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Evaluation of Thermal Treatability of Caucasian Fir (*Abies nordmanniana* (Link.) Spach.) Treated with Heated Tanalith-C of CCA above and below the Fibre Saturation Point

İlker USTA
Hacettepe University, Wood Products Industrial Engineering, 06532 Beytepe, Ankara - TURKEY

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Abstract: Caucasian fir (*Abies nordmanniana* (Link.) Spach.) was treated using the full-cell process with heated tanalith-C at elevated temperatures from 5 to 70 °C above and below the fibre saturation point (FSP: 32.1%) (in 40% and 20% moisture content (MC) levels). Thermal treatability was determined on the basis of preservative uptake (the percentage of void volume filled, VVF%) in transverse flow (a combination of tangential and radial directions) and triplex flow (based on all 3 flow directions). To characterise the treatability, analysis of the coefficient of transverse thermal conductivity was also performed above and below the FSP. Thermal conductivity (Tc) increased markedly with increasing temperature at either MC level. Tc was found to be relatively high at 40% MC due to the contribution of free water in the lumens. However, VVF% did not follow the evolution of the temperature in both flow directions at either MC level. The VVF% variation seemed to depend on the FSP, e.g., it showed almost a parabolic trend in 20% MC, and reached the highest values in either direction at around 30 °C.

Key Words: Caucasian fir, Thermal treatment, FSP, Elevated temperatures, Thermal conductivity

Isıtılmış Tanalith-C (CCA) ile Lif Doygunluğu Noktası Üzerinde ve Altında Emprenye Edilen Doğu Karadeniz Göknarının Isı Destekli Korunabilirlik Özelliğinin Belirlenmesi

Özet: Doğu Karadeniz göknarı (*Abies nordmanniana* (Link.) Spach.), 5 °C`den 70 °C`ye kadar değişen sıcaklıklarda ısıtılmış tanalith-C kullanılarak -dolu hücre yöntemine göre- lif doygunluğu noktası (LDN: %32.1) üzerinde ve altında (%40 ve %20 rutubet miktarında) emprenye edildi. Isı destekli korunabilirlik özelliği, koruyucu sıvı içerilme miktarının (KSİ%, ağaç malzeme hücre boşluklarının koruyucu sıvıyla içerilmesi yüzdesi) belirlenmesiyle -teğet ve radyal ayrımı olmaksızın- liflere dik yönde ve tüm akış yönlerinin açık olduğu tripleks yönde niceliksel olarak tanımlandı. Bu özelliğin nitelenmesi açısından, liflere dik yöndeki ısı iletme katsayısı da ayrıca belirlendi. Isı iletme özelliği (IİÖ) sonuçları, -LDN üzerinde ve altında- sıcaklığın artmasına bağlı olarak IİÖ`nin göreceli bir şekilde arttığını gösterdi. Özellikle, hücre boşluğundaki serbest su miktarının fazla olduğu %40 rutubet miktarındaki ısı iletme değerleri, %20 rutubet miktarına göre, daha yüksek bulundu. Emprenye sonuçları ise, LDN üzerinde ve altında -hem liflere dik hem de tripleks yönde- sürekli olarak artan ısı değişimine bağlı olarak KSİ% değerlerinin doğrusal bir şekilde artmadığını gösterdi. Bununla birlikte, KSİ% değişiminin, LDN etkisinde kaldığı görüldü. Örneğin, %20 rutubet miktarındaki KSİ% değişimi, her iki akış yönünde de parabolik bir görünümde -daha düzenli bir nicelikte- gerçekleşirken, en yüksek değerlerine 30 °C seviyesinde ulaştı.

Anahtar Sözcükler: Doğu Karadeniz göknarı, Termik koruma, LDN, Sıcaklık artırımı, Isı iletme

Introduction

In recent years, much attention has been paid to the protection of the environment. Many researchers in the field of wood preservation have studied novel preservation techniques; these have no or less impact on the environment. Thermal treatment technology is one of these.

It has long been known that the different intrinsic wood properties (such as durability, sorption behaviour, shrinkage and swelling behaviour, and strength properties) are changed by thermal treatment at elevated temperatures (Stamm, 1964; Burmester, 1973; Giebeler, 1983). The level of change very much depends on wood species and the process conditions, in which the

* Correspondence to: iusta@hacettepe.edu.tr

temperature and the absence of oxygen play a predominant role.

However, only recently in Europe have attempts been made to develop industrially applicable technology to thermally modify wood. Developments took place in the Netherlands (Ruyter, 1989; Boonstra et al., 1998; Tjeerdsma et al., 1998; Militz and Tjeerdsma, 2000), France (Dirol et al., 1993; Vernois, 2000), Germany (Rapp and Sailer, 2000; Rapp et al., 2000); and Finland (Viitanen et al., 1994; Jämsä and Viitaniemi, 1998; Syrjänen et al., 2000). The total production capacity of heat-treated wood in 2001 was estimated at approximately 165,000 m^3, and was planned by the industry to increase to some 265,000 m^3 in 2003 (Militz, 2002).

Langrish and Walker (1993) stated that knowledge of the thermal properties (such as the specific heat, the thermal conductivity, and the thermal diffusivity) is not only of importance when considering the insulating and fire resistant properties of wood. It is also needed to determine the steaming time for peeler logs and the warm-up time for timber prior to kiln drying. Furthermore, it is a necessity in preservative treatment, especially for thermal treatment processes.

Thermal conductivity is expressed by the coefficient of thermal conductivity (k). This is a measure of the quantity of heat in calories that will flow during a unit of time (s) through a body 1-cm thick with a surface area of 1 cm^2, when a difference of 1 ºC is maintained between the 2 surfaces, i.e. k is measured in (kcal m / h ºC)* (Kollmann and Cote, 1968).

In general, the thermal conductivity of wood is low because its structure is porous. Dry wood is one of the poorest conductors of heat due in part to the low conductivity of the actual cell wall materials, and in part to the cellular nature of wood, which in its dry state contains within the cell cavities a large volume of air –one of the poorest conductors known (Desch and Dinwoodie, 1996). When examining the heat flow in slender pieces of timber or round wood, the longitudinal heat flow can be neglected even though the thermal conductivity in the axial (longitudinal) direction is about twice the conductivity in the transverse (tangential, radial) direction (Siau, 1984). Important differences do not exist between the radial and tangential directions (Ratcliffe, 1964a, 1964b, 1964c). Knigge and Schulz (1966) determined the coefficients of thermal conductivity of different woods at 20 ºC for 3 flow directions (in kcal m / h ºC) as follows: 0.191–0.284 (axial), 0.104–0.151 (radial), and 0.090–0.140 (tangential).

Thermal conductivity (Tc) is influenced by various factors, such as wood structure, density, moisture, temperature, extractives, and defects (checks, knots, cross grain) (Steinhagen, 1977). Sova et al. (1970) stated that Tc increases proportionally with wood density, moisture content (MC), and temperature. According to Kollmann (1951), when moisture is increased or reduced below the fibre saturation point (FSP) by 1%, Tc is increased or reduced from 0.7% to 1.18%. However, above the FSP, the increase or reduction is somewhat higher, and in general wood with an MC higher than 40% has an approximately 1/3 higher conductivity than dry wood. Furthermore, Tc is affected by temperature. Kanter (1957) determined the k (kcal m / h ºC) for birch at temperatures between 20 and 80 ºC at different MCs, and found variations from 0.17 to 0.21 at 20% MC and from 0.22 to 0.27 at 40% MC.

Little is known about the relationships between thermal conductivity and transverse permeability, but the thermal treatability of Caucasian fir –a refractory softwood species– deserves further investigation.

It may be natural to assume that the preservatives that produce a sediment (i.e. water bornes) when heated would not be suitable for preservative treatment, because when wood is placed in a hot preservative, moisture evaporates and the air contained in the cell cavities expands (Hunt and Garratt, 1967). On the other hand, heating the water borne solutions may be more useful to improve fluid absorption for the treatment of refractory species when the full-cell preservative treatment is operated by heated preservative chemicals (e.g., tanalith-C of CCA). The objective of the present study, therefore, was to clarify the role of the solution's temperature on the permeability of Caucasian fir in both transverse and triplex directions at elevated temperatures ranging from 5 to 70 ºC and at MC levels above and below the FSP.

* In the English system, thermal conductivity (k) is measured as the quantity of heat in British thermal units (Btu) that will flow in 1 h through a body 1-in thick and 1 ft^2 when a difference if 1 ºF is maintained between the 2 surfaces. 1 Btu in / h ft^2 ºF = 0.12404 kcal m / h ºC

Materials and Methods

Samples were collected from the segments of logs used in a previous study (Usta, 2004), and prepared in equilateral triangle cross-sections (Figure 1). Long (in 25 mm length for 240 samples) and short samples were produced (10 mm for 60). As the physical properties of Caucasian fir, such as the FSP (32.1%) and the volumetric shrinkage value (βv: 11.8%), had been determined in our preliminary report (Usta, 2004), the short samples were used for the determination of oven-dry density and the initial MC by an oven-drying process.

The levels of MC were 40% and 20% due to the FSP. The long samples were marked for triplex flow and T&R (transverse flow, a combination of tangential and radial directions), and dried sequentially using the schedule K in accordance with the recommended kiln-drying schedule for *Abies* species (Pratt, 1974). Thereafter, T&R samples were double sealed with ABS solvent cement (polymer dissolved in methyl ethyl ketone, Durapipe).

Data assembly and experimental processing were conducted according to the "novel guide" (Usta and Hale, 2004). A flow chart was also designed for the determination of thermal conductivity according to the literature (MacLean, 1941; FPL, 1952; Deliinski, 1977; Tsoumis, 1991) (Figure 2). To observe the effect of temperature on the coefficient of thermal conductivity (k) of Caucasian fir, thermal conductivity at 0 °C (k_0) was initially determined. Since k_0 is mainly influenced by changes in MC and nominal density (R, based on oven-dry weight and volume at the current MC), R was determined by the following relationship: R = (βv / FSP) using data previously reported by Usta (2004).

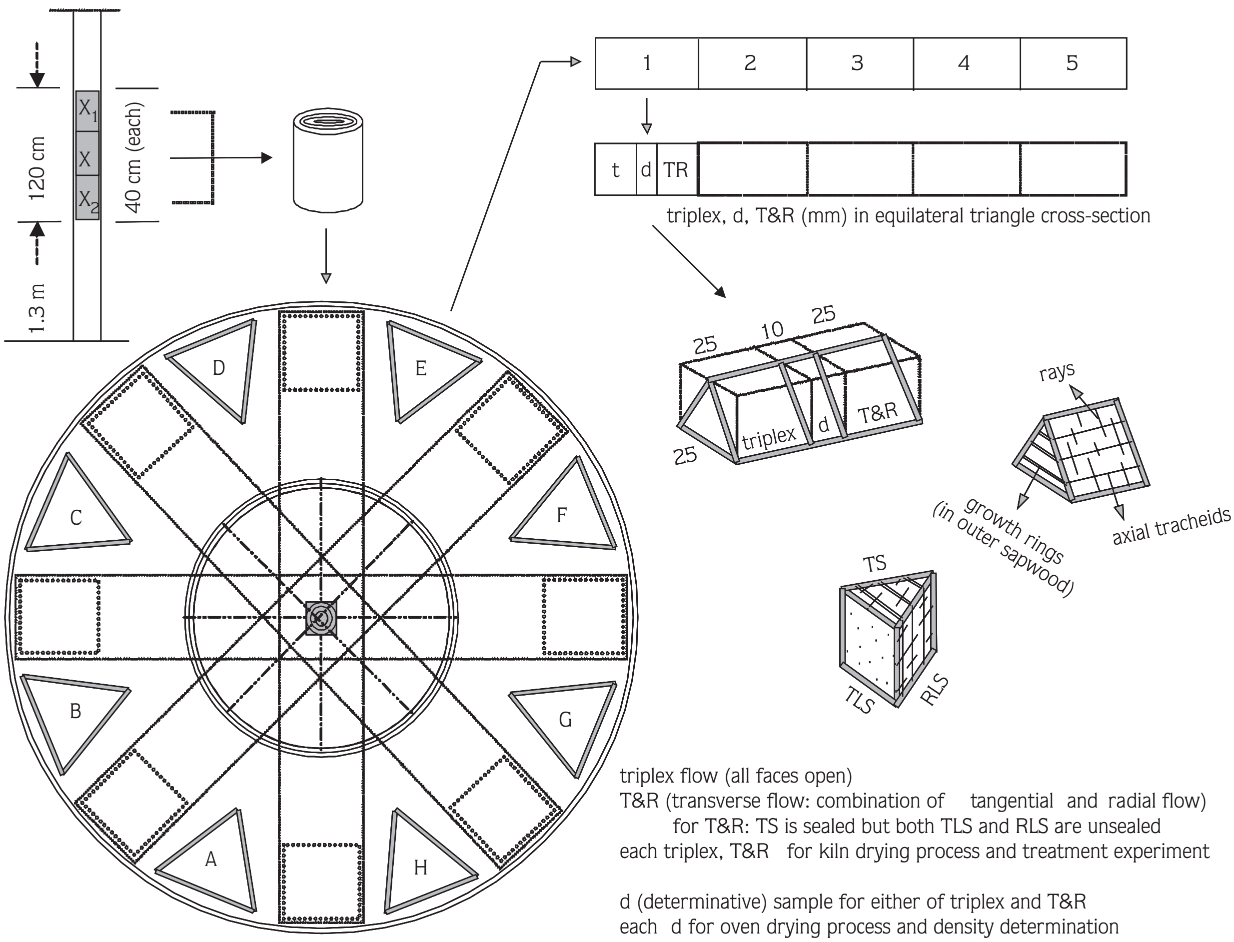

Figure 1. A diagram showing the collection and preparation of the experimental samples in equilateral triangle cross-section for determination of thermal treatability of Caucasian fir (dimensions not to scale).

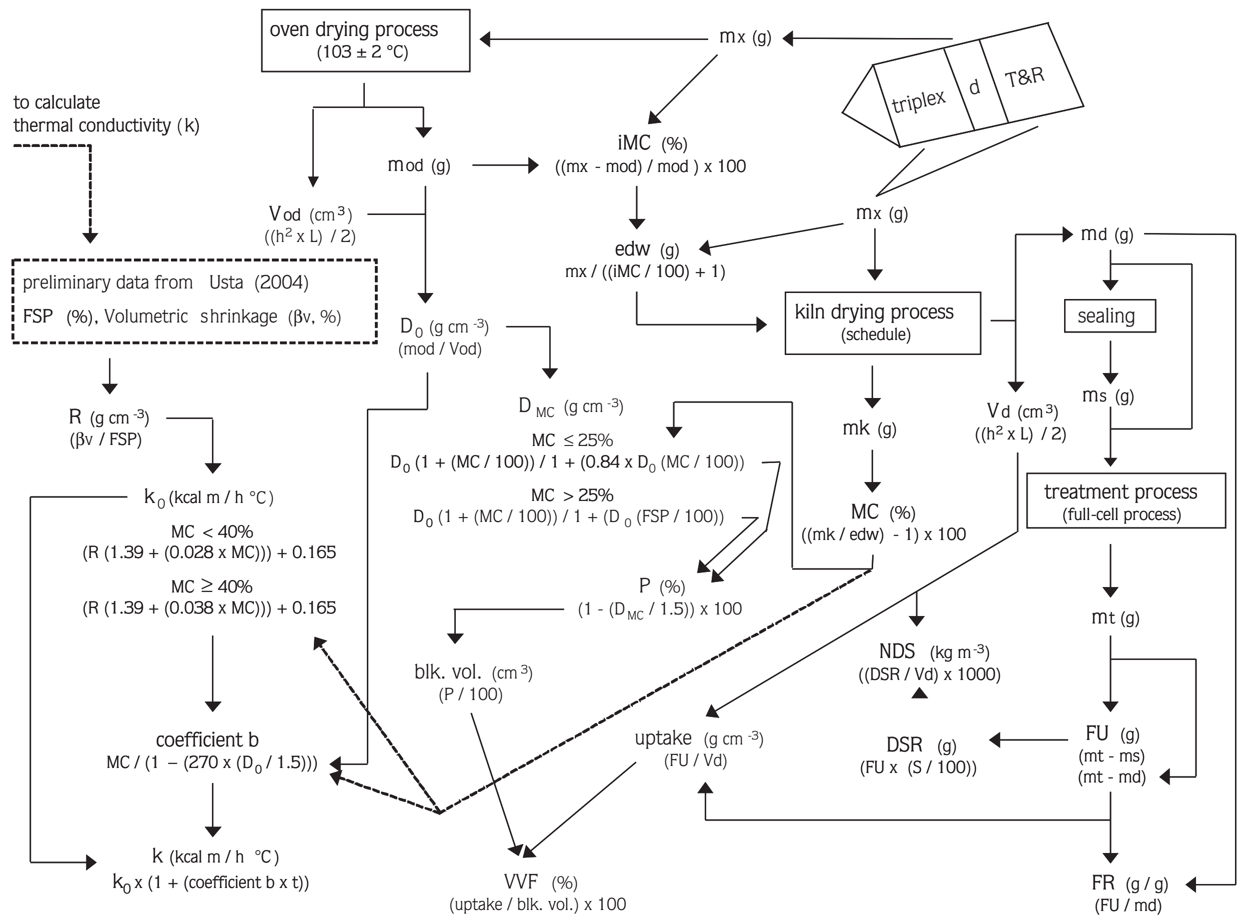

Figure 2. A schematic representation of data assembly and the experimental processing. Citations of abbreviations and definitions used in this guide were shown sequentially on the base of the process. FSP (%), fibre saturation point; R (g cm^{-3}), nominal density based on oven-dry weight and volume at the current moisture content; k_0 (kcal m / h ºC), coefficient of thermal conductivity at 0 ºC; coefficient b, a proportionality coefficient related to the effect of density and moisture content (MC); *k* (kcal m / h ºC), coefficient of thermal conductivity by the effect of temperature; t (ºC), temperature; mx (g), the green mass; Vod (cm^3), block volume after oven drying; mod (g), oven-dried mass; D_0 (g cm^{-3}), oven-dry wood density; D_{MC} (g cm^{-3}), wood density at respective moisture contents (MC ≤ 25%, equal to-and/or-below 25 percent moisture content; MC > 25%, above 25 percent moisture content); P (%), porosity; blk. vol. (cm^3), the amount of space available in a given block volume; iMC (%), initial moisture content; edw (g), estimated dry weight after kiln drying; mk (g), mass of the sample for control the current MC at regular intervals; MC (%), current moisture content at regular intervals during drying; Vd (cm^3), block volume after kiln drying; md (g), mass after kiln drying (before treatment); ms (g), sealed mass; mt (g), mass after treatment process; FU (g), fluid uptake (the gross preservative solution absorption), FR (g/g), fluid retention (preservative retention on a whole-block basis); NDS (kg m^{-3}), net dry salt absorbed after impregnation; DSR (g), dry salt retention on the base of its strength; S (%), strength of solution; uptake (g cm^{-3}), dry salt loadings based on weight per volume; VVF (%), void volume filled by preservative solution.

Thermal treatment process: Recent efforts concerning the thermal treatment of wood have led to the development of several new processes introduced on the European market during the last few years (e.g., Plato-process (PLATO BV, the Netherlands), Ratification Process (NOW New Option Wood, France), Bois perdure (BCI-MBS, France), OHT-process (Oil-Heat Treatment, Menz-Holz, Germany), and Thermo-Wood process (Stora, Finnforest, Finland)). These processes have in common the treatment of sawn wood at elevated temperatures in the range between 160 and 260 ºC. The main differences between the processes are to be seen in the process conditions (process steps, oxygen or nitrogen, steaming, wet or dry process, use of oils, steering schedules etc.) and they have been published in several patents (EP0018446, 1982; EP892031709, 1989; EP0612595, 1994; EP0623433, 1994; EP0622163, 1994; EP0759137, 1995; US5678324, 1997).

In comparison with the existing industrial processes overviewed, our thermal treatment process has been designed on a pilot scale with heating as the solution to lower temperatures. The treatment operation was carried out using a model pressure treatment plant according to BS 4072 (1987 part 2). The samples were treated with a 2% concentration of heated tanalith-C at 14 elevated temperatures from 5 to 70 °C, in addition to a non-heated (control) treatment, by a mild full-cell treatment schedule of 15 minutes vacuum (–0.84 bar) followed by pressure (1 bar) for 5 min. No final vacuum was used. A non-heated (control) treatment was also performed for comparisons. Each sample was weighed before and after a 5 min drip period following treatment to obtain the theoretically maximum possible solution uptake and dry salt absorption based on void volume (%) (McQuire, 1975).

Results and Discussion

Thermal conductivity: The coefficient of thermal conductivity (k) at elevated temperatures was calculated based on R (0.368 g cm^{-3}) (Figure 3). It was observed that the coefficient of thermal conductivity at 0 ºC (k_0, kcal m / h ºC) was 0.109 (0.883 Btu in / h ft^2 ºF) at 20% MC and 0.153 (1.236 Btu in / h ft^2 ºF) at 40% MC. Furthermore, the results indicate that k is a linear function of the temperature. When this relationship was established for each MC separately, there was a good correlation (R^2 = 1) between temperature and k (Figure 3). Let $k_{i,j}$ be the thermal conductivity at temperature i (ºC) and MC j (%). The extreme values were $k_{5,20}$ = 0.110 and $k_{5,40}$ = 0.157, and $k_{70,20}$ = 0.130 and $k_{70,40}$ = 0.211. The ratios $k_{5,40}/k_{5,20}$ and $k_{70,40}/k_{70,20}$ thus became 1.42 and 1.62, respectively. The following relationships are suggested: (y = 0.153 + 0.0041 x) for MC 40% and (y = 0.109 + 0.0015 x) for MC 20%. It should be noted that these experimental results show good agreement with data from the literature for a refractory softwood species (Steinhagen, 1977; Tsoumis, 1991).

Although the thermal conductivity of Caucasian fir appeared to be fairly low at either MC level, we assume that these values reflected intrinsic information on the internal properties and fine structure of cell wall constituents of this species. This characteristic renders Caucasian fir eminently suitable for use in timber frame house construction, indoor panelling, and wall cladding and/or sheathing. In addition, Siau (1984) stated that the low conductivity of wood accounts for the considerable time interval required to bring a large-diameter pole or bolting to a uniform, elevated temperature by the application of heat to its external surfaces. Such unsteady-state heating is, therefore, an important part of wood treatment.

Treatability by heated solution: The effects of temperature on the percentage of void volume filled (VVF%) with heated tanalith-C at different MC levels above and below the FSP are given in Table 1 for both triplex and transverse (T&R) flow, and changes in VVF% at the elevated temperatures from 5 to 70 ºC are shown in Figure 4.

In general, the results showed that there are upward and downward trends in VVF% at the elevated temperatures at either MC level. All these trends indicated the dependence of permeability (fluid flow) on MC and partially on the temperatures. In regard to the elevated temperatures, the trends at the dry samples (at 20% MC) indicated a close relation to the VVF% variation pattern in

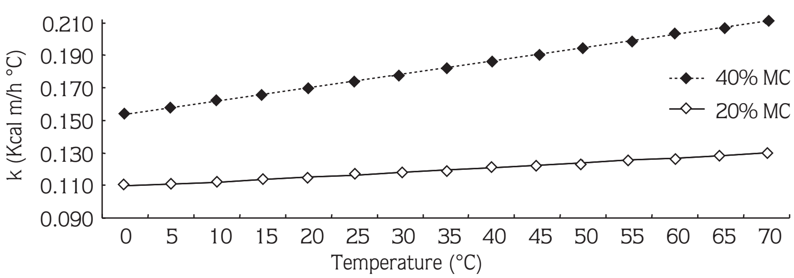

Figure 3. Evolution of the coefficient of transverse thermal conductivity in Caucasian fir above and below the fibre saturation point over the elevated temperature. MC: Moisture content

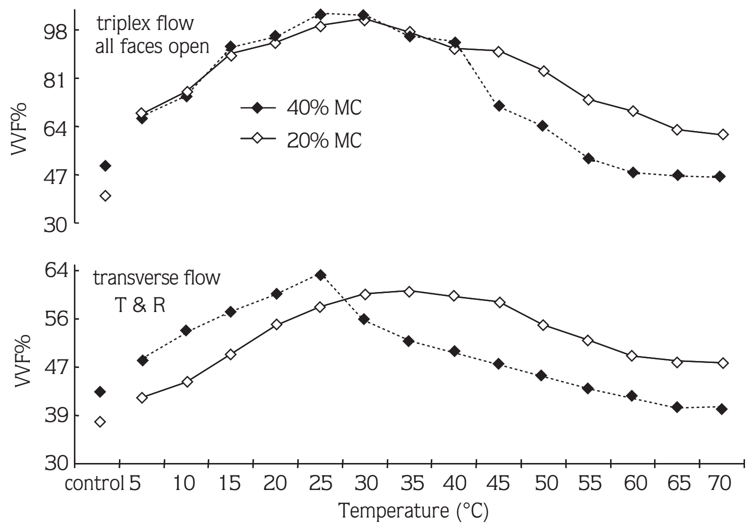

Figure 4. Changes in the percentage of void volume filled (VVF%) with heated preservative solution at elevated temperatures for triplex flow and transverse flow in 40% and 20% MC (moisture content).

either flow direction. However, the trends of the wet samples (at 40% MC) did not show a specific pattern. As shown in Table 1, VVF% increased and reached a peak at around 30 °C at both MC levels in each flow direction, after which it decreased either dramatically (at 40%) or slowly (at 20%) to around the level of the non-heated (control) samples.

In addition, the VVF% variation at temperatures from 5 °C to 25 °C in transverse flow appeared to be always greater at 40% MC than at 20% MC, and reached the highest value at 25 °C. This trend was also seen in triplex flow. According to Langrish and Walker (1993), this outstanding performance could be technically suitable and advantageous for thermal treatment in the artificial drying of green wood, where the rapid conduction of heat throughout the stack is essential.

It has been noted that the thermal treatability of Caucasian fir based on the trend of VVF% variations over the designed levels of temperature may be of 3 different types for either MC level in each flow direction (with a statistically significant difference at $P < 0.05$) (Figure 4).

In type 1 (consistent with either flow direction), VVF% increased for temperatures increasing from 5 °C to 25 °C at 40% MC, and from 5 °C to 30 °C at 20% MC. In the case of the highest value, VVF% was numerically superior at 30 °C in triplex flow, and at 35 °C in transverse flow.

In type 2 (pattern of transverse -T&R- flow), VVF% at 40% MC decreased dramatically after 25 °C and assumed lower values in comparison with those for 20% MC. In addition, the results of VVF% at the higher temperatures showed a similar pattern for either of these 2 MC levels, i.e. there were steady changes after 60 °C, with narrow fluctuations at 40% MC.

In type 3 (pattern of triplex flow), the VVF% variation at 20% MC showed trends similar to those observed for transverse flow. However, there was a different trend at 40% MC. The VVF% variation at 40% MC was initially consistent with the trends at 20% MC until 40 °C, but after that it apparently decreased.

It is clear from Table 1 and Figure 4 that Caucasian fir has low thermal treatability features, especially in the transverse direction (perpendicular to the fibre axis), where there is high resistance to flow due to the interruption of the path by the poorly conducting air-filled lumens. Based on the VVF% with heated tanalith-C, it was observed that the greatest preservative uptake occurred at around 30 °C in both transverse and triplex directions, and higher temperatures did not improve the uptake most likely due to the effects of vaporisation, particularly at higher MC (40%). This result indicates that heating the solution causes the air in the wood to expand and some of it is forced out. Consequently, 20% MC and temperature around 30 °C (not less than 25 °C and not

Table 1. Effect of temperature on the percentage of void volume filled (VVF%) with heated preservative solution in triplex and transverse (T&R) directions at 40% and 20% moisture content.

Temperature (°C)	Triplex flow		Transverse (T&R) flow	
	40%	20%	40%	20%
Control	54.9 ± 0.16 a	59.3 ± 0.03 a	43.0 ± 0.99 a	40.1 ± 0.18 a
5	67.5 ± 1.09 b	68.0 ± 1.61 b	48.2 ± 0.24 b	41.7 ± 1.67 ab
10	74.6 ± 0.84 c	76.1 ± 0.67 c	53.3 ± 0.27 c	44.2 ± 0.85 b
15	91.3 ± 3.17 d	89.5 ± 211 d	56.5 ± 0.51 d	49.3 ± 0.28 c
20	95.2 ± 0.76 d	93.5 ± 2.09 d	59.9 ± 0.92 e	54.2 ± 0.23 dh
25	102.8 ± 0.08 e	99.3 ± 0.93 e	63.3 ± 0.30 f	57.6 ± 0.95 eg
30	103.0 ± 0.06 e	101.6 ± 0.99 e	55.5 ± 0.51 gcd	59.8 ± 1.31 ef
35	95.1 ± 0.07 d	97.1 ± 0.59 e	51.6 ± 0.58 hc	60.3 ± 0.32 f
40	93.3 ± 0.25 d	91.2 ± 1.08 d	49.8 ± 1.78 hib	59.4 ± 0.78 f
45	70.9 ± 2.40 fbc	90.4 ± 1.23 d	47.5 ± 0.51 ikb	58.4 ± 0.40 fg
50	64.4 ± 2.13 gb	83.7 ± 0.14 f	46.1 ± 0.94 k	54.4 ± 0.40 h
55	52.5 ± 2.36 a	73.4 ± 0.68 c	43.4 ± 1.46 ma	51.7 ± 0.74 i
60	47.4 ± 1.05 h	69.4 ± 0.40 b	41.9 ± 0.86 mna	48.9 ± 1.16 kc
65	46.6 ± 0.62 h	62.7 ± 3.41 ba	40.1 ± 1.05 np	48.1 ± 1.05 kc
70	46.4 ± 0.83 h	60.9 ± 1.69 a	39.9 ± 0.87 p	47.7 ± 1.73 kc

Values shown are the means with the standard deviations of 4 replicates
Means that are not significantly different at $P < 0.05$ level (Tukey comparison test) have the same letter in a given column.
Control samples were treated with non-heated preservative solution

more than 35 °C) were highlighted as reliable specifications for heating the tanalith-C.

These experimental findings provided important clues for the improvement of preservative uptake in Caucasian fir, which is a refractory softwood species, treated with heated tanalith-C. In the future, however, more detailed studies on this species at different experimental conditions—including the use of other preservative chemicals—will be necessary to identify efficient utilisation for the thermal treatment process.

Conclusions

This study has shown preservative uptake in Caucasian fir—a refractory species—can be improved by heated tanalith-C (CCA) to make it more suitable for use with a mild thermal treatment process under the conditions of 20% MC and 30 °C temperature. This application may provide new industrial uses in wood preservation. As the piping—for transferring the heated preservative solution to the cylinder—was designed in this study without steaming, additional work on using steam piping between the tank and the cylinder is now under way. Laboratory studies will soon be performed in order to optimise the processing parameters and to evaluate the process economics.

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