darker.

3.4 Water repellency and dimensional stability

Wood became more water repellent after the heat treatment. The heat treated wood achieved water repellent efficiencies of between 28 to 46% indicating less water uptake

Table 3 Change in colour coordinates (CIE 1976-$L^*a^*b^*$system) after treatment
Tab. 3 Änderung der Farbwerte (CIE 1976 $L^*a^*b^*$) nach der Wärmebehandlung

	Colour coordinates			Change in colour coordinates of treated specimens compared to untreated specimens				Change in colour coordinates of specimens treated in heated oil compared to specimens treated in fresh oil			
Specimens	L^*	a^*	b^*	ΔL^*	Δa^*	Δb^*	ΔE^*	ΔL^*	Δa^*	Δb^*	ΔE^*
Control specimens	75.51	6.62	23.65	0	0	0	0				
Treated in fresh Oil	61.4	10.75	31.83	−14.11	4.13	8.18	16.82	0	0	0	0
Treated in 3 h Heated Oil	60.76	11.3	31.32	−14.76	4.68	7.67	17.28	0.55	0.55	−0.51	0.93
Treated in 9 h Heated Oil	61.32	11.35	31.06	−14.19	4.74	7.41	16.69	0.61	0.61	−0.77	1.15
Treated in 15 h Heated Oil	61.55	11.38	31.19	−13.96	4.76	7.54	16.57	0.63	0.63	−0.63	1.1
Treated in 21 h Heated Oil	62.62	10.81	31.62	−12.9	4.2	7.97	15.78	0.25	0.25	−0.21	0.41
Treated in 27 h Heated Oil	62.42	10.96	31.26	−13.1	4.34	7.61	15.76	0.21	0.21	−0.57	0.64

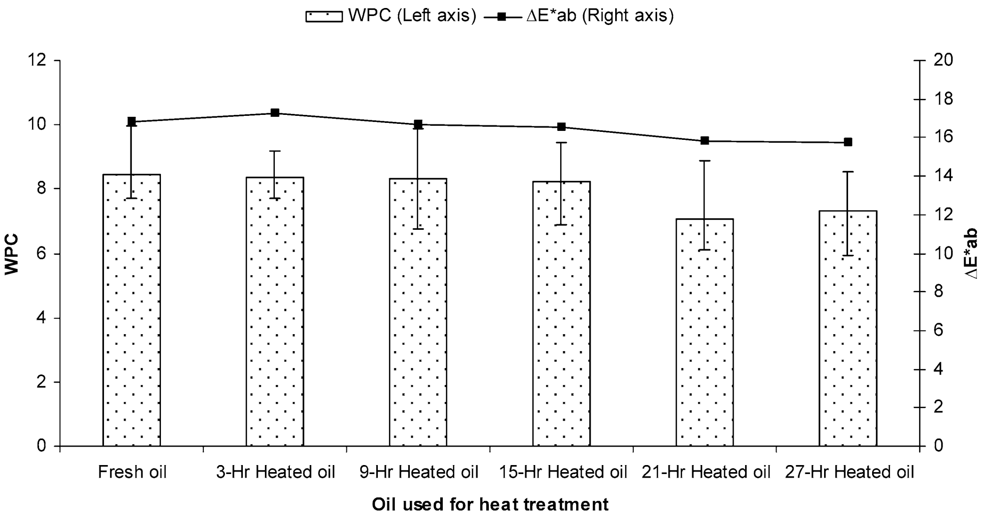


Fig. 4 Relationship between weight percentage change (WPC) and total colour variation and heating age of oil used for treatment
Abb. 4 Zusammenhang zwischen Massenänderung (WPC), gesamter Farbänderung und Dauer der Vorerhitzung des für die Behandlung verwendeten Öls

than untreated wood (Fig. 6). Similarly, volumetric swelling of the treated wood was decreased by 29–31%. This trend is consistent with previous findings (Stamm et al. 1946; Buro 1954; Sailer et al. 2000a; Wang and Cooper 2005). There was almost no difference in water absorption for wood treated using fresh oil and preheated oil for less than 21 h. However, wood treated using oil preheated for 21 h and 27 h showed significant increase in the water absorption (Fig. 5) and significant drop (around 35%) in WRE (Fig. 6). This is probably due to less oil uptake by these specimens compared to those treated in fresher oil. However, no significant difference was found in volumetric swelling percentage among different treatments. This indicates that oil uptake has more influence on water absorption than on volumetric swelling. This implies that chemical changes due to the heat treatment can improve the wood stability to a greater extent than the reduction in water absorption (Wang and Cooper 2005; Temiz et al. 2006). According to Rowell and Banks (1985) hemicelluloses, non-crystalline cellulose, surfaces of crystalline cellulose and to a small degree lignin are responsi-

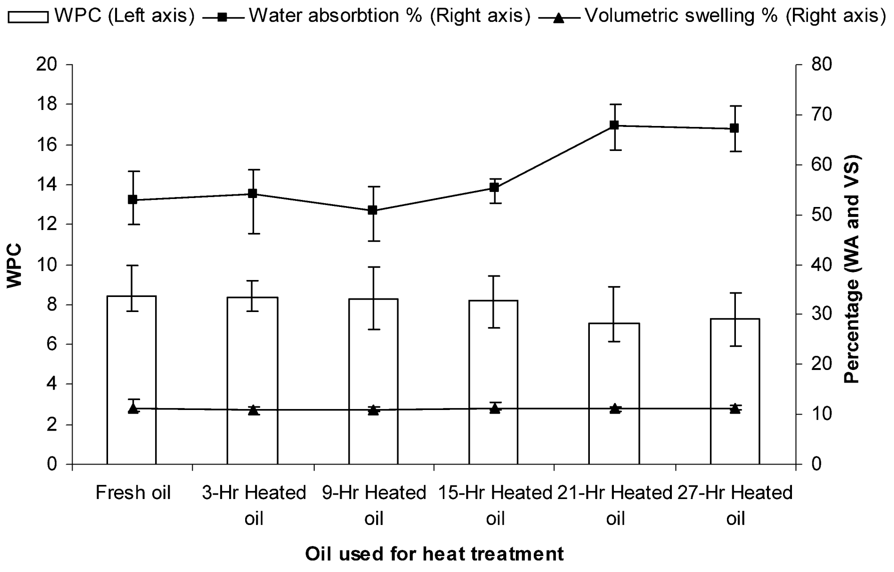


Fig. 5 WPC, water absorption percentage and volumetric swelling percentage of specimens heat treated using oil of different heating age
Abb. 5 WPC, prozentuale Wasseraufnahme und prozentuale Volumenquellung der Prüfkörper in Abhängigkeit der Dauer der Vorerhitzung des verwendeten Öls

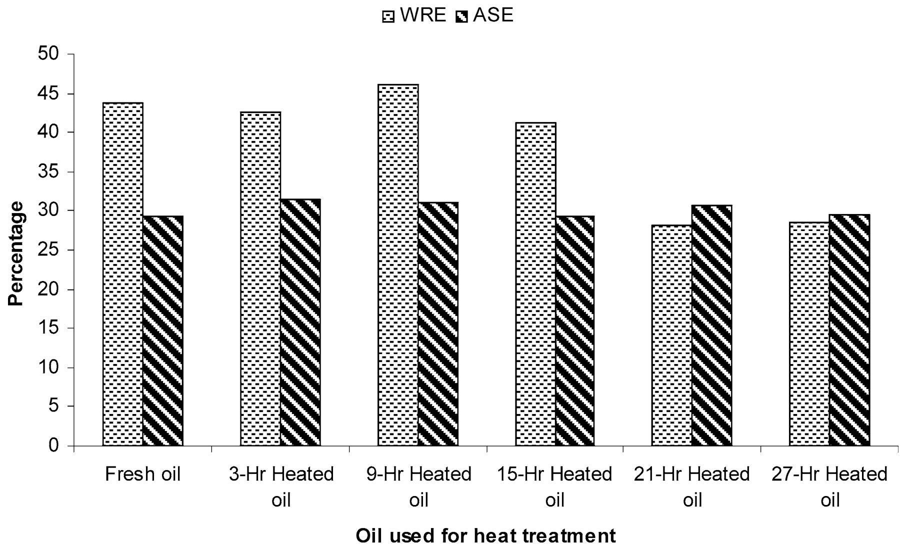


Fig. 6 Water repellent efficiency and Anti-swelling efficiency of heat treated wood using oil of different heating age compared to untreated control
Abb. 6 Wasserabstoßungsvermögen und Quellresistenzvermögen von Holz in Abhängigkeit der Dauer der Vorerhitzung des verwendeten Öls im Vergleich zum unbehandelten Holz

ble for hygroscopicity of wood. Of these, the hemicelluloses are the most hygroscopic. In conclusion, improved dimensional stability and water repellency in treated wood should be mainly due to decomposition of hemicelluloses at high temperatures (Seborg et al. 1953; Hillis 1984; Evans 2003).

4 Conclusion

After heat treatment of radiata pine wood in raw linseed oil at 180°C for 3 hours, the wood darkened and became less hygroscopic and dimensionally stable. The effects of oil aging (up to 27 hours) on properties of heat treated wood were investigated. The results showed that oil viscosity increased by 22% with the heating period. The oil uptake in treated wood decreased by 16.23% with heating age of oil which is believed to be due to an increase in oil viscosity. Water repellent efficiency is also affected (around 35%) by heating age of oil used for heat treatment. However, no significant effect of oil aging was observed on total colour variation and anti swelling efficiency of the treated wood. This implies that improvement in dimensional stability of the heat treated wood in oil is mainly due to chemical changes in wood due to high temperature. Therefore, heating age of oil (up to 27 hours) used for heat treatment does not affect colour change and dimensional stability of treated wood. Further work is recommended to investigate the impact of longer heating age of oil on the wood properties in the heat treatment.

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