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SHEAR AND ADHESION STRENGTH OF OPEN AND CLOSED SYSTEM HEAT-TREATED WOOD SAMPLES

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ABSTRACT

This paper investigated the effects of heat treatment in open (atmospheric pressure) and a closed (under pressure) system on the shear and adhesion strength of Scots pine (*Pinus sylvestris*) wood. In addition, pull-off adherence testing was carried out of the coatings with water-based, polyurethane-based and oil/wax-based varnishes. Shear strength decreased significantly after heat treatment in Scots pine (31 % to 39 %) in the open system, while it decreased between 2 % and 38% in the closed system without glue. The shear strength of the wood samples glued with glue was higher than the samples without glue at the laboratory scale. The lower shear strength of modified wood could be attributed to other factors, such as the reduced chemical bonding or mechanical interlocking of adhesives, and the reduced strength of the brittle modified wood substrate. With increasing, heat treatment temperature adherence decreased. Maximum pull-off adhesion (4,80 MPa) was observed in the control samples coated with polyurethane-based varnish.

Keywords: Heat treatment, mechanical properties, *Pinus sylvestris*, pull-off adhesion, shear strength, varnish.

INTRODUCTION

Due to the negative impact of petroleum-based materials on the environment and the pressure on these materials, the use of bio-based materials becomes important. Bio-based materials play an important role in a sustainable environment (Petersen *et al.* 2001, Lee and Wang 2006, Miyagawa *et al.* 2007, Weiss *et al.* 2012). Wood, one of the most abundant renewable biopolymer composites, has been widely used as an engineering material for centuries, owing to its excellent mechanical properties, low density and low cost (Devi *et al.* 2004, Klemm *et al.* 2011, Jiang *et al.* 2017, Suttie *et al.* 2017). The use of wood material in the building industry in Europe is increasing (Eurostat 2019). Therefore, the material to be used in the construction industry is expected to show high strength. Most native wood species grown in the European region have low strength due to their structure (Larnoy *et al.* 2018). These wood species also require conservation methods (Weiss *et al.* 2012). Modification techniques that can partially eliminate these disadvantages include wood impregnation methods, or treatments with acetylation, furfurylation, reactive oil, or dimethylol dihydroxyethyleneurea (DMDHEU) (Hill 2006, Homan and Jorissen 2004, Kwon *et al.* 2007). Thermal (heat) treatment is performed via heating the wood without the use of any chemicals. The lack of chemical usage makes it one of the most common commercial wood modification methods (Lee *et al.* 2018).

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With the protective layer applied to the surface of wood material, pull-off test is the most preferred method for determining the long-term durability performance of the material (Allen 1987, Ozdemir and Hiziroglu 2007). The roughness of wood surfaces, wettability, surface energy, type of varnish and layer thickness applied, air humidity, air temperature are all properties that are affected by the adhesion (Ahola 1995). Different types of water-based varnishes applied on different wood species with different methods, hardness, gloss and surface adhesion resistance are lower than solvent-based varnishes (Sangermano *et al.* 2005). Other studies have shown that surface roughness affects adhesion resistance (Ozdemir and Hiziroglu 2007, Ozcan *et al.* 2012). According to the results obtained, heat treatment and chemical modification applied before the coating process significantly affect the adhesion resistance (Ozdemir and Hiziroglu 2007, Ozcan *et al.* 2012, Taghiyari and Samadarpour 2015). Especially with high temperature heat treatment, adhesion resistance is significantly reduced due to the degradation of hemicellulose in the wood cell wall structure (Ozcan *et al.* 2012). However, in another study removal of volatile degradation by-products during the treatment performed under vacuum is reduce hemicelluloses degradation limiting (Candelier *et al.* 2013a). Therefore, it is emphasized that the decrease in mechanical properties is less and provides better performance in the heat treatments performed under vacuum (Candelier *et al.* 2013b).

However, although some studies have examined heat treatment under vacuum, no data have been reported on the effect of such treatments on shearing and adhesion strength of wood coatings. The aim of this study was to determine the influence of heat treatment under vacuum and in the air on surface modifications. To this purpose, the changes in surface adhesion of pine wood samples subjected to thermal treatment in air and under vacuum were investigated. In addition, after the heat treatments, the surfaces of the samples were coated with three type of coatings that included water-based, polyurethane-based and oil-based varnishes, and the strength of the surface adhesion was measured.

MATERIALS AND METHODS

Materials

Scots pine (*Pinus sylvestris* L.) specimens were prepared from sapwood blocks in dimensions of 15 mm (tangential) × 75 mm (radial) × 150 mm (longitudinal). The oven-dry density of the pine samples used was 660 kg/m³.

The shearing strength test was carried out using the PVAC dispersion adhesive (commercial name-Jowacoll 102,54) together with a hardener 195,54. Both products were manufactured by JOWAT AG in Detmold. The gluing masses were prepared by weight ($\pm 0,01$ g) in accordance with the manufacturer's recommendation at the ratio of 100 mass parts of glue to 15 mass parts of the hardener. The gluing process was carried out using the following parameters: Glue application (one-sided) - 180 g/m², open assembly time - 5 min., pressing - screw clamps, Pressure - about 0,8 MPa, Pressing time - 24 hours.

Heat and vacuum heat treatment

Prior to the heat treatment, all the specimens were oven dried at 103 °C to 0 % moisture content. Wood samples were subjected to heat treatment at 190 °C to 212 °C for Scots pine samples for 2 h. In the first method, heat treatment (open system) was done in an oven (Memmert). No water vapor or other gas was in the environment. The samples were placed into the oven when the target temperature was reached. In the second method (closed system), oven-dried samples were placed in a vacuum pressure chamber (Jeio Tech) once a vacuum of 675 mmHg was achieved. Both methods were carried out on laboratory scale. The processes performed are different from the methods used in the industry. After the heat treatments, the mass loss as a result of these heat treatments was determined by oven drying (Equation 1). Finally, the modified samples were stored for two weeks in a controlled environment at 20 °C and 65 % relative humidity (RH).

$$\text{Mass loss} = \frac{m_1 - m_2}{m_1} \times 100 \quad (1)$$

Strength determination of wood

In wood science, the strength of glue bonds is frequently determined using double-cut samples containing lap joints (Figure 1a). The principal scheme of loading employed in this kind of investigation is the consequence of stipulations of the BSI EN 205 (2003) standard and involves stretching of the sample along its axis, which allows determining the shear strength of the glue bonds (Figure 1a). In addition, shear strength strengths of heat-treated samples (without glue) were also investigated.

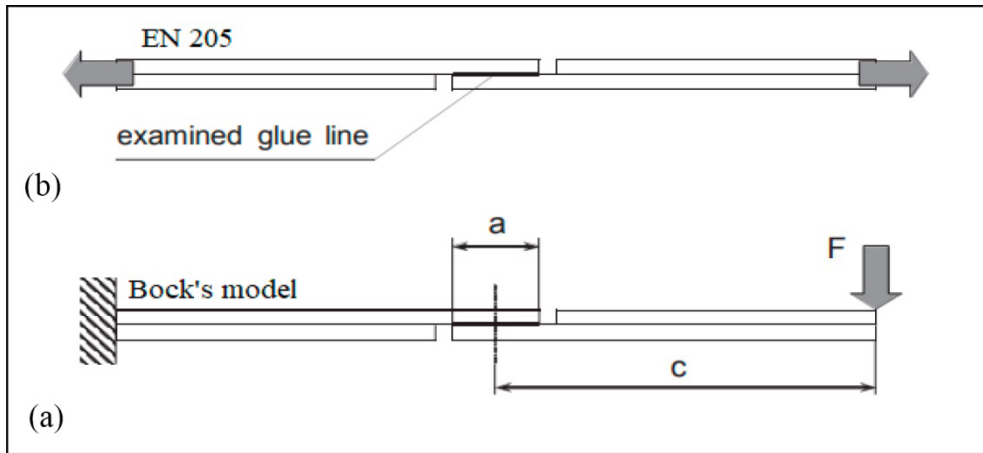


Figure 1: Methods of strength determination of glue bonds employing double-cut samples (a= 10 mm, b=20 mm, c = 55 mm).

During the performed laboratory tests, the authors determined the force F_{\max} destroying the joint. Double-cut samples with dimension: a = 10 mm, c = 55 mm (designations shown in Figure 1b- Method 2) and width b = 20 mm were used in the experiments. Only glued and non-glued samples were tested according to both EN 205 (2003) and Mock's method. Samples with surface treatment were not tested. For purposes of laboratory experiments, the total of 4 samples were prepared which were kept in an air-conditioned chamber with constant air temperature of $20\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ and relative air humidity of $65\% \pm 5\%$ for 7 days. Next, the samples were subjected to investigations in accordance with the load design shown in Figure 1b using, for this purpose, a special test machine MZ and recording the mean breaking force F_{\max} . The value of the F_{\max} force was used, according to the Bock's linear model, to determine the maximum normal stress according to Equation 2.

$$\sigma_{z, \max} = \frac{F_{\max}}{ab} + \frac{6F_{\max}c}{a^2b} \quad (2)$$

Measurement of adhesion strength of the samples

Three types of varnish, namely, water-based (AQUA), polyurethane-based (PUR) and oil/wax-based (OIL+WAX), were applied to the samples. Varnishes were applied on the samples by hand using a roller brush at a spread rate of 100 g/m^2 .

Adhesion properties of the samples were examined after the coating. The adhesion of coatings was evaluated by means of a pull-off test according to EN ISO 4624 (2002) standard.

RESULT AND DISCUSSION

Mass loss changes

The mass loss values of Scots pine samples which were heat treated after reaching oven dry weight are given in Table 1. In addition, standard deviation values are given in parentheses.

Mass losses of 4,24 % to 6,51 % were observed in the open system in Scots pine samples treated at 190 °C to 212 °C. However, lower mass losses were obtained after heat treatment in the closed system for 212 °C. Mass loss in the range of 4,40 % to 5,59 % was observed in the VHT (vacuum heat treatment) samples of Scots pine. Mass loss is one of the most important features that change during heat treatment. The mass loss depending on the heat treatment medium, temperature, and duration (Esteves and Pereira 2009, Kutnar *et al.* 2013) that is related to the wood species too. Heat treatment of wood can also cause oxidation and burning out the extractives, and evaporation of moisture content as well, eventually increasing mass loss. For Scots pine heat treated at 210 °C, a mass loss of 13,6 % was reported (Sivrikaya *et al.* 2015). In another study, Scotch Pine (*Pinus sylvestris*) heat treated at 145 °C, 165 °C and 185 °C, mass losses of 3,3 %, 7,5 % and 11 % were obtained, respectively. However, in the study presented here heat treatment at 212 °C resulted in a mass loss of only 6,51 % and at temperatures below 190 °C it was quite low (Table 1). This process is laboratory scale. Therefore, that could be one of the explanations of the difference in mass loss. The length of the process, the time at modification temperature, the fact that your oven dried your sample, all of that could have affected the mass loss of the wood. After heat treatment in water, steam and air environments, the weight loss in the wood of deciduous trees was higher than in that in coniferous trees (Esteves and Pereira 2009). However, it was revealed that the wood of coniferous trees is more sensitive to heat treatment in a dry environment. The lack of oxygen during the heat treatment in a vacuum environment reduces the weight loss values. In the study of Ferrari *et al.* (2013) weight loss values increased as an effect of a reduced vacuum medium, but at low temperatures, oxygen in the environment was not important. The results of our study showed that at 190 °C, there was no statistically significant difference between pine wood subjected to heat treatment in the open and closed system.

Shearing strength determined with the assistance of the EN 205 (2003) and bock’s model

The shear strength results and the corresponding wood failures are shown in Table 1.

Table 1: Strength determination results of non-glue and glue samples.

		non-glue samples		glue samples	
	Mass loss (%)	EN 205 standard (MPa)	Bock’s model (MPa)	EN 205 standard (MPa)	Bock’s model (MPa)
Control	-	4,35 (0,58)	1,43	6,90 (0,94)	2,05 (0,36)
HT-190	4,24 (0,23)	2,99 (0,16)	1,35	4,70 (0,91)	2,01 (0,31)
HT-212	6,51 (0,16)	2,61 (0,23)	1,20	4,69 (0,77)	1,67 (0,15)
VHT-190	4,40 (0,10)	4,28 (0,29)	1,40	6,14 (0,80)	2,70 (0,57)
VHT-212	5,59 (0,46)	2,65 (0,19)	0,98	4,97 (0,40)	1,66 (0,04)

In parentheses Standard Deviation HT: heat treatment, VHT: Vacuum heat treatment

After heat treatment, the shear strength values of the samples decreased by 31-39 % in EN 205 (2003). The shear strength was moderately improved by performing the heat treatment in the closed system. Decreases in wood mechanical resistance properties may occur depending on the heat treatment temperature and duration. These decreases are explained by the loss of material in the wood raw material (Rusch 1973, Kartal 2006). Weight losses are low in heat treatments performed in the closed system. This caused the shear strength decline to be limited.

The shearing strength values of the samples using PVAC glue increased. In the pine control samples using glue, the shearing strength value increased by 37 % compared to the samples without glue. In the control

samples, the shearing strength of 6,90 MPa decreased by 36-44 % after heat treatment in normal weather conditions and decreased to 4,70 MPa. The treatment temperature did not significantly affect the shearing strength of Scots pine, only in the case of PVAC adhesive. However, the treatment temperature significantly affected the shearing strength of Scots pine only in the case of PVAC adhesive in the closed system. With this adhesive, Scots pine wood could be bonded better at the lower vacuum heat treatment temperature of 190 °C while the opposite occurred for heat treatment in air. Heat-treatment affects permeability significantly due to loss of bound water and irreversible hydrogen bonding in the course of water movements within the vessel perforations and pore system of cell walls (Taghiyari 2013). In another study, permeabilities decreased with increased heating times (0,98 cm/min to 0,039 cm/min for 0,5 h to 160 h at 100 °C, respectively). Further dramatic decreases in permeability occurred with increasing pretreatment temperatures (Byron and Dalby 1987). The poor bonding of heat treated wood could be also explained by its poorer wettability (Bastani *et al.* 2015), which could also block the proper curing of adhesives (Boonstra *et al.* 2006). Thermal modification adversely affects adhesion resistance. This was reported by Sahin Kol *et al.* (2009) by testing different adhesives. Thereagainst, thermally modified wood could be bonded satisfactorily with hot curing adhesives with the treatment temperature having no effect on the shear strength, as shown from the decreased wood failure percentages of Scots pine after thermal modification (Table 1).

A very low wood failure percentage of 80 % to 85 % was sighted for HT and VHT-212 heat treated wood accompanied by a weak grooved surface. Similar behaviour was observed in most of the cases for HT nad VHT-190 wood with high wood failure percentages (90 %) at comparable levels to controls (95 %) and grooved surface appearance due to its brittle nature. An exception was noted for the rather lower failure of 80 % for VHT-212 heat treated wood glued with PVAc adhesive. It means that the bond line was stronger compared to the HT and VHT-190 heat treated wood itself, and therefore the reduction in shear strength was mainly due to the reduction of wood strength.

Adhesion results

The results of the pull-off adhesion testing (MPa) for Scots pine are shown in Figure 2. With the AQUA water-based varnish, heat-treatment resulted in decreased adhesion of wood. In the pine specimens, it significantly decreased the adhesion. However, adhesion strength increased in the closed system treated samples. Although there was no significant difference in the Scots pine samples under normal heat treatment, adhesion strength increased significantly in the closed system treated samples. The VHT-190 °C samples showed the same adhesion strength as the controls.

Higher adhesion values were obtained with the polyurethane varnish surface treatment compared to the other surface treatments. An adhesion value of 4,80 MPa was obtained in the Scots pine control samples. In pine samples, adhesion decreased with heat treatment application. The adhesion value decreased in parallel with the increase of the heat treatment temperature. The maximum adhesion (4,61 MPa) was found in the VHT-190 °C heat-treated Scots pine samples. The adhesion value in the heat-treated samples produced in the closed system was higher than in the heat-treated samples produced under atmospheric conditions.

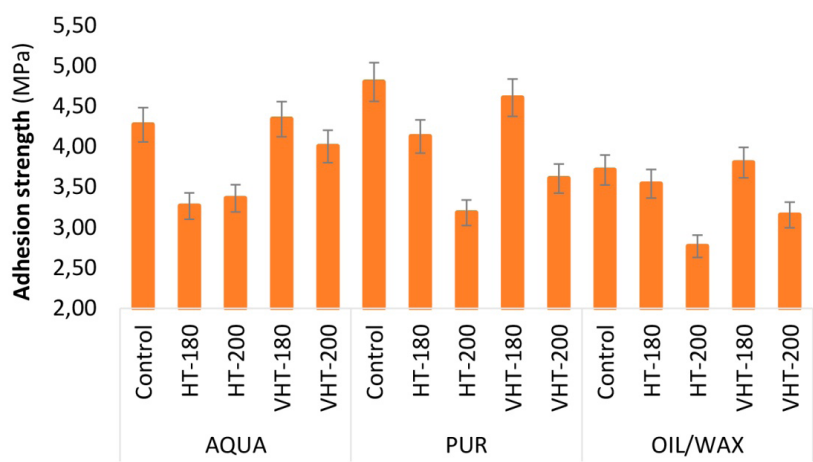


Figure 2: Pull-off adhesion (MPa) of *Pinus sylvestris* wood coated with three different varnishes.

For the surface treatment with OIL + WAX-based varnishes, adhesion values of 3,71 MPa for the Scots pine control samples. The adhesion value decreased to 2,77 MPa for Scots pine samples with heat treatment at 212 °C under normal atmospheric conditions.

Ozdemir *et al.* (2015) and Söğütü *et al.* (2016) found increased roughness and mechanical and chemical bonding between the varnish and the material, thus increasing the adhesion strength.

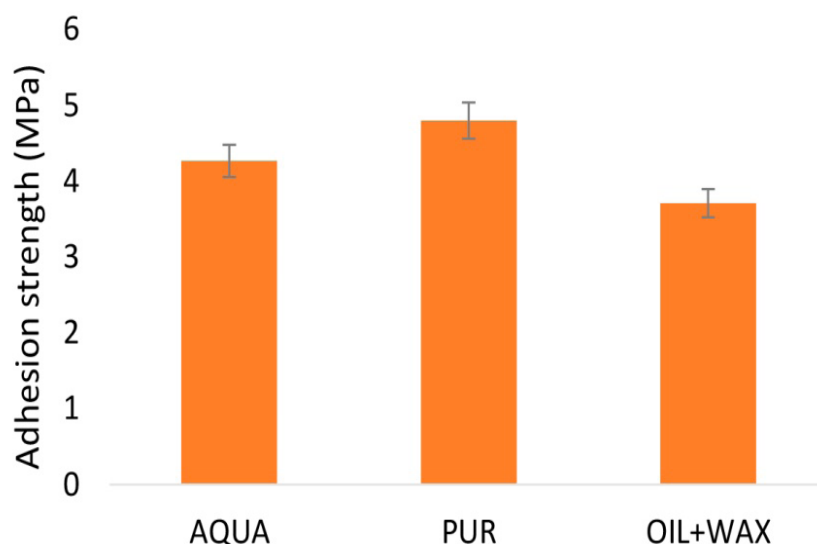


Figure 3: Pull-off adhesion (MPa) of control wood according coated.

The maximum pull-off adhesion (4,80 MPa) was observed in the pine samples coated with PUR, while the minimum (3,71 MPa) was seen in the OIL+WAX-coated samples (Figure 3).

One of the main reasons for the decreased adhesion is the existence of the less polar groups for bonding in heat treated wood (Inari *et al.* 2007, Sernek *et al.* 2008). Changes in the chemical, physical and structural properties of wood after heat treatment can affect the ability of adhesives to laminate the wood surface. Heat treatment affects adhesion in three ways. (1) Heat treatment causes “case hardening” of the wood surface, thus decreasing the wood wettability and leading to poorer adhesion. (2) The solvent-repellant case hardening is due to the change of numerous chemical groups on the carbohydrates and lignin sites on the wood surface. (3) The heavy surface oxidation, and groups changing and rearrangements (Sernek *et al.* 2008). The lower adhesion values in the heat-treated samples were caused by micro cracks on the sample cell walls. It is reported that heat treatment results in the occurrence of microcracks in the wood structure and thermal degradation of cell wall polymers (Korkut and Budakci 2009, Bayani *et al.* 2019). On the other hand, as the formation of micro checks results in a significant increase in the permeability of solid woods (Taghiyari *et al.* 2013), the coating pull-off strength was, therefore, expected to increase because the coating could more easily penetrate into the texture of the more permeable substrate and provide mechanical anchoring (Ekstedt 2002). The high adhesion value of the PUR varnishes was thought to have resulted from this situation. Micro cracks in the wood cell walls and the deeper penetration of the PUR varnish caused the formation of a chemical bond between the PUR/wood-cell wall components.

In some of the variations, the adhesion value increased. This increase was related to the decrease in moisture content (Sönmez *et al.* 2011, Hering *et al.* 2012, Goli *et al.* 2014) as well as to the irreversible hydrogen bonding in the course of water movements within the pore system of the cell walls (Borrega and Karenlampi 2010, Taghiyari *et al.* 2013). However, heat treatment at high temperatures causes the degradation of the hemicellulose found in the wood cell walls and therefore, the mechanical properties of the wood decrease. Moreover, cell walls are also thinned due to high temperatures (Hill 2006, Taghiyari 2011, Taghiyari *et al.* 2013, Taghiyari and Moradi 2014). Therefore, the significant decrease of the adhesion of the coating of wood was related to the degradation of hemicelluloses and the consequent decrease in the mechanical properties of

the substrate.

In a literature study on the effect of surface roughness on varnish adhesion strength, it was stated that the highest adhesion strength was determined with the use of polyurethane varnish and the lowest with water-based varnish (Söğütü *et al.* 2016). This was explained by the fact that polyurethane varnishes complete their reactions on the surface of the wood and therefore achieve higher varnish adhesion strength values. The low adhesion strength of water-based varnish can be attributed to the reduced wetting property as a result of chemical changes in the heat-treated wood (Kocaefe *et al.* 2008).

Due to the reduced hydrophilicity of the material after heat treatment, the water-based and oil-based varnishes could not wet the surface sufficiently and could not penetrate to provide sufficient mechanical adhesion. Therefore, the bonding between the varnish layer and the material surface was weaker than with the PUR varnish type.

CONCLUSIONS

In this study, the open and closed system treated wood samples were compared with regard to AQUA, PUR and OIL+WAX varnishes. Adhesion values of the coated samples were compared. The weight loss modification in the closed system is less than in the open system. It can be concluded that the process in the present study that focused on the goal of reducing mass loss in modified samples was successful and played an important role in improving strength properties without any damage. However, we cannot predict what the results would be for industrial-scale treatment on large-sized timber. The use of closed system modifications has its advantages for the industry, especially the fact that the modifications take less time and hence have a lower energy consumption, but some of the properties obtained could affect the types of products to produce.

Heat-treatment of solid woods at 145 °C generally tends to increase the pull-off adhesion as a result of lower moisture content and irreversible hydrogen bonding in the cell wall. However, heat treatment at 185 °C significantly decreases the adhesion due to the degradation of hemicellulose in the cell-wall structure of the wood. In addition, the lower adhesion values after the in closed system conditions were found to be closed to the values of the controls after heat treatment performed. As was demonstrated by the samples in this study, the closed system is desirable in places where a high adhesion property is required. Heat-treated samples can be used in indoor applications. However, the surface treatment applied must be determined according to the place and conditions of use.

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