

MECHANICAL PROPERTIES OF SCOTS PINE (*Pinus sylvestris*) WITH IMPREGNATION MODIFIERS

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Abstract:

Wood modification is a promising alternative to improving the performance of wood without harmful side effects. This study investigates impregnation modification and its effects on the mechanical properties of solid wood, which has been paid limited attention to previously. Scots pine (*Pinus sylvestris*) wood was impregnated with four modifiers with an impregnation chamber. The mechanical properties of the modified samples were determined with bending strength, hardness, and impact strength tests. The modifiers penetrated into the wood differently, depending on the quality of the modifier. The melamine-impregnated samples attained the best impregnation, which had an influence on the mechanical properties, but the orientation of the effect varied. It is concluded that the significance of each mechanical property in the modified wood product must be evaluated case by case.

Key words: impregnation; Scots pine; mechanical properties; bending; hardness; impact strength.

INTRODUCTION

It is well known that wood is a natural structural material with a great number of applications. Although wood has many excellent material properties, such as good isolation and high strength in relation to weight, some of its inherent properties restrain its comprehensive utilization. These properties can be improved by modification, which means improvement of one or more of the disadvantages of wood (Hill 2006).

One of the methods of wood modification is impregnation, where the desired performance changes are achieved by filling the wood substance with an inert material. The fixation of the impregnant within the cell wall can occur either by monomer impregnation or by diffusion. In monomer impregnation, impregnate polymerization within the cell wall occurs afterwards, while in diffusion, a soluble material is diffused into the cell wall, making it insoluble (Hill 2006). The best known impregnation methods are, inter alia, full cell treatment Bethell and empty cell treatment Lowry and Rueping. The Bethell method has an initial vacuum as the first step in the process, and this is the most noticeable difference to the empty cell treatment methods (Archer and Lebow 2006).

Impregnation depends on several variables, such as the material and process properties. The longitudinal position of transport cells contributes to better penetration of the impregnant in the longitudinal direction compared to radial or tangential directions. In radial directions, the penetration passes through parenchyma rays (Larnøy *et al.* 2005; Tondi *et al.* 2013). The properties of the impregnate liquid, for example the maximum molecular diameter or hydrogen-bonding ability, also affect the penetration (Hill 2006).

Additionally, the impregnant may polymerize due to the acidity of wood, making it unable to penetrate into the cell wall layers (Chen 2009).

The moisture properties and weathering performance of the selected modifiers have been presented previously in the study of Lahtela and Kärki (2014). In addition, some studies have reported the effects of each modifier. It has been found that melamine treatment can protect solid wood against weathering and infestation by wood staining fungi, as well as reduce the water absorption (Rapp and Peek 1999; Hansmann *et al.* 2006; Lahtela and Kärki 2014). Some studies have shown that potassium or sodium silicate, commonly known as water glass, treatment enhances the durability of wood and decreases fungal growth (Mai and Militz 2004; Pfeffer *et al.* 2011). Hydrophobicity and resistance against decay and mould have been found to improve after tall oil derivate and silicone emulsion treatment (Temiz *et al.* 2008; Ghosh *et al.* 2009). In spite of several studies with selected modifiers, investigations into their mechanical properties are limited. Generally, strength losses have been found after waterborne-preservative treatment (Winandy and Rowell 2013) due to waterborne preservative chemicals reacting with the wood cell wall components (Simsek *et al.* 2010).

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OBJECTIVE

In this study, solid wood was treated with environmentally nontoxic modifiers, and the mechanical properties were investigated. Advanced modified wood products have been found to have improved moisture features, but significant improvements in wood strength have not been recorded. Improved strength properties could allow the utilization of wood in more demanding applications. Therefore, the aim of this paper is to find out the influence of modifiers on the mechanical properties of wood. Impregnation modification is also a relatively unexplored method (Hill 2006), which also creates challenges for its research.

METHOD

Scots pine (*Pinus sylvestris*) wood samples were impregnated with four modifiers, melamine, water glass, silicone, and tall oil. Before the impregnation, the samples were cut and planed into the size 20 × 95 × 1000 mm. The features of the wood material were measured before treatment, and the values are given in Table 1.

Table 1

Average properties before treatment [Values in the parentheses indicate standard deviations]

Feature	Value
Density (kg/m ³)	447.6 (46.9)
Moisture content (%)	11.7 (2.5)
Rate of growth (mm/a)	2.7 (1.5)
Heartwood ratio (%)	45.7 (43.3)

The impregnation was carried out by using a registered pressure apparatus (YIT Teollisuus Oy), at a pressure of 10 bars for 120min, without added heat. After impregnation, the samples were dried in an oven at 103°C for 24h. Due to the more responsive qualities of the modifier, the melamine- and tall oil -impregnated samples required slightly different treatment. The tall oil -impregnated samples were allowed to stabilize for a day before drying and the melamine-impregnated samples were dried at a lower (90°C) temperature.

Impregnation was performed individually by each of the four modifier sets, consisting of 15, both sapwood and heartwood, samples. The modifiers were melamine, water glass, silicone, and tall oil. The melamine solution (Prefere 70 0592 L) was supplied by Dynea Chemicals Oy (Hamina, Finland). The water glass solution, specifically sodium silicate (Zeopol 33), was acquired from J.M. Huber Finland Oyj (Hamina, Finland) and diluted 1:1 with water. The silicone solution was made of a silicone product (001 7100) from Tikkurila Oy (Vantaa, Finland), mixed with water at the ratio 1:9. The tall oil (EP608) was obtained from Ekopine Oy (Oulu, Finland) and diluted 1:1 with wood turpentine (T2501) from Kiilto Oy (Lempäälä, Finland). The properties of the modifiers are presented in Table 2.

Table 2

Properties of used modifiers according to the manufacturers [*at 20 °C **Relative density]

	Melamine	Water glass	Silicone	Tall oil
Property				
Density (g/cm ³)	1.19*	1.38*	1.00**	0.86**
Solid content (%)	46.2 – 48.2	37.0	–	–
pH	9.7 – 10.0	11.2	c. 7	
Viscosity (mPas)	8 – 14	250	–	4 000

The results of the modification were measured by weight percent gain (WPG) according to the following equation:

$$WPG(\%) = \frac{Mm - Mu}{Mu} \times 100 \quad (1)$$

where: *Mm* is the mass after modification;

Mu is the mass before modification.

Weight gain is a commonly reported value related to wood modification (Hill 2006). In addition to WPG, similar modification figures have been reported in other ways. For example, Sint *et al.* (2013) and Tondi *et al.* (2013) have determined other values for treated samples. These values, solution uptake (SU (kg/m³)), relative solution uptake (RSU), and impregnation rate (I.R.), have been determined as follows:

$$RSU(\%) = \frac{W_3 - W_2}{W_1} \times 100 \quad (2)$$

where: W_3 is the mass of the sample after impregnation, in kg;
 W_2 is the mass of the conditioned sample before impregnation, in kg;
 W_1 is the oven-dry mass of the sample before impregnation, in kg.

$$SU(kg / m^3) = \frac{W_3 - W_2}{V_{AD}} \quad (3)$$

where: W_3 is the mass of the sample after impregnation, in kg;
 W_2 is the mass of the conditioned sample before impregnation, in kg;
 V_{AD} is the volume of the conditioned sample before impregnation, in m^3 .

$$I.R.(%) = \frac{W_w - D_w}{D_w} \times 100 \quad (4)$$

where: W_w is the wet weight;
 D_w is the dry weight.

After the impregnation and the drying, the mechanical properties of the modified wood were determined by bending, hardness, and impact strength tests. The test samples were produced from treated materials according to the used standards. The tests were carried out with 15 samples of each category, without visible cracks and knots. The set of samples consisted of 7 heartwood and 8 sapwood samples. The three-point bending and hardness (Brinell) tests were performed with a Zwick Roell Z020 testing machine in accordance with ISO 3133 (1975) and EN 1534 (2000), respectively. The specimens for the bending test were 20×20×380mm in size, and for the hardness test they were 20×50×100mm in size. The bending strength specimens were set on the testing machine in the longitudinal direction, and the penetrated surfaces of the specimens were located horizontally. This setting ensured that the effects of the modifiers were congruent in every specimen, regardless the magnitude of penetration, and the grain direction was not changed. The impact strength was determined with a Zwick 5102 Model impact tester according to EN ISO 179-1 (2010). The size of the impact test specimens was 4×10×80 mm.

RESULTS&DISCUSSION

Treatment

The impregnation results are listed in Table 3 by different parameters. The results show that the properties of the treatment solution have an effect on the penetration into wood. The WPGs of the treated wood are rather small, expect for the melamine-treated wood, whose favourable result may be caused by lower viscosity (Tondi *et al.* 2013). It has been assumed previously that the high water content of the impregnation solution may result in a weak WPG (Lahtela and Kärki 2014), which can be confirmed by the analysis of the uptake results (SU, RSU, I.R.). For example, the silicone-treated wood has nearly the same average values as the melamine-treated wood in the uptake results, but not in the WPG. The results of the WPG show great evaporation for the water glass- and silicone-treated samples compared to the uptake results, whereas the melamine- and tall oil -treated samples have minor relative evaporation from the uptake results to the final results (WPG).

Table 3

Average treatment results and standard deviations [Values in the parentheses indicate standard deviations]

Property	Melamine	Water glass	Silicone	Tall oil
WPG (%)	38.7 (24.7)	2.8 (6.2)	6.9 (8.7)	7.3 (5.3)
sapwood	57.0 (15.6)	3.5 (4.7)	12.1 (6.7)	7.7 (3.3)
heartwood	17.8 (14.3)	1.9 (7.5)	0.9 (6.7)	6.8 (6.9)
RSU (%)	76.6 (47.5)	20.0 (13.2)	67.7 (36.4)	11.4 (6.0)
sapwood	111.6 (36.4)	18.6 (7.1)	86.7 (38.2)	12.6 (5.4)
heartwood	36.6 (18.2)	21.6 (17.6)	46.0 (17.0)	10.0 (6.4)
SU (kg/m ³)	352.6 (202.5)	93.6 (64.3)	315.8 (153.4)	52.7 (26.7)
sapwood	510.7 (130.8)	84.0 (28.7)	399.8 (152.5)	57.9 (23.0)
heartwood	172.0 (84.2)	104.5 (87.7)	219.9 (81.2)	46.8 (29.3)
I.R. (%)	69.6 (42.9)	18.5 (12.2)	60.9 (31.2)	10.5 (5.8)
sapwood	101.2 (32.7)	17.1 (6.0)	77.0 (32.5)	11.7 (5.6)
heartwood	33.5 (16.6)	20.0 (16.5)	42.5 (15.5)	9.1 (5.8)

The impregnation is also dependent on the wood material properties, like aspiration, penetration direction, and the ratio of wood parts. Drying aspirates to the wood pits, which reduces the wood permeability (Langrish and Walker 2006). The penetration of the solution across the grain is more limited compared to the movement along the grain (Archer and Lebow 2006). Permeability varies between wood parts, for example heartwood is impermeable due to, inter alia, aspiration and extractives (Langrish and Walker 2006). The results of the analysis show that in addition to WPG, an uptake test is needed to confirm the results.

Mechanical properties

The mechanical properties are presented in Figs. 1-3. The average results are presented as bar charts with error bars, which reflects the standard deviations.

Bending

The effect of impregnation on the bending strength is presented in Fig. 1. The results show that the bending strength is decreased for the all impregnated samples, with a decline of 22.9 % at worst. The differences between the sapwood and heartwood samples in bending are close to insignificant. The standard deviations of the bending results are slightly controlled after the impregnation.

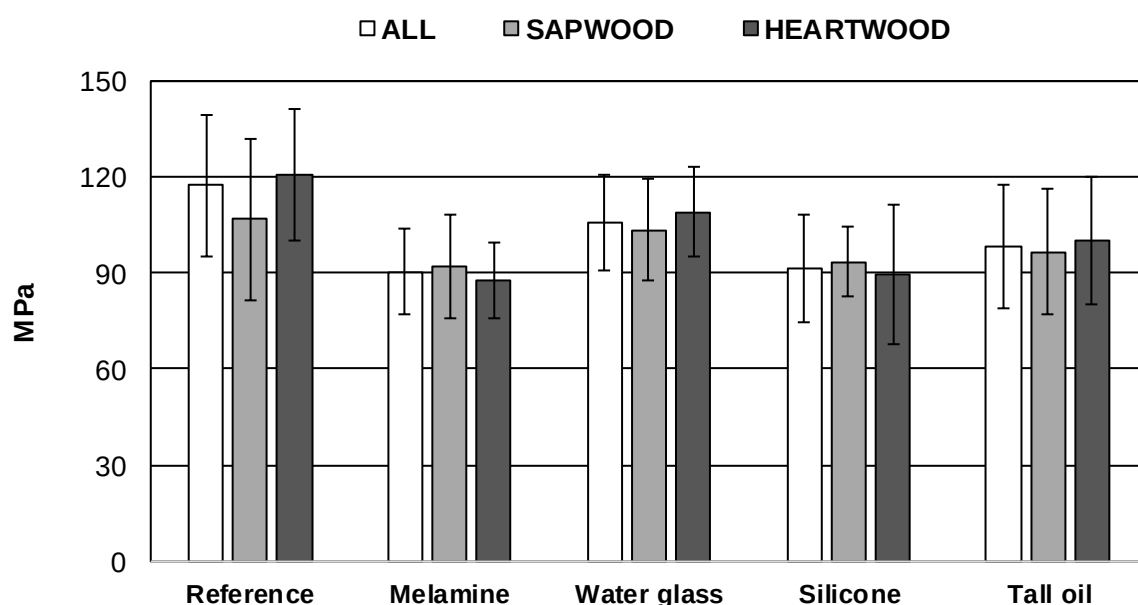


Fig. 1.

Bending strength of impregnated wood: white bars correspond to all specimens, light grey bars correspond to the sapwood specimens, and dark grey bars correspond to the heartwood specimens.

Previous studies have noted that impregnation with various modifiers may improve the bending strength of wood (Esteves *et al.* 2011, Esteves *et al.* 2014). The bending strength depends on the treatment parameters. For example, the bending strength is decreased with increasing treatment temperature (Poncsak *et al.* 2006), but a moderate treatment temperature may improve it (Rautkari *et al.* 2014). Also specific gravity, the total processing time, initial moisture content, and wood species influence the bending strength (Wang and Wang 1999; Shi *et al.* 2007; Kielmann *et al.* 2013). Kielmann *et al.* (2013) state that WPG has no major effect on the bending properties. An analysis of measurements revealed that the bending result decreased with increasing specimen dimensions (Scholotzhauer *et al.* 2015).

Hardness

Hardness was measured by Brinell hardness method, and the results are given in Fig. 2. The Brinell hardness is a reliable and widely used method in Europe for determining the hardness of wood (Heräjärvi 2004). Other test methods may require changes in the measurements due to the soft nature of wood material (Esteves *et al.* 2014).

As can be seen in Fig. 2, the melamine treatment has produced the highest increment on hardness. Gindl *et al.* (2004) have previously stated that melamine can alter the hardness of softwood to correspond

the hardness of hardwood. Water glass and tall oil treatment have stabilized the hardness of the wood between sapwood and heartwood. The hardness of the silicone-treated samples is similar to the hardness of the untreated wood. The standard deviations of the hardness results are remarkably high after impregnation modification, which may be due to the heterogeneous nature of wood.

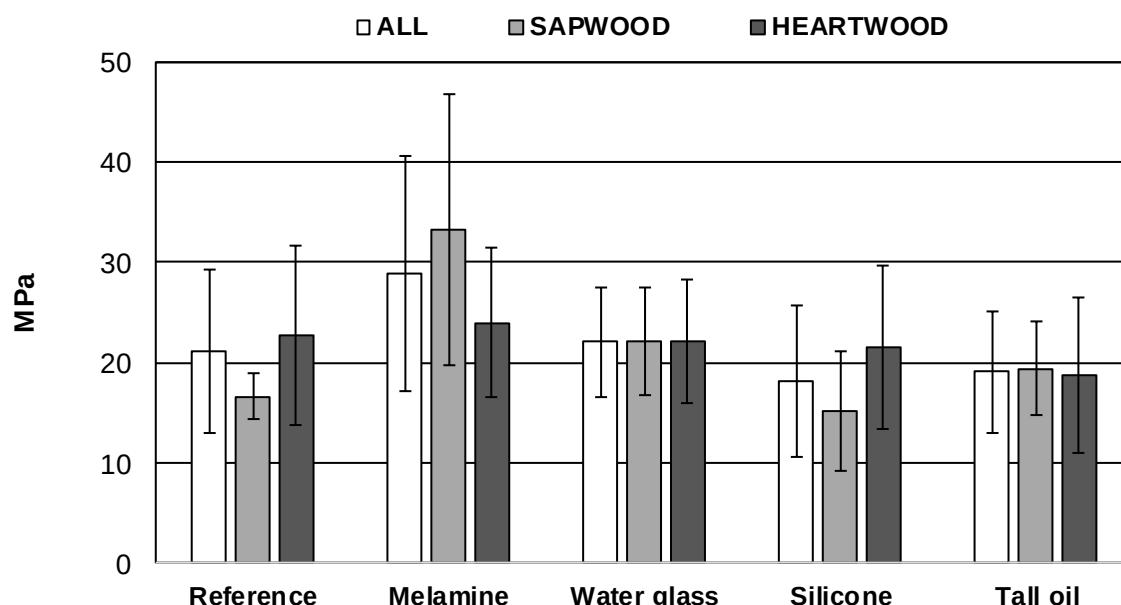


Fig. 2.

Hardness of impregnated wood: white bars correspond to all specimens, light grey bars correspond to the sapwood specimens, and dark grey bars correspond to the heartwood specimens.

The hardness of wood is dependent on the properties of native wood, for example grain direction and density (Holmberg 2000; Heräjärvi 2004). In addition, the viewing direction has an influence on the magnitude of hardness. For example, hardness is a greater in the tangential direction than in the radial direction (Kurt and Özçiğci 2009) and latewood is harder than earlywood (Rautkari *et al.* 2009; Grekin and Verkasalo 2013).

Treatment also influences on the hardness of wood, depending on the parameters. The hardness of densified wood has been found to increase (Rautkari *et al.* 2009; Candan *et al.* 2013), but for example, the effects of heat treatment on the hardness appear to be slightly ambiguous (Kocaepe *et al.* 2010; Priadi and Hiziroglu 2013). However, it has been observed that the treatment parameters have a significant effect on the hardness (Shi *et al.* 2007; Laine *et al.* 2013). The impact of the variables mentioned above can be minimized by using the same species and treatment conditions in all tests, and then the different parts of the wood cannot cause a significant deviation on the results. The impregnation solution was a singular variable in this study.

Several studies have indicated that the impregnation of wood can increase the hardness (Hansmann *et al.* 2006; Esteves *et al.* 2011, Atar *et al.* 2011). Esteves *et al.* (2014) have noted that the increase of hardness is proportional to the weight gain. The increased hardness of impregnated wood may be caused by increased density (Atar *et al.* 2011), but according to Epmeier *et al.* (2004), the denser wood does not automatically cause an increase in hardness. Gindl *et al.* (2004) have found that 2 mm is a minimum penetration depth to achieve increase in hardness.

Impact strength

Impact strength represents a distinct mechanical property which is affected by the dynamic load. Also the shorter loading time separates it from the other mechanical tests. The impact strength properties are presented in Fig. 3. Significant deteriorations were observed in the impact strength when the impregnation was successful, for example in the melamine-treated samples, of which the average value was 52.3% from the corresponding result of the reference samples. According to Kielmann *et al.* (2013), the impact strength of melamine-treated wood is reduced because the cured resin is rigid and brittle in the wood structure. The standard deviations of the impact strengths are relatively high.

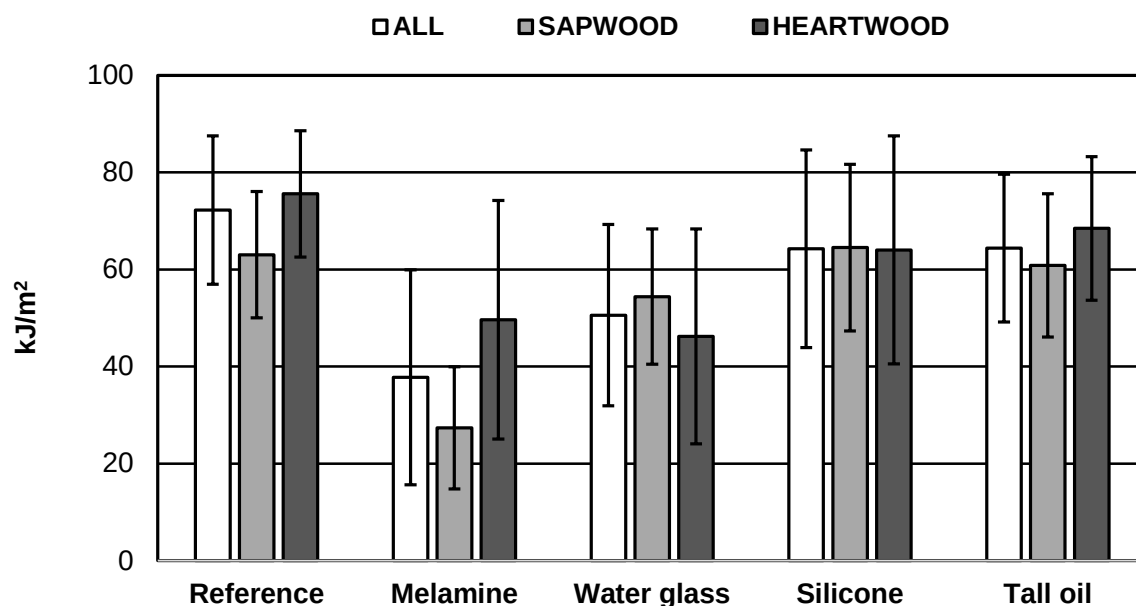


Fig. 3.

Impact strength of impregnated wood: white bars correspond to all specimens, light grey bars correspond to the sapwood specimens, and dark grey bars correspond to the heartwood specimens.

Modified wood is fragile due to the decreased mobility of the cell wall components (Dieste *et al.* 2008). The impact bending strength is sensitive for changes of wood (Kärkkäinen 2003), and the increased WPG value and heat treatment parameters reduce the impact strength (Lande *et al.* 2004, Korkut *et al.* 2008, Rautkari *et al.* 2014). Epmeier *et al.* (2004) indicate that several modification methods reduce the impact strength of wood, correlating negatively with dimensional stability. Therefore, attention has to be paid to the crucial requirements of modified wood material, for example the strength and moisture properties.

The limited number of studies and a wide variation in the reference values suggest that further studies of the impact strength are needed to get more knowledge. Kärkkäinen (2003) has stated that the impact bending strength of wood must be considered as valid only within the standard used.

CONCLUSIONS

The effect of impregnation on the mechanical properties of wood was investigated in this study. The penetration of the impregnation solution into the wood was also assessed. Awareness of the features of modified wood may allow the utilization of wood in wider applications where for example a harder surface is required.

It was found that, in addition to material properties, the qualities of the impregnation solution affected the results. The changes to the mechanical properties of the impregnated wood were different. The bending strength stayed close to stable, but the hardness and impact strength were sensitive to the modification, and the WPG played a significant role. The typical heterogeneous nature of wood material led to wide deviations in the results, which may have confused the analysis.

In the light of the results, the mechanical properties altered after impregnation depending on the impregnation solution. Some post-treatment, e.g. thermal modification, could have a more favourable effect on the wood qualities, which should be investigated in further research.

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