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SURFACE FINISHES FOR THERMALLY MODIFIED BEECH WOOD

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ABSTRACT

Natural oils and waxes are commonly used for wooden furniture and parquet surfaces. In the present study, linseed oil, hard wax oil and hard wax were applied as the surface finish on thermally modified beech wood. Adhesion of surface finishes to wood was evaluated by both Cross-cut test and Pull-off test. The impact resistance of the surface finishes was determined according to the standard. The film hardness was determined by the pencil test according to the standard. According to the Cross-cut test, the adhesion of oil surface finishes to native wood and to thermally modified wood was the same. Adhesion of wax surface finish to the thermally modified wood was lower than to native wood. According to the Pull-off test, the adhesion of oil surface finishes to thermally modified wood was lower than to native wood. Adhesion of wax surface finish to the thermally modified wood was the same as the adhesion to native wood. Thermally modified wood showed the increased surface hardness of the selected oil and wax surface finishes under static load (Pencil hardness test); however, under dynamic load (Impact resistance test), it showed increased brittleness.

Key words: adhesion, beech, hardness, impact resistance, oil, thermally modified wood, wax**.**

INTRODUCTION

Thermally modified timber is wood which composition of the cell wall material, physical properties, mechanical, aesthetical, and biological properties are modified by exposure to the temperature higher than 160 °C and conditions of decreased oxygen availability. There are various procedures to carry out this process; most of which differ in the way they exclude air/oxygen from the system. The main heat treatment processes in Europe are covered by patents; and wood processes and wood products are signed by names such as FWD = Feuchte – Wärme – Druck Behandlung / Moisture – Heat – Pressure treatment in steam atmosphere in Deutschland, PlatoWood® in Saturated steam/heated air in Netherland, ThermoWood® in steam atmosphere in Finland, Le Bois Perdure = Perdure ® in steam atmosphere in France, Retiwood® in nitrogen or other gas atmosphere in France, Oil – Heat - Treated Wood = OHT® in vegetable Oil in Germany and TermoVuoto = VacWood® in vacuum atmosphere in Italy (REINPRECHT and VIDHOLDOVÁ 2011, SANDBERG *et al*. 2017). Different processes result in different chemical heat-induced changes in wood. A heat treatment always results in darkening of wood what is often explained by formation of coloured degradation products from hemicelluloses and extractive compounds (PONCSAK *et al*. 2011, KAČÍKOVÁ and KAČÍK 2011, VIDHOLDOVÁ *et al*. 2019). The formation of oxidation products, such as quinones, can be a reason for darker wood colour (TJEERDSMA *et al*. 1998, DZURENDA 2018a,b). The darkening is often seen positively, especially in temperate hardwoods, imitating and looking like tropical wood species (JIROUŠ-RAJKOVIĆ and MIKLEČIĆ 2019).

Besides improved stability, reduced hygroscopicity and smaller dimensional changes (AYTIN *et al*. 2015, BEKHTA *et al*. 2017, AHMED *et al*. 2017, ANDOR and LAGAŇA 2018), heat-treated wood also has some shortcomings, such as loss of toughness, reduced tensile and bending strengths, unstable colour when exterior exposure, and appearance of surface cracking (ŽIVKOVIĆ *et al*. 2008). The results HERRERA *et al.* (2018) indicate acceptable photostability of thermally modified wood, when wood is coated with decorative waterborne polyurethane and industrial UV-hardened coatings. For the same coatings, HERRERA *et al.* (2015) reported improved adhesion to thermally modified wood when compared with unmodified wood. Dimensional stability of the heat-treated wood reduces peeling of coatings. As a result of migration of fats, resins and other non-polar or less polar substances contained in wood, as well as due to degradation of hydroxyl groups in wood polysaccharides, the wood surface becomes more hydrophobic (HAKKOU *et al*. 2005, KÚDELA *et al.* 2018). When coniferous wood is treated by high temperature, the resin is poured (impregnated) from the resin canals into the surrounding parts of wood and does not protrude to the surface. It has been found that the resistance of heat-treated wood to weathering (UV light and moisture changes) is not changed significantly when compared to untreated wood, making a surface treatment with coatings necessary. Low temperature radio-frequency (RF) discharge plasma was used to improve the surface and adhesive properties of wood. The enhancement of wettability of wood is necessary to promote adhesion of adhesives and coatings (NOVÁK *et al*. 2015).

Coatings based on oil wet the surface of thermally modified wood well. The coatings based on waterborne substances, due to lower absorption of water by the surface have longer time of hardening and penetration.

Above mentioned facts significantly influence the interaction between the surface of thermally modified wood and coating material; and subsequently the mechanical and resistance properties of the surface finish. The penetration of coating systems into thermally modified wood was not found to differ from unmodified wood, although excessive penetration of solventborne oil was found occasionally for thermally modified wood. The adhesion strength of waterborne coatings depended on the system used (ALTGEN and MILITZ 2017). The mechanical properties of surface finish were researched on veneers modified by silicone resins (SLABEJOVÁ *et al*. 2018) and on pigmented surface finishes for interior use (SLABEJOVÁ and ŠMIDRIAKOVÁ 2018). CHANG *et al.* (2019) indicated that the hardness, mass retention, tensile strength, abrasion resistance, and lightfastness of oil-modified refined lacquer films decreased as more drying oils were blended with lacquer. MIKLEČIČ *et al*. (2017) measured adhesion of surface finishes to thermally modified wood after weathering aging. SAHA *et al*. (2010) monitored wetting of modified waterborne coating materials applied on thermally modified wood.

Coatings based on natural and synthetic oils and waxes, or in combination with aqueous dispersions, belong to the group of "green" and penetrating coating materials which enhance the natural wood grain and appearance. However, oil-based surface finishes have limited quality characteristics (low hardness, problematic light stability, and low resistance to liquids – detergents, food, and chemicals). They are poorly resistant to abrasion when used for stressed wood products (furniture surfaces – worktops) and wood flooring. Oils do not protect surfaces from weathering discolouration. They are only recommended for finishing of thermally modified wood products that are kept away from direct sunlight and rain (JIROUŠ-RAJKOVIĆ and MIKLEČIĆ 2019). Semi-transparent oil-based stains with small amount of colouring agent showed the weathering resistance from two to five years, although the results differed by regions (KIM 2018). Epoxidized camelina oil and acrylated epoxidized camelina oil are promising candidates for UV-curable coating applications. These oil coatings showed good pencil hardness and strong adhesion to wood substrates (LI *et al.* 2018).

The presented work deals with evaluation of selected properties of oil and wax surface finishes on thermally treated beech wood: the adhesion, impact resistance, and the surface hardness.

MATERIALS AND METHODS

Materials

In the experiments, thermally treated and thermally untreated beech boards (native) were used. The boards were radially cut. The test specimens were made from beech wood:

1 without treatment – native wood,
2 thermally modified at 175 °C for 4 hours (TM 175 °C).

Wood was provided by the company TECHNI – PAL Polkanová (Slovakia). The dimensions of the test specimens were 1000 × 100 × 20 mm. The specimens for testing of resistance to abrasion were with dimensions of 100 × 100 × 20 mm. The test specimen surface was grinded with sandpapers with grid number of P60 and then with the number of P80. For surface finishing, the following coating materials were used:

- Linseed oil (Nochema) – is used to make a base coat on wood or other absorbent surfaces under the coatings of oil or synthetic coating materials.
- Hard wax oil (Renojava s.r.o.) – is a mixture of hard wax oil, siccative and aliphatic solvent. It is used to treat all types of parquets and interior furniture with both normal and high loads.
- Hard wax (Hartwachs, Adler) – is a hard wax without solvents, based on natural oils and wax. It contains linseed oil, beeswax, carnauba wax, and cobalt-zircone siccative.

The following surface finishes were made:

1 linseed oil – 1 coat – the average film thickness of 40 ± 10 μm (linseed oil),
2 linseed oil + hard wax oil – 1 coat + 2 coats – the average film thickness of 80 ± 10 μm (linseed oil + oil),
3 hard wax – 1 coat – the average film thickness of 50 ± 10 μm (wax).

The surface finishes were made for all test specimens according to the recommendations listed in technical sheets.

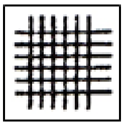
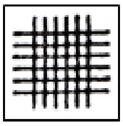
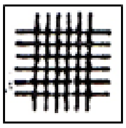
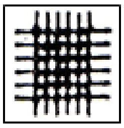
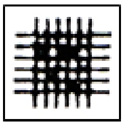
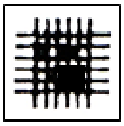
Adhesion tests

Adhesion strength of the coating films on thermally modified wood and unmodified wood was determined by the Pull-off test according to the standard STN EN ISO 4624 (2016) and by the Cross-cut test according to the standard STN EN ISO 2409 (2013).

The Cross-cut test was done as follows: a crosshatch pattern was cut through the coating film to the substrate. The adhesion of the coating film was classified according to the standard STN EN ISO 2409 (2013) (Tab. 1). The figures are examples for a cross-cut within each step of the classification. The percentages stated are based on the visual impression given by the pictures and the same percentages will not necessarily be reproduced with digital imaging.

The testing machine PosiTest AT-M (Qualitest, Canada) was used for Pull-off test. Small 20 mm diameter dollies were glued to the coating using two-component epoxy resin (Pattex Repair Epoxy). After 24 h of curing at 20 °C and a relative air humidity of 60%, perimeters of glued dollies were carefully incised to prevent propagation of failures outside the tested area. Pulling was carried at a rate of 1 mm/min up to separation of the dolly from the surface. After each test, the fracture (Fig. 1) was evaluated visually using a stereomicroscope LEICA MZ 9.5 with magnification of 4 ×.

Tab. 1 Evaluation of the cross-cut area.

Classification	0	1	2	3	4	5
Surface of cross-cut area from which flaking has occurred. (Example for six parallel cuts)	 none	 < 5%	 5% – 15%	 15% – 35%	 35% – 65%	 > 65%

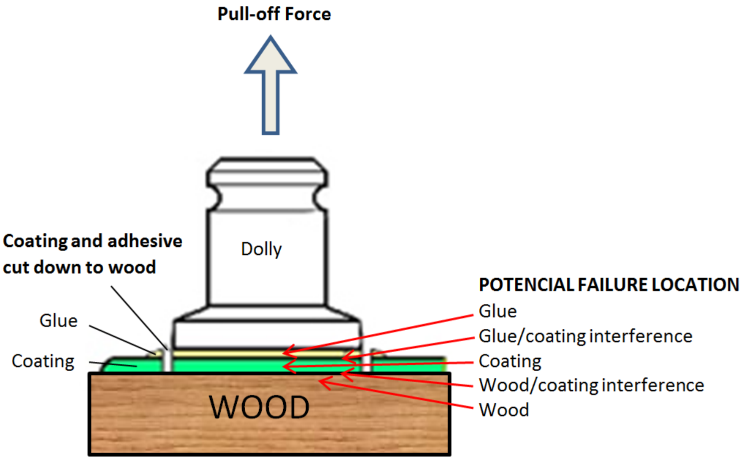


Fig. 1 Classification of failure location for the Pull-off strength test.

The impact resistance test and film hardness

The impact resistance of the surface finishes was determined according to the standard STN EN ISO 6272-2 (2011). The intrusion (diameter of the intrusion) was measured at drop height of 400 mm and the surface finish was evaluated subjectively according to Tab. 2.

Tab. 2 Impact resistance: degree and evaluation.

Degree	Visual evaluation
1	No visible changes
2	No cracks on the surface and the intrusion was only slightly visible
3	Visible light cracks on the surface, typically one to two circular cracks around the intrusion
4	Visible large cracks at the intrusion
5	Visible cracks were also off-site of intrusion, peeling of the coating

The film hardness was determined by the pencil test according to the standard STN EN ISO 15184 (2012). The results of the test were evaluated according to the pencil that scratched the surface (Tab. 3). The test started with the softest pencil (pencil number 1).

Tab. 3 Degrees of film hardness.

Pencil Number	1	2	3	4	5	6	7	8	9	10	11	12	13
Pencil Hardness	**3B**	**2B**	**B**	**HB**	**F**	**H**	**3H**	**4H**	**5H**	**6H**	**7H**	**8H**	**9H**

RESULTS AND DISCUSSION

The results of selected physical-mechanical properties of the tested surface finishes on native beech wood and thermally modified beech wood are summarised in Tab. 4.

Tab. 4 Physical-mechanical properties of tested surface treatments.

Surface finish	Linseed oil		Linseed oil + oil		Wax	
Treated surface	Native	TM 175 °C	Native	TM 175 °C	Native	TM 175 °C
Adhesion – degree [0-5] (Cross-cut test)	2	2	2	2	3	4
Adhesion [MPa] (Pull-off test)	5.41 (0.30)	5.08 (0.18)	5.19 (0.25)	4.72 (0.43)	4.43 (0.23)	4.43 (0.30)
Impact resistance – degree [1-5] – diameter of the intrusion [mm]	 2 5.6	 3 5.5	 3 5.3	 3 5.4	 2 5.2	 3 5.2
Pencil hardness – degree [1-13]	6	7	5	6	8	8

NOTE: In presence is value of standard deviation

Adhesion

The oil surface finishes (linseed oil, linseed oil + oil) showed good adhesion at the Cross-cut test. The surface finish has flaked along the edges and/or at the intersections of the cuts. A cross-cut area up to 15% was affected on both tested wood substrates. The adhesion of wax surface finish to native wood was lower; the cross-cut area up to 35% was affected on native wood (classified as 3) and up to 65% (classified as 4) on thermally modified wood. This situation is documented in Fig. 2.

NATIVE	TM 175 °C
WAX	

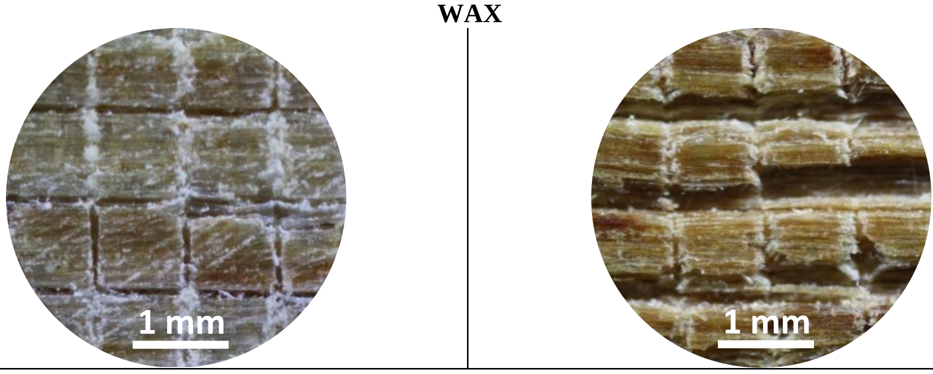


Fig. 2 Grid damage on surface finish after Cross-cut adhesion testing.

NEJAD *et al.* (2013) reported the adhesion polyurethane and acrylic-based coatings on oil-treated wood and native wood. A cross-cut area up to 5% was affected on native wood, but on oil-treated wood, a cross-cut area greater than 65% was affected.

The linseed oil reached the highest adhesion measured by Pull-off strength test and the wax the lowest one. The analysis of the average adhesion of these surface coatings to native wood and to thermally modified wood by 2-factor ANOVA showed that not only the type of coating has a significant influence on the adhesion, but also the thermal modification has the influence on the adhesion (p-value < 0.0001, respectively p-value = 0.001; Tab. 5). Interaction between coating and thermal modification was found to be statistically less significant (p-value = 0.044; Tab. 5). The analysis of adhesion to native wood and to thermally modified wood by Duncan test showed that statistically less significantly differences was found for both linseed oil surface finish and linseed oil + oil surface finish (p-value = 0.009, respectively 0.008; Tab. 5). The adhesion of wax surface finish to the surface of native wood and thermally modified wood was at same level (p-value = 0.974; statistically insignificant; Tab. 5).

Tab. 5 Basic analysis of variance for adhesion.

Factors	Sum of Squares	Degrees of Freedom	Variance	F-test	Level of significance *p-value*
Treated surface (native wood – thermally modified wood)	1.072	1.000	1.072	12.476	0.001
Surface finish (linseed oil – linseed oil + oil – wax)	6.844	2.000	3.422	39.827	0.000
Interaction treated surface * surface finish	0.570	2.000	0.285	3.320	0.044
Absolute member	1427.303	1	1427.303	16611.35	0
Error	4.640	54.000	0.086		

The important are not only the measured values of adhesion, but also the analysis of the damaged area after pull-off testing. Fig. 3 shows that pulling-off the dolly from the surface of native wood with linseed oil surface finish caused pulling-off the wood surface layer up to 1 mm deep. The adhesion was higher than cohesion of wood surface layer, as expected. The same damage was noticed also after pull-off test on thermally modified wood with linseed oil surface finish (Fig. 3). It can be assumed that the adhesion of the coating film was higher than the cohesion of wood surface layer. From Tab. 4 follows the cohesion of thermally modified wood surface layer adhesion with the linseed oil was lower than the cohesion to native wood.

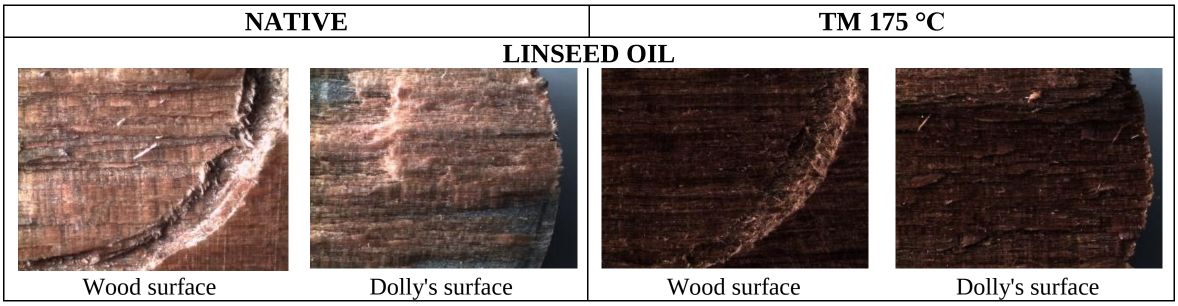


Fig. 3 Surfaces of wood and a dolly after pulling-off the surface finish of linseed oil.

After pull-off test on native wood with linseed oil + oil surface finish (Fig. 4), the damage was on the interface of linseed oil + oil, as supposed. Pulling off the wood fibres occurred on the area smaller than 5%. It can be stated the measured value represents the size of the coating film cohesion. Similar results were also reported by SLABEJOVÁ and VIDHOLDOVÁ (2019a) who evaluated the adhesion of the oil coating film and alkyd coating film to aged wood and to wood attacked by fungi. In Pull-off test, the cohesive break occurred mostly in the oil coating film.

In the case of thermally modified wood, the damage occurred in the surface layers of wood (Fig. 4). We assume that the measured value represented the size of cohesion of the surface layers of wood; and the adhesion and also the cohesion of the coating film were higher. SLABEJOVÁ and VIDHOLDOVÁ (2019b) described the similar damage occurred on an oil surface finish on wood attacked by fungi. The wood surface layers were weakened and the break occurred in the oil-impregnated layer of wood.

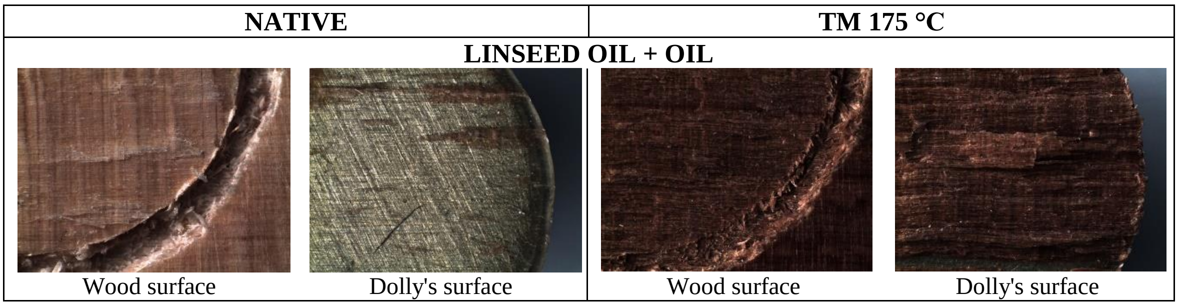


Fig. 4 Surfaces of wood and a dolly after pull-off test of surface finish of linseed oil + oil.

Fig. 5 shows that after pulling-off the dolly from the native wood surface with wax surface finish, the damage was on the interface of coating film-wood. It can be stated the measured value was the size of adhesion.

In the case of thermally modified wood, after pulling-off the dolly, the damage occurred on the interface of wood – coating film and partly in the surface layers of wood (to 30%). At breaking in the interface, the cut wood fibres were plucked from the surface. We assume the measured value was the size of adhesion. From Tab. 4, we can conclude that the adhesion of wax coating film to thermally modified wood was similar to the adhesion to native wood. The results KÚDELA *et al.* (2018) showed the substantially worsened wetting of the thermally modified beech wood. They also reported poorer drop spreading over the wood surface. This fact can affect the surface treatment quality with coating materials applied on thermally treated wood negatively. According to HERRERA *et al.* (2015) the improved hydrophobicity was not a critical factor for the application of coating systems and previous sanding process could improve coatability of modified wood.

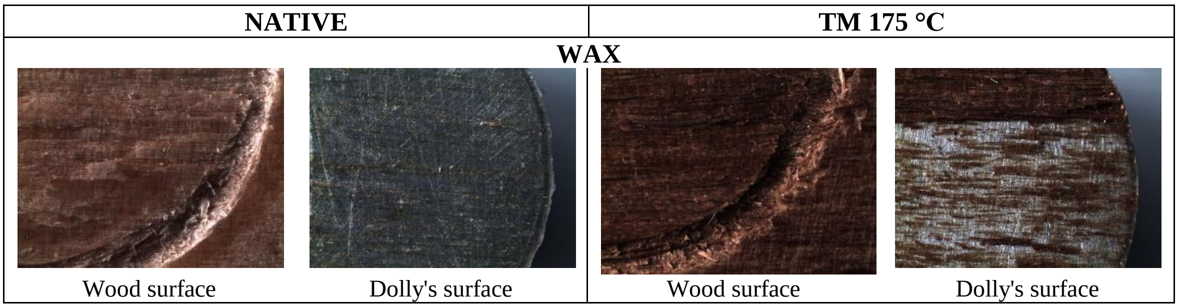


Fig. 5 Surfaces of wood and a dolly after pull-off test of wax surface finish.

Impact resistance

On thermally modified wood with the linseed oil surface finish, the damage at a drop height of 400 mm was graded as 3 (visible light cracks on the surface, typically one to two circular cracks around the intrusion), the degree of damage on native wood was of grade 2 (Tab. 4). On thermally modified wood with the linseed oil + oil surface finish, the damage at a drop height of 400 mm was graded as 3; and the same on native wood. On thermally modified wood with the wax surface finish, the degree of damage at a drop height of 400 mm was of grade 3; and the damage on native wood was graded as 2 (Tab. 4). The intrusions at a drop height of 400 mm are comparable with the intrusions on pigmented polyester-polyurethane surface finish on MDF veneered with beech veneer reported by SLABEJOVÁ and

ŠMIDRIAKOVÁ (2018). On beech veneer with silicone coating, under the same conditions, diameter of the intrusions was by a half smaller (2.5–3 mm) (SLABEJOVÁ *et al*. 2018). Impact resistance of a surface finish is influenced by quality of the coating and also by hardness of the substrate. Diameter of the intrusion depends on hardness of the substrate; and damage of the coating film depends on the film's brittleness and elasticity. TESAŘOVÁ *et al*. (2017) reported a hypothesis about the relationship between the physical-mechanical properties of lacquers films and the ultimate tensile stress of free coating films. On the tested oil surface finishes, the biggest damage was graded as 3; while SLABEJOVÁ and ŠMIDRIAKOVÁ (2018) and SLABEJOVÁ *et al*. (2018) reported the degree of damage of grades 4 or 5 on polyester-polyurethane and silicone surface finishes. However, oil-based surface finishes have low hardness and it show low brittleness.

Film hardness

The wax surface finish showed the highest film hardness (degree 8; Tab. 4); and the surface hardness was not affected by the wood surface quality (thermally modified wood). Surprisingly, surface finishes of linseed oil and linseed oil + oil had better scratch resistance on thermally modified wood than on native wood. NEJAD *et al.* (2013) reported similar results of higher pencil hardness for polyurethane and acrylic-based coatings on oil-treated wood if compared with the hardness on native wood. The surface hardness (Pencil hardness test) is a property of a coating film; but it was demonstrated that the measured hardness is influenced by the hardness of substrate.

CONCLUSION

Based on the results we can conclude:

- According to the Cross-cut test, the adhesion of oil surface finishes (linseed oil, linseed oil + oil) to native wood and to thermally modified wood was the same. Adhesion of wax surface finish to the thermally modified wood was lower than to native wood.
- According to the Pull-off test, the adhesion of oil surface finishes (linseed oil, linseed oil + oil) to thermally modified wood was lower than to native wood. Adhesion of wax surface finish to the thermally modified wood was the same as the adhesion to native wood.
- The best adhesion to wood surface was found for linseed oil surface finish, the worst adhesion for wax surface finish.
- Thermally modified wood showed the increased surface hardness of the selected oil and wax surface finishes under static load (Pencil hardness test); however, under dynamic load (Impact resistance test), it showed increased brittleness.

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