

## INTERLAYER WITH MICROCAPSULES AND ITS INFLUENCE ON THE SURFACE FINISH QUALITY OF WOOD

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### ABSTRACT

The paper deals with the quality of standard and modified surface finishes on beech, oak and spruce wood intended into exterior. Modified surface finish was created by the same coating materials as a standard surface finish, but it contained an interlayer with microcapsules. The quality of the surface finishes was assessed according to the film hardness, impact resistance, the resistance to scuffing, adhesion tests, the Cross-cut test and the Pull-off test for adhesion. The interlayer with microcapsules had no impact on the surface hardness of the surface finishes on beech wood and spruce wood, but it positively affected the film hardness of oak wood. The modified surface finish on beech wood and spruce wood showed a low impact resistance at a low drop height. It was more fragile and the impact resistance test resulted in the cracks formation. The modified surface finish was more resistant to scuffing. The adhesion of the standard and modified surface finishes to individual tree species, tested according to the Cross-cut test, was the same or minimally different. According to the Pull-off test, the standard surface finish showed higher adhesion to beech wood and oak wood when compared with the modified surface finish. On spruce wood, the Pull-off test for adhesion determined cohesion of wood surface layers.

**Key words:** adhesion, hardness, impact resistance, modified surface finish, resistance against scuffing

### INTRODUCTION

The exterior pigmented surface finish is intended to protect wood, enhance the product aesthetically, and cover the substrate. Requirements for exterior wood finishing are put for the weather resistance mainly. In the world, the research is carried out using transparent and pigmented surface finishes. “To extend the lifetime of wood and maintain its natural look, the research and development of clear coatings with minimal use of harmful chemicals has become very important” (MIKLEČIĆ *et al.* 2017).

The resistance of the surface finish to weathering is not the only property that determines the quality. Many properties – visual, chemical, resistant, physical-mechanical – give information about how the surface finish will behave during the use. The important physical-mechanical property of the exterior surface finishes is the adhesion. In several works, the influences of type of surface finish, parts of wood, moisture, wood-destroying fungi, aging, and the surface pre-treatment on the adhesion were investigated (HAZIR and KOC 2019, SLABEJOVÁ and VIDHOLDOVÁ 2019a, b, MIKLEČIĆ *et al.* 2017, VIDHOLDOVÁ *et al.* 2017, COOL and HERNÁNDEZ 2016, UGULINO and HERNÁNDEZ 2016, ŠOMŠÁK and

REINPRECHT, 2015, TOLVAJ *et al.* 2014, PODGORSKI *et al.* 2010, BULIAN and GRAYSTONE 2009, DELPECH and COUTINHO 2000, DE MEIJER and MILITZ 1998).

The surface finishes created by coating materials were evaluated according to appearance and physical-mechanical properties (HAZIR and KOC 2019, SLABEJOVÁ *et al.* 2019, SLABEJOVÁ *et al.* 2018, SLABEJOVÁ and ŠMIDRIAKOVÁ 2018, TESAŘOVÁ *et al.* 2017, SALCA *et al.* 2017, YONG *et al.* 2017, BEKHTA *et al.* 2014, MODRÁK and MANDULÁK 2013, SCRINZI *et al.* 2010).

To increase protection of wood and surface finish, the coatings can be adapted. The coatings can be modified with nano-technological products (CATALDI *et al.* 2017, MIKLEČIĆ *et al.* 2017, REINPRECHT and VIDHOLDOVÁ 2017, WETHTHIMUNI *et al.* 2016, KUMAR *et al.* 2015, KAYGIN and AKGUN 2009, LEE *et al.* 2003).

The presented work deals with pigmented surface finish intended to exterior. The surface finish contained a modifying interlayer with nano-technological product. The influence of the modifying intermediate layer on selected physical-mechanical properties of the surface finish was monitored.

## MATERIALS AND METHODS

### Test specimens

Beech wood (*Fagus sylvatica L.*), oak wood (*Quercus petraea L.*), and spruce wood (*Picea abies /Karst./ L.*) were used in the experiment. The dimensions of the test specimens were 250 mm × 80 mm × 20 mm, and the moisture content of 8 % ± 2 %.

The test specimens' surface was sanded according to the recommendations listed in technical sheets for the coating materials. The test specimens were surface finished on all sides by low-pressure spraying.

### Coating materials

The following representative coating materials (ADLER) for pigmented surface finishing into exterior were selected:

- Aquawood TIG HighRes Weiss – a base coat for wood, water-based protective impregnation with good covering ability; it is designed for a three-layer coating system with Aquawood Intercare SH and Aquawood – Spritzlack XT.
- Aquawood Intercare SH – water-based interlayer with low content of solvents, enriched with microcapsules for sealing of cracks caused by weathering.
- Aquawood Spritzlack XT – water-based pigmented finishing coat with excellent resistance to weathering.

Two types of surface finish were made:

- Standard surface finish – 1 coat with the Aquawood TIG HighRes Weiss (the average film thickness of 100 µm in wet condition) and 1 coat with the Aquawood Spritzlack XT (150-180 µm in wet condition).
- Modified surface finish – 1 coat of the Aquawood TIG HighRes Weiss (100 µm in wet condition), 1 coat of the *modifying coating material* Aquawood Intercare SH (150-180 µm in wet condition) and 1 coat of the Aquawood Spritzlack XT (150-180 µm in wet condition).

### Thickness of the coating film

Two methods were chosen to measure the film thickness:

- Destructive method – using the SuperPig SP 1100 instrument.
- Non-destructive method – using the ultrasonic instrument PosiTector 200.

### Film hardness

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 (Table 1). The test started with the softest pencil – number 1.

Tab. 1 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

### Impact resistance

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 and the surface finish was evaluated subjectively according to Table 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

### Surface resistance against scuffing

Evaluation of the surface finish resistance against scuffing was determined according to the standard STN EN ISO 7784-3 (2006). The coefficient of the resistance against scuffing  $K_T$  was calculated according to the formula:

$$K_T = (m_1 - m_2)/F \quad (1)$$

Where:  $m_1$  – specimen weight before sanding (g),

$m_2$  – specimen weight after sanding (g),

$F$  – correction coefficient of the used pair of abrasive papers ( $F = 1,052$ ).

Sanding number  $Z_T$  was calculated according to the formula:

$$Z_T = n * F \quad (2)$$

Where:  $n$  – the number of revolutions until the surface is sanded down to the surface,

$F$  – correction coefficient of the used abrasive papers ( $F = 1,052$ ).

The surface sanding number  $Z_T$  is determined when the tested surface finish is sanded down to the surface to at least 25 % of the sanded surface.

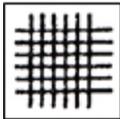
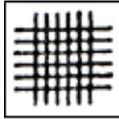
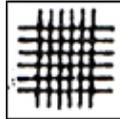
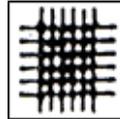
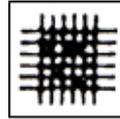
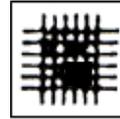
### Adhesion tests

Adhesion of the coating films to 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 testing machine PosiTest AT-M (Qualitest, Canada) was used for the 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 was evaluated visually using a stereo-microscope LEICA MZ 9.5 with magnification of 4 ×.

The Cross-cut test was done as follows: a cross hatch 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) (Table 3). 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.

**Tab. 3 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%

## RESULTS AND DISCUSSION

### Thickness of coating film

The film thickness of surface finishes was determined by two methods (destructive, non-destructive, Table 4). The coating film thickness determined by the two different methods was similar. More significant difference was noticed only for the standard surface in the case of spruce wood (destructive method 138  $\mu\text{m}$ ; non-destructive method 108  $\mu\text{m}$ ). In all cases, the thickness of the coating film determined by the destructive method was slightly higher than the thickness determined by the non-destructive method. These slight differences may be due to the fact that if measured by the destructive method, the part of the film impregnating the surface layer of wood is included in the thickness of the coating film. In the ultrasonic non-destructive method, these surface layers are probably not counted to the film thickness, but are considered as a substrate. Particularly on the spruce wood, the wood surface layer could be impregnated deeper (from a microscopic point of view).

Knowing the thickness of the coating film is not only needed to determine the adhesion by the Cross-cut test, but the thickness also affects quality of the surface finish. HUNDHAUSEN *et al.* (2018) and PALIJA *et al.* (2018) focused on factors that affect the thickness of a coating film in industrial production; because the effect of thickness on quality is significant.

**Tab. 4 Coating films thickness.**

Sample	Coating film thickness [ $\mu\text{m}$ ]					
	Beech – 2	Beech – 3	Oak – 2	Oak – 3	Spruce – 2	Spruce – 3
<b>Destructive</b>	166	256	140	270	138	218
<b>Non- destructive</b>	158	237	129	251	108	206

### Film hardness

The surface hardness of the coating film is a degree, which corresponds with the pencil which damaged the surface as the first (Table 5). Both types of surface finish in the case of spruce wood and beech wood showed the film hardness of 6. On oak wood, the standard surface finish reached the hardness of 8 and the modified surface finish the hardness of 9. The results show that higher surface hardness could be reached by a harder substrate, a slightly thicker coating film, or by the interaction of the two factors. The highest hardness of the coating

film was achieved by the modified surface finishes on oak wood (Table 5). From comparison of the two types of surface finish in the case of spruce wood and beech wood, we can conclude that the surface hardness was not affected by the interlayer. The results in this work are similar to the statements by HAZIR and KOC (2019) that higher surface hardness may not provide higher impact resistance, but a harder surface may be more fragile.

**Tab. 5 Surface hardness of coating films.**

Degree of film hardness						
Sample	Beech- 2	Beech- 3	Oak- 2	Oak- 3	Spruce - 2	Spruce- 3
Pencil number	6	6	8	9	6	6

### Impact resistance of surface finish

The impact resistance, (the diameter of the intrusion) was measured and the damage on the surface finish was evaluated subjectively according to Table 6. The modification of surface finish did not increase the impact resistance if compared to the standard surface finish. On spruce wood, the modified surface finish showed low impact resistance even at a drop height of 50 mm. At this drop height, a semi-circular crack was formed around the intrusion in the surface finish. This was also observed on beech wood at drop heights of 100 mm and 200 mm, and on oak wood at a drop height of 200 mm. We can conclude that the modified surface finish is less resistant than the standard surface finish. It is more fragile and therefore cracks appear on it sooner.

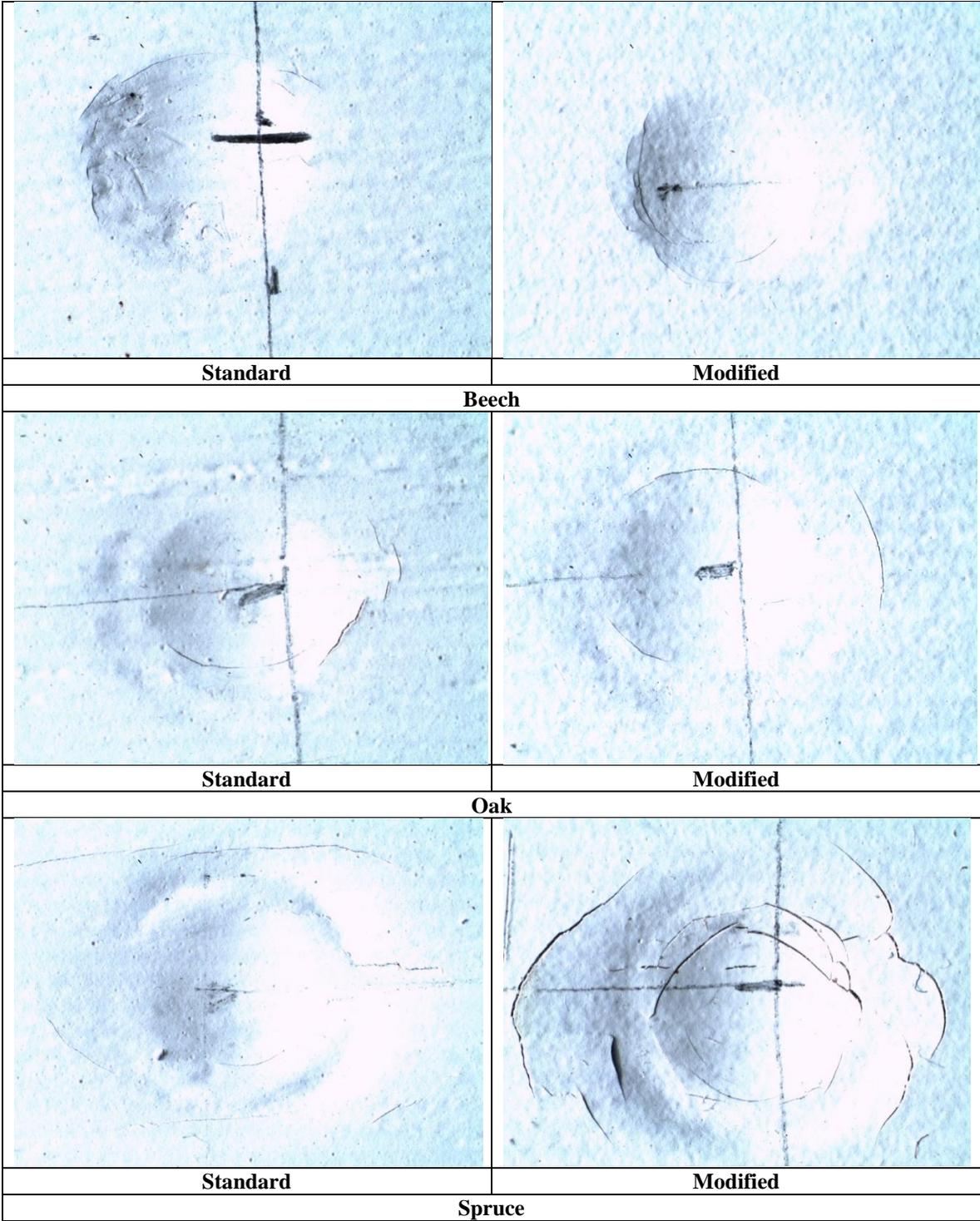
However, the total damage of both types of surface finish was greater in the case of spruce wood than on beech wood or oak wood. The lowest impact resistance (grade 5) was measured for both types of surface finish in the case of spruce wood at the maximum drop height of 400 mm (Fig. 1). In this case, the cracks were formed in the surface finish outside the intrusion. This finding shows that the modified surface finish may be more prone to cracking when hailing or during mechanical damage caused by little rocks. The purpose of the inter-layer is to seal the micro-cracks formed during aging of the surface finish in the exterior. But we found that the modified surface finish is more prone to cracking under mechanical loading.

**Tab. 6 Degree of change on the surface and diameter of the intrusions.**

Sample	Drop height [mm] and Degree of change					
	10	25	50	100	200	400
<b>BEECH - 2</b>	1	1	2	2	2	3
<b>Ø mm</b>	0	1	3	4	4	5
<b>BEECH - 3</b>	1	2	2	3	3	3
<b>Ø mm</b>	0	2	2	4	4	5
<b>OAK - 2</b>	1	2	2	2	2	3
<b>Ø mm</b>	0	2	3	4	5	5
<b>OAK - 3</b>	1	1	2	2	3	3
<b>Ø mm</b>	0	1	2	4	4.5	5
<b>SPRUCE - 2</b>	1	2	2	3	3	5
<b>Ø mm</b>	0	1	3	4	5	5.5
<b>SPRUCE - 3</b>	1	2	3	3	4	5
<b>Ø mm</b>	0	2	4	4.5	5	6

Impact resistance of the coating increases with increasing thickness of the coating to some extent (SLABEJOVÁ 2012, SLABEJOVÁ *et al.* 2018). In our case, the coating film of the modified surface finish had higher thickness than the standard surface finish and this fact also confirms the above mentioned statement. HAZIR and KOC (2019) claim that the impact

resistance of the surface finish is significantly influenced by the type of surface finish. That means it is influenced by all the components present in the coating film. This was also confirmed by our results; the modifying interlayer had an effect on the impact resistance of surface finish at some drop heights.



**Fig. 1** Surface finishes after impact resistance test at a drop height of 400 mm; evaluated visually using a stereomicroscope (with magnification of 4 ×).

### Surface finish resistance against scuffing

When evaluating the resistance against scuffing, the modified surface finish showed higher resistance than the standard surface finish on all three tree species. The coefficient of the coating film resistance against scuffing  $K_T$  for the modified surface finish was smaller than the one for the standard surface finish (Table 7).

Tab. 7 The coefficient of resistance against scuffing  $K_T$  and sanding number  $Z_T$ .

Surfac efinish resistance agains scuffing						
Sample	Beech – 2	Beech – 3	Oak – 2	Oak – 3	Spruce – 2	Spruce – 3
$K_T$	0.057	0.038	0.057	0.038	0.066	0.019
$Z_T$	841.6	1578	736.4	1683.2	946.8	1472.8

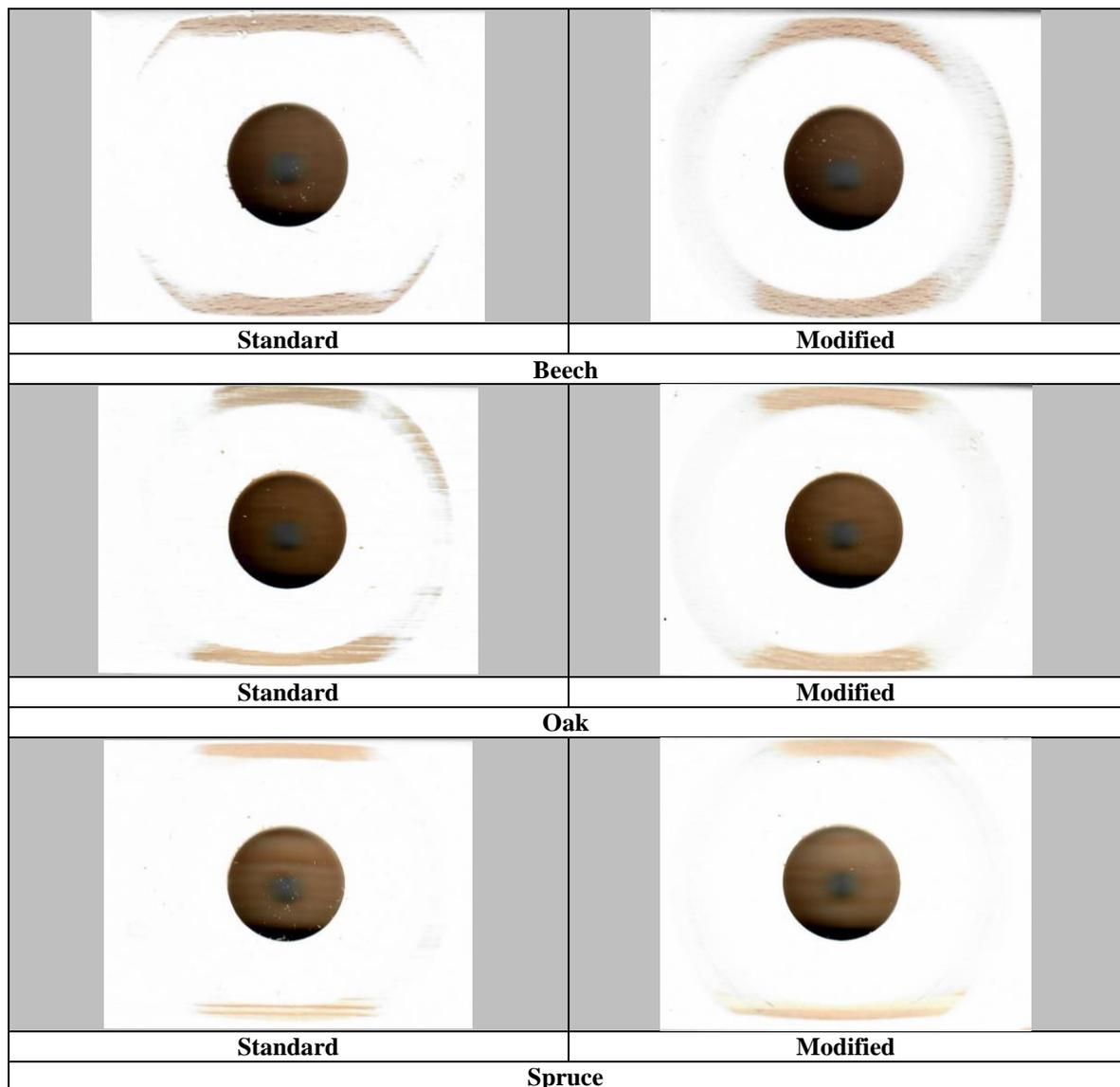


Fig. 2 Scans of the surface finishes after resistance to scuffing test.

The higher resistance against scuffing was also confirmed by the other coefficient, the sanding number  $Z_T$  (Table 7). For the standard surface finish, at least 25 % sanding to the substrate (Fig. 2) occurred after only 700 revolutions on oak wood, after 800 revolutions on beech wood, and after 900 revolutions on spruce wood. For the modified surface finish, at

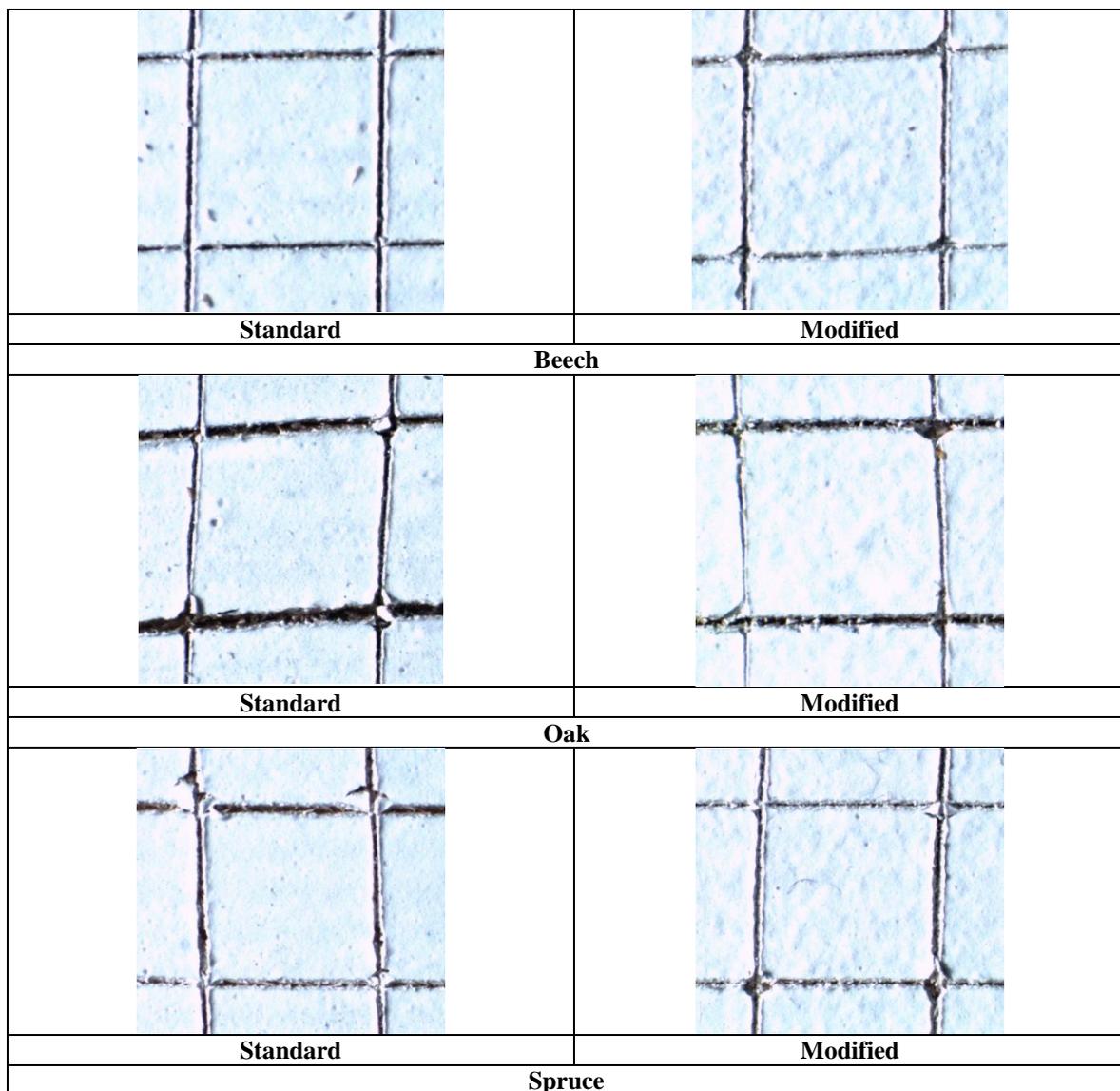
least 25% sanding to the substrate (Fig. 2) occurred after 1600 revolutions on oak wood, after 1500 revolutions on beech wood, and after 1400 revolutions on spruce wood.

**Adhesion of the coating film according to the Cross-cut test**

The adhesion determined by the Cross cut test was evaluated according to Table 8. The adhesion of both the modified and standard surface finishes to oak wood and spruce wood was of grade 2. The adhesion of modified surface finish to beech wood was of grade 2 and the adhesion of standard surface finish of grade 1. It can be stated that the adhesion of the standard and modified surface finish to the individual wood surfaces was the same or slightly different (Fig. 3).

**Tab. 8 Degree of damage at Cross-cut testing.**

	Beech-2	Beech-3	Oak-2	Oak-3	Spruce-2	Spruce-3
<b>Classification</b>	<b>1</b>	<b>2</b>	<b>2</b>	<b>2</b>	<b>2</b>	<b>2</b>



**Fig. 3 Surface finishes after the Cross-cut test evaluated visually using a stereomicroscope (with magnification of 4 ×).**

### Adhesion of the coating film according to the Pull-off test

Statistical evaluation of adhesion is given in Table 9. The results of the two-factor analysis showed that the adhesion of coating film was statistically significantly influenced by the tree species. Also the type of surface finish and the interaction "tree species – type of surface finish" showed a high significant impact on the adhesion of the coating film.

Tab. 9 Basic table of analysis of variance for specific adhesion.

	Sum of squares	Degree of freedom	Variance	F – test	p – significance level F - test
Abs. member	2900.808	1	2900.808	8239.296	0.00000000000000
Tree species	84.427	2	42.214	119.901	0.00000000000000
Type of surface finish	25.193	1	25.193	71.557	0.00000000000040
Interaction "tree species – type of surface finish"	24.165	2	12.083	34.319	0.00000000000606
Error	23.237	66	0.352		

Fig. 4 shows that the standard surface finish if compared with the modified surface finish reached higher adhesion to both beech wood and oak wood. On spruce wood, there was no difference in the adhesion between the standard and modified surface finishes. The modified surface finish in the case of spruce wood had a higher variance of values of adhesion.

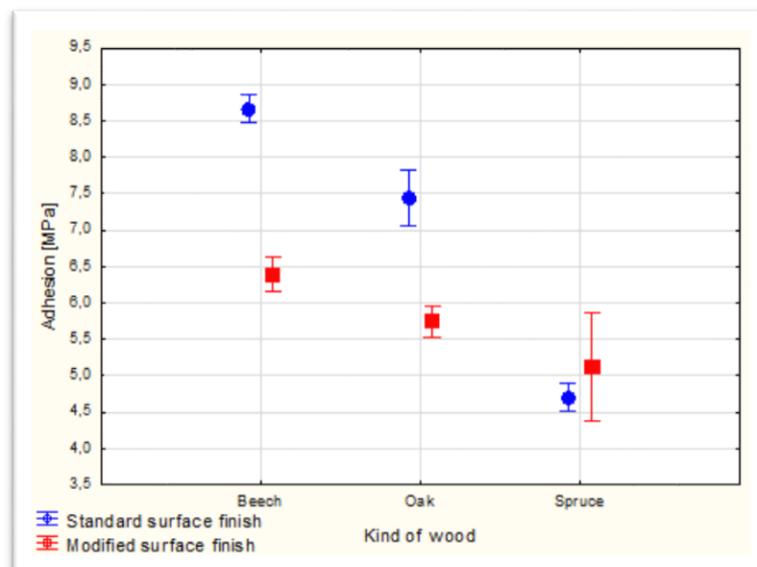


Fig. 4 Surface finishes' adhesion to individual tree species.

The fracture in the system "wood – coating film – metal dolly" was evaluated visually using a microscope at 4 × magnification. Fig. 6 a), c) and e) show the fracture on the standard surface finish after separation of the dolly. The figures show the surfaces of the metal dollies with the coating film separated from beech, oak, and spruce wood. On the surface of the dolly, areas of the coating film with separated wood fibres can be seen by a microscope. On beech wood and oak wood, separation of wood fibres was minimal and the separated wood fibres did not exceed 10 % of the area under the dolly. On the contrary, on spruce wood, separation of wood fibres was the most significant and, in some cases, reached more than 45 % of the area under the dolly.

Fig. 5 a) to f) show wood surfaces after separation of the dollies. The separated wood fibres are visible. On beech wood and oak wood, after separation of the dolly, the fracture occurred at the interface “wood – coatingfilm”. The fibres were separated to a small extent, up to 10 % only. Such type of fracture confirms that the measured value of adhesion is the adhesion of coating film to wood surface. On spruce wood, the separation of the surface layers of wood occurred in larger extent. This type of fracture can be considered a cohesive fracture in the surface layers of wood.

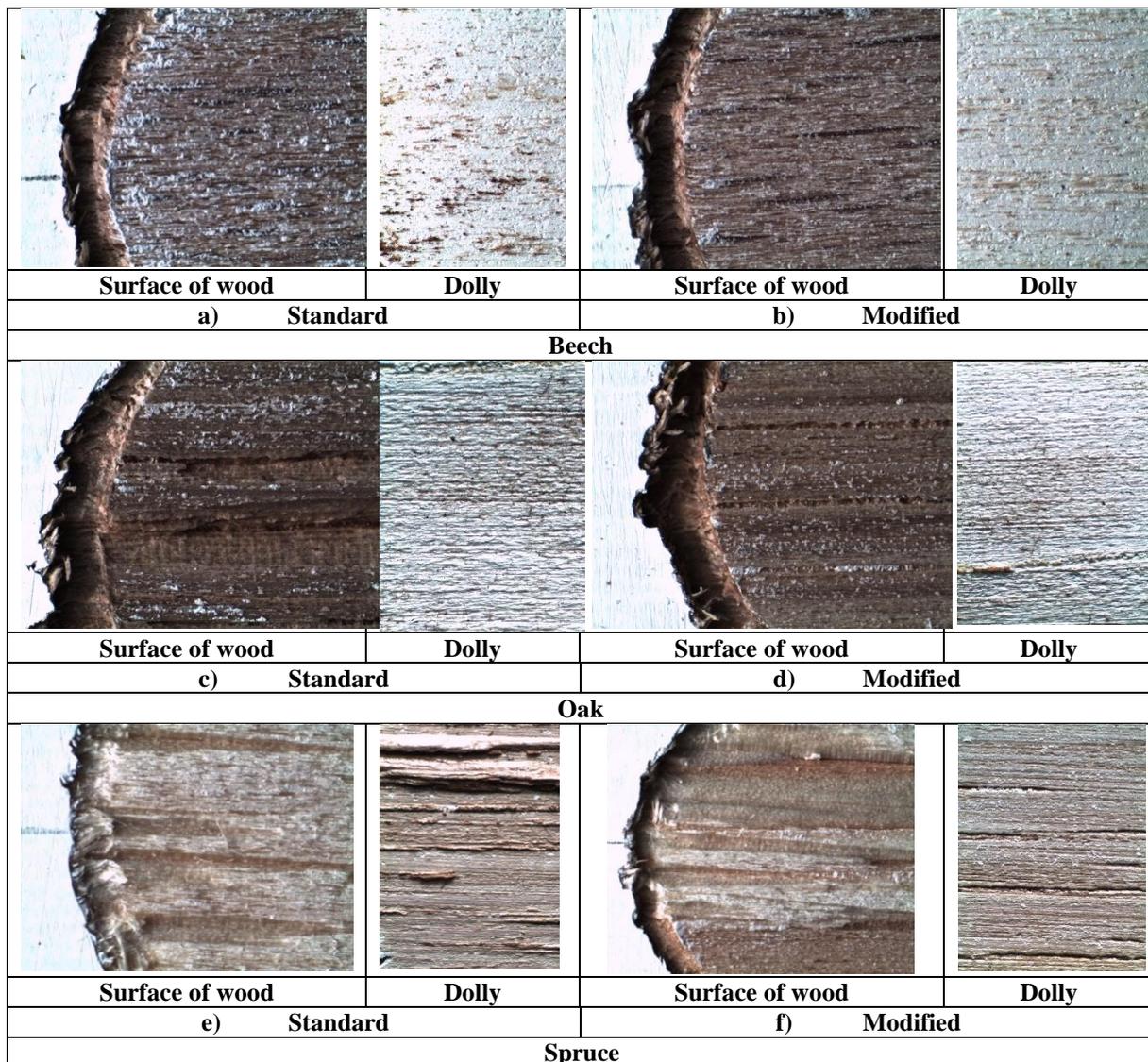


Fig. 5 Surface finishes after the Pull-off test; evaluated visually using a stereomicroscope (with magnification of 4 ×).

The adhesion of the coating film of the modified surface finish to beech wood and to oak wood was lower if compared with the standard surface finish. The modifying intermediate layer showed a negative effect on the adhesion of coating film to the substrate. Fig. 5 b) and d) show that after the dolly was separated, there was little separation of the surface wood fibres. The fracture can be described as an adhesion fracture. At these two tree species, the amount of separated wood fibres did not exceed 10 % of the area under the dolly.

Fig. 5 b) and d) show that after the dolly was separated almost no coating film remained on the wood surface. As with the standard surface finish, so the modified surface finish, the largest amount of fibres were separated from spruce wood (more than 45 % of the area under

the dolly; Fig. 5 f). Again, it can be stated that, on spruce wood, the fracture can be considered as a cohesive fracture in the surface layers of wood. Fig. 4 show that the adhesion of the standard surface finish and the modified surface finish to beech wood and oak wood was different. On spruce wood, the difference was not registered because the fracture occurred in the surface layers of wood; so the measured values were similar.

The high significant impact of surface finish on the adhesion was also confirmed by HAZIR and KOC (2019), SLABEJOVÁ and VIDHOLDOVÁ (2019b), MIKLEČIĆ, *et al.* (2017) and DELPECH and COUTINHO (2000).

According to the results by HAZIR and KOC (2019), a type of coating was an effective factor for the adhesion strength, surface coating hardness, layer thickness, and rapid deformation test.

Comparison of the two methods of the adhesion testing shows that the Cross-cut test has less informative value than the Pull-off test. According to the Cross-cut test, the modifying interlayer had no impact on the adhesion of coating film. From the results it is seen that the modifying layer reduced the adhesion of the coating film to hard tree species (oak, beech). Such a conclusion cannot be draw for the surface finish in the case of spruce wood because during the test, a failure occurred in the surface layers of wood, i.e. it was a cohesive fracture.

## CONCLUSION

From the results of properties of the tested surface finishes, the following conclusions can be drawn:

- The surface hardness of the coating film was not affected by the modifying layer. The interlayer did not increase the surface hardness of surface finish.
- The surface hardness of the surface finish was influenced by tree species.
- Impact resistance of the surface finish, at a maximum drop height of 400 mm, was not affected by the modifying layer. At a drop height of 200 mm, the modified surface finish was less resistant and more fragile in comparison with the standard surface finish.
- The resistance against scuffing of the surface finish was increased by the modifying layer.
- The modifying layer in the surface finish had impact on the adhesion of the coating film. If compared the two surface finishes, the standard surface finish reached a statistically significantly higher adhesion to beech wood and to oak wood than the modified surface finish (evaluated according to the Cross-cut test).

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## ACKNOWLEDGEMENTS

This work was supported by the Slovak Research and Development Agency under the contract No. APVV-16-0177.

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