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EFFECTS OF HEAT TREATMENT ON THE ADHESION STRENGTH, PENDULUM HARDNESS, SURFACE ROUGHNESS, COLOR AND GLOSSINESS OF *SCOTS PINE* LAMINATED PARQUET WITH TWO DIFFERENT TYPES OF UV VARNISH APPLICATION

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ABSTRACT

The objective of this study was to investigate the surface properties of a UV-system applied on laminated parquet made with untreated and heat treated wood (ThermoWood). In this study, wood specimens prepared from Scots pine (*Pinus sylvestris*) wood were heat treated according to ThermoWood method at 190°C for 2 hours and at 212°C for 1 and 2 hours adhesion strength, pendulum hardness, surface roughness, colour and glossiness were determined. The UV-system was applied in two different types according to manufacturer recommendations. Results show that lightness and glossiness decreases and red colour tone increases with heat treatment. Pendulum hardness increased initially, decreasing afterwards with the intensity of the heat treatment. Tests showed that adhesion generally decreased with heat treatment. No significant differences were found for the surface roughness although a slight decrease was observed.

Keywords: Heat modification, lightness, *Pinus sylvestris*, surface properties, thermowood process.

INTRODUCTION

Nowadays laminate flooring is preferred over solid wood due to its dimensional stability, lower cost and similar appearance. The flooring indoors is exposed to various conditions that can change color, brightness, surface roughness and hardness of laminate flooring surface. Testing the upper surface of laminate flooring is important to give us information for its utilization. One of the most important factors in laminated parquet production is the wood species selected for the outer layer. The most widely used wood species are American red oak, white oak, beech, pine, walnut, afrormosia, merbau, maple and iroko. Each species gives laminated parquet a characteristic color, gloss, surface roughness and hardness.

Heat treatment processes have been conquering a higher market share in the last years. One of the most successful processes is Thermowood®. The treatment is done with steam, with less than 3 to 5% oxygen without using pressure and with a minimum air speed of 10 m/s. The process begins with a rapid increase in temperature of the oven with heat and steam up to 100°C, followed by a gradual increase up to 130°C to near zero humidity. Then, heat treatment is carried at temperatures between 185°C and 230°C for 2 to 3 hours. This process is also one of the most studied processes in the last years (Korkut and Aytin 2015, Moliński et al. 2016) along with other methods as for instance those based in heat treatment and vacuum like VAP HolzSysteme® (Batista *et al.* 2006a,b)

Color is the most studied surface property that changes with heat treatment. It is a well-known fact that wood becomes darker and at the same time lightness decreases as reported by many authors with different wood species (Bekhta and Niemz 2003, Chen *et al*. 2012, Dubey *et al*. 2012; Esteves *et al.* 2007, Mitsui *et al*. 2001, Sundqvist 2002). In the last few years gloss changes due to heat treatment have also been studied (Aksoy *et al.* 2011, Bekhta *et al*. 2014, Karamanoglu and Akyildiz 2013).

Wettability of wood surface decreases with heat treatments as reported by several authors (Hakkou *et al*. 2005, Pecina and Paprzycki 1988, Pétrissans *et al*. 2003, Dos Santos and Goncalves 2016). In accordance to Hakkou *et al*. (2005), that studied the wettability change with heat treatment for poplar, pine, spruce, and beech, this change could be due to a modification of the conformational arrangement of wood biopolymers resulting from the loss of residual water or, more probably, from the plasticization of lignin. This decrease in the surface wettability slows down the absorption of glues and varnishes and consequently reduces adhesion, which means that normal finishes are usually unfit to heat-treated wood (Sernek *et al.* 2007). Nevertheless there are varnishes and glues that can be adapted for this type of wood.

Hardness is a very important property in flooring, especially in the outer layer, and the results reported before prove that hardness changes depends on wood species, conditions of treatment and even with the direction of the tests (Shi *et al*. 2007). For instance Poncsak *et al*. (2006) studied heat-treated birch and mentioned a slight hardness increase while Korkut *et al.*

(2008) concluded that janka-hardness decreased with heat-treatment for Scots pine wood. Boonstra *et al.*, (2007) reported that Brinell hardness parallel to the grain increased significantly (48%) and hardness perpendicular to the grain increased slightly (5%) for spruce.

Korkut and Guller, (2008) studied the effects of heat treatment on the physical properties and surface roughness of red-bud maple and concluded that surface roughness decreased with increasing temperature treatment and treatment times. (Korkut *et al*. 2013) reported the effect of heat treatment on surface properties of wild cherry. These authors concluded that glossiness and surface roughness decreased with heat treatment compared to those of control specimens. Similar results were reported for heat treated red river gum tree (*Eucalyptus camaldulensis*) (Unsal and Ayrilmis 2005), black pine (*Pinus nigra*) (Gunduz *et al*. 2008) and turkish Hazel (*Corylus colurna*) (Korkut *et al.* 2008a).

The paper reports on the changes in colour (ΔE, ΔL^*, Δa^* and Δb^*), glossiness (60°) (ISO 2813), surface roughness (R_a, R_y, R_z and R_q) (ISO 4287), surface adhesion resistance (MPa) (ASTM D 4541) and pendulum hardness (ANS/ISO 1522) of coated heat treated Scots pine laminated parquet produced in KPS company (Merkez/Duzce, Turkey).

MATERIALS AND METHODS

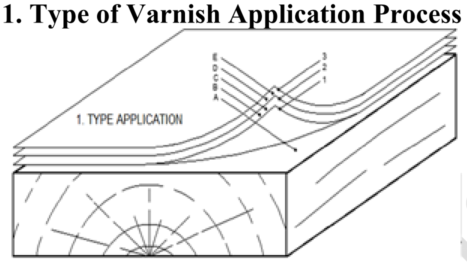
Heat Treatment Process

The heat treatment was performed according to ThermoWood® process at 190°C for 2 h, 212°C for 1 h and 2 h in a private commercial Novawood Factory in Gerede - Bolu, Turkey. Heat treated and untreated wood samples were conditioned to 12% equilibrium moisture content in a special room at 20 ± 2°C and 65 ± 5% relative humidity (ISO 554 1976).

Laminated Parquet Flooring Material

In this study, laminated parquet flooring made with Scots pine (*Pinus sylvestris* L.), produced by KPS Company was selected for the tests. The production methods of laminated parquet flooring were shown in Figure 1. Five samples were prepared for each test and 6 measurements (5 x 6 = A total of 30 measurements) were made in each sample. The tangential surface of samples measuring 100 mm by 100 mm by 20 mm were used for all measurements. Wood colorant paint was used on the experimental samples. All laminated parquet flooring materials samples were conditioned to 12% MC in a conditioning room at 20°C (± 2) and with 65% (± 5) RH (ISO 554 1976). The equilibrium MC of the samples was roughly 12 per cent after conditioning.

A43-0646 – UV sanding sealer is a type of varnish consisting of epoxy acrylic resin and ultraviolet ray curing sealers with solidity of (wt %) 95-97, and density of (20°C, g/cm^3) 1:15 to 1:20. N93-0910 nanolacke UV matt varnish is a type of varnish consisting of polyacrylic-based resin, nano-containing minerals, nanocomposites ultra violet curing (UV) varnish with solidity of (wt %) 95-100, and density of (20°C, g/cm^3) 1:09 to 1:15. Both of these varnishes are transparent and their application field are solid hardwood, chipboard and similar types of wooden materials.

<table>
<tr><td colspan="2">1. Type of Varnish Application Process
 </td></tr>
<tr><td>A. Sanding (3 cylinders) & Calibrating Machines</td><td>80-120-220 grit sandpaper</td></tr>
<tr><td>1. A43-0646-UV Sanding Sealer</td><td>50 g/m^2</td></tr>
<tr><td>B. UV lamp drying (mercury)</td><td>2x80 W</td></tr>
<tr><td>C. Sanding 2 cylinders</td><td>280-320 grit sandpaper (Pressure 1.5 bar)</td></tr>
<tr><td>2. N93-0910 Nanolacke UV Matt Varnish</td><td>7.5 g/m^2</td></tr>
<tr><td>D. UV lamp drying</td><td>2x80 W</td></tr>
<tr><td>3. N93-0910 Nanolacke UV Matt Varnish</td><td>7.5 g/m^2</td></tr>
<tr><td>E. UV lamp drying</td><td>400 W</td></tr>
<tr><td colspan="2">Polishing production line speed of 10 m/min.</td></tr>
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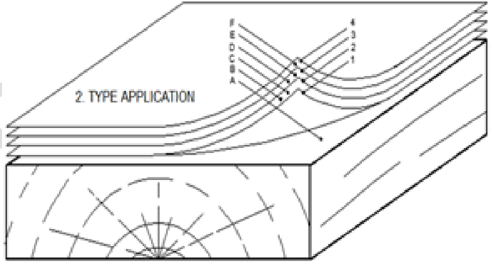 </td></tr>
<tr><td>A. Sanding (3 cylinders) & Calibrating Machines</td><td>80-120-220 grit sandpaper</td></tr>
<tr><td>1. A43-0646-UV Sanding Sealer</td><td>35 g/m^2</td></tr>
<tr><td>B. UV lamp drying (mercury)</td><td>2x80 W</td></tr>
<tr><td>2. A43-0646-UV Sanding Sealer</td><td>35 g/m^2</td></tr>
<tr><td>C. UV lamp drying</td><td>400 W</td></tr>
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<tr><td>F. UV lamp drying</td><td>400 W</td></tr>
<tr><td colspan="2">Polishing production line speed of 10 m/min.</td></tr>
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Fig. 1. Two different types of UV varnish application process.

Glossiness and Color Measurement

Glossiness measurements in coated wood with one and two layers were made in accordance to ISO 2813 (1994) in a Novo-Gloss Trio (Rhopoint Instruments Ltd., UK.). The measurements were made in perpendicular and parallel to the grain directions at an angle of 60^o (Figure 2.a). The color change of laminated parquet produced from heat treated and untreated Scots pine, coated with one and two layers of finishing were analysed by a The Datacolor 110 (Wavelength resolution 10 nm, measurement geometry $D/8^o$) with a D65 standard illuminant. Color parameters were measured using thirty replicates of each sample and an average value was reported. The CIELAB system characterized by three parameters, L^*, a^*, and b^* was used. The L^* axis represents the lightness, $+a^*$ is the red, minus a^* for green, $+b^*$ for yellow, minus b^* for blue, and L^* varies from 100 (white) to zero (black) (Zhang *et al*. 2009). Total color difference (ΔE^*) was calculated using Equation 1. (Figure 2.b).

$$\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2} \qquad (1)$$

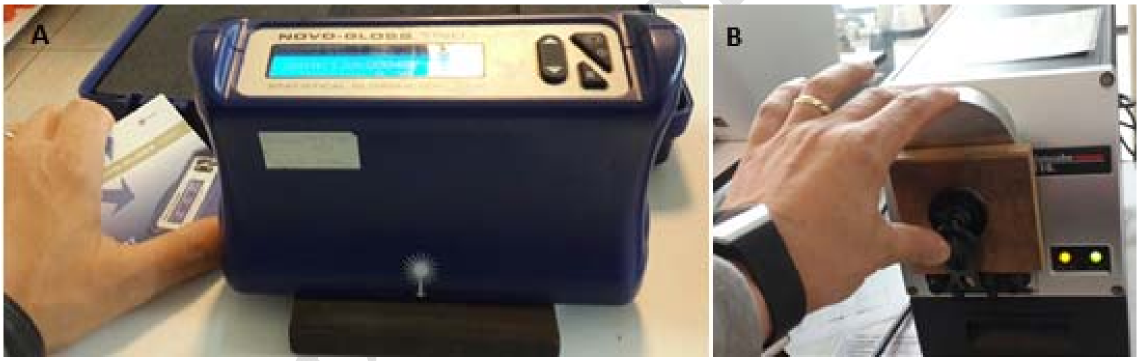

Fig. 2. a) Novo-gloss trio (60°) and b) The datacolor 110 spectrophotometer.

A total of 480 data were measured for glossiness tests (Heat treatment 4 x layer 2 x measuring direction 2 x N 30= 480 measurement). The 960 data were measured for color tests (Heat treatment 4 x layer 2 x color difference 4 x N 30 = 960 measurement).

Surface Roughness

Surface roughness of all the samples was measured using a Mitutoyo Surftest SJ-301 device. Surface roughness of the samples was measured by using a stylus-type profilometer in Figure 3 (Mitutoyo 2015). In this device; R_a, Ry, R_z and R_q test were measured according to ISO 4287 1997. Four roughness parameters were determined: mean arithmetic deviation of profile (R_a), mean peak to-valley height (R_z), root mean square roughness (R_q), and maximum roughness (R_y). These parameters were commonly used in previous studies to evaluate surface characteristics of wood and wood composites such as veneer (Stumbo 1963). Roughness values were measured with a sensitivity of 0.5μm. The length of scanning line (Lt) was 15mm and the cut-off was λ=2.5mm. Specification of roughness parameters is described by Hiziroglu (1996), Hiziroglu and Graham (1998).

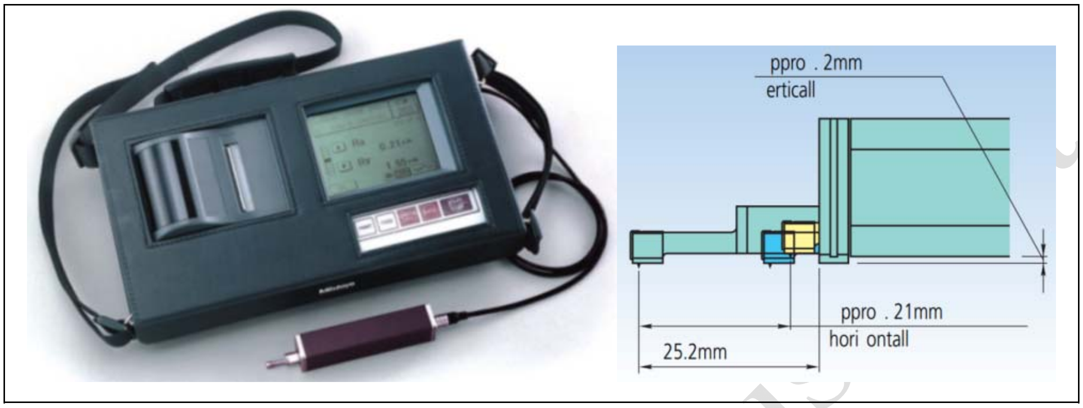

Fig. 3. Schematic description of the mitutoyo surftest SJ-301 (Mitutoyo, 2015).

A total of 960 data were measured for surface roughness tests (Heat treatment 4 x Layer 2 x Roughness values 4 x N 30 = 960 measurement).

Adhesion Strength Test

In the study, the adhesion strength was determined in accordance to ASTM D-4541 (1995) in 1 ton (10 kN) ALSA electromechanical universal testing machine (Figure 4). 404 plastic steel epoxy strong adhesive was used (Ayata, 2014). The steel test cylinders with Ø 20 mm were attached to the sample surfaces at room temperature (~ 20ºC) via the help of a cast system (Demirci *et al.* 2013). Glued samples were then fixed by means of tools. All sample specimens were expected to dry for 24 hours. Glue residues were removed with a cutter.

The adhesion strength (X) was calculated in terms of MPa using the equation below (Budakci, 2003).

$$X = 4F / \pi . d^2 \qquad (2)$$

Where;

F = the rupture force (Newton)

d = the diameter of the experiment cylinder (mm) (ASTM D-4541, 1995).

Fig. 4. Adhesion test machine and test sample.

A total of 480 data were measured for adhesion strength tests (Heat treatment 4 x Layer 2 x N 10 = 80 measurement).

Pendulum Hardness

Conditioned samples were subsequently subjected to the König pendulum hardness test to detect the hardness of the varnish coating according to ASTM D 4366-95 (1984). Test panels were placed on the panel table and a pendulum was gently placed on the panel surface. The pendulum was then deflected through 6° and released while simultaneously starting the oscillation counter. The number of oscillations for the amplitude to decrease from 6° to 3° was determined to be the König hardness. Thirty replications were conducted on separate specimens for each treatment group (Figure 5) (Cakicier *at al.* 2011a and Cakicier *at al.* 2011b). Devices in the sample surface (63 ± 3.3 in HRC hardness and 5 ± 0.0005 mm in diameter) determine the hardness layer according to the oscillating pendulum swing with two balls (Ayata, 2014). A total of 240 data were measured for pendulum hardness tests (Heat treatment 4 x Layer 2 x N 30 = 240 measurement).

Fig. 5. König pendulum hardness.

Statistical Analysis

In the experiments, statistical analysis was performed for a total of 2720 data (Glossiness 480 + color 960 + surface roughness 960 + adhesion strength 80 + pendulum hardness 240 = 2720 measurement). Statistical evaluations were analyzed by IBM SPSS 17 Software Package program.

RESULTS AND DISCUSSION

Color and Glossiness

Table 1, table 2, table 3 and table 4 presents colour parameters, glossiness, pendulum hardness, adhesion strength and surface roughness parameters for untreated and heat treated wood coated with one or two layers.

Table 1. Statistical data for color difference values in untreated and heat treated wood coated with one and two layers Scots pine.

Heat treatment	Layer Thickness	N	Total Color			Color Lightness		
			Mean	*HG*	Std. Dev.	Mean	*HG*	Std. Dev.
Control	1 coat	30	85,44	A	0,34	79,30	A	0,45
	2 coats	30	84,66	B	0,10	78,19	B	0,20
190°C - 2 hours	1 coat	30	61,25	E	1,32	48,56	E	1,43
	2 coats	30	62,97	D	0,79	49,76	D	0,84
212°C - 1 hour	1 coat	30	60,40	F	0,50	47,01	F	0,40
	2 coats	30	65,95	C	0,48	52,40	C	0,64
212°C - 2 hours	1 coat	30	54,77	G	0,14	41,59	G	0,20
	2 coats	30	52,14	H	0,35	39,63	H	0,29
Heat treatment	Layer Thickness	N	Red Color Tone			Yellow Color Tone		
			Mean	*HG*	Std. Dev.	Mean	*HG*	Std. Dev.
Control	1 coat	30	2,89	H	0,22	31,67	E	0,27
	2 coats	30	3,38	G	0,14	32,29	D	0,27
190°C - 2 hours	1 coat	30	12,72	E	0,48	35,08	C	0,52
	2 coats	30	12,94	D	0,28	36,35	B	0,39
212°C - 1 hour	1 coat	30	13,93	C	0,11	35,27	C	0,41
	2 coats	30	12,38	F	0,26	38,08	A	0,17
212°C - 2 hours	1 coat	30	14,73	A	0,04	32,44	D	0,21
	2 coats	30	14,60	B	0,09	30,57	F	0,58

HG: Homogeneity Group, Std. Dev.: Standard Deviation, Mean: Average, N: Number of Measurements

Table 2. Statistical data for glossiness values in untreated and heat treated wood coated with one and two layers Scots pine.

Heat treatment	Layer Thickness	N	Glossiness perpendicular			Glossiness parallel		
			Mean	*HG*	Std. Dev.	Mean	*HG*	Std. Dev.
Control	1 coat	30	17,81	B	0,34	24,66	A	0,50
	2 coats	30	18,34	A	0,46	25,14	A	0,30
190°C - 2 hours	1 coat	30	17,48	BC	1,31	22,66	B	1,21
	2 coats	30	17,23	C	0,79	22,71	B	2,83
212°C - 1 hour	1 coat	30	16,64	D	0,82	22,45	B	1,09
	2 coats	30	16,44	D	0,98	22,67	B	1,94
212°C - 2 hours	1 coat	30	16,25	D	1,13	21,77	BC	1,55
	2 coats	30	15,70	E	1,15	20,98	C	2,31

HG: Homogeneity Group, Std. Dev.: Standard Deviation, Mean: Average, N: Number of Measurements

In relation to color parameters, lightness decreased with the increase of the heat treatment despite the number of layers of the coating. For the less intense treatment (190ºC, 2 hours), lightness decreased approximately 38% in relation to initial lightness and for the most intense treatment (212ºC, 2 hours) the decrease was approximately 48%. Simultaneously, red color tone increased while yellow color tone did not show any consistent change. Similar results were reported before by several authors with uncoated heat treated wood (Bekhta and Niemz, 2003; Dubey *et al.*, 2012; Esteves *et al.,* 2007; Mitsui *et al.*, 2001). Glossiness, measured parallel and perpendicular to the grain decreased with the intensity of the heat treatment which is in accordance to the reported by other authors (Aksoy *et al.,* 2011;

Karamanoglu and Akyildiz, 2013). There seems to be no influence on glossiness due to the coating.

Pendulum Hardness, Adhesion and Surface Roughness

Pendulum hardness increased initially, decreasing afterwards with the intensity of the heat treatment. Coating increased the hardness of the surface since pendulum hardness is higher for the samples with two coats. These results are in accordance to results presented before that stated that hardness changes depends on wood species, conditions of treatment and even with the direction of the tests (Shi *et al*., 2007). Tests showed that adhesion strength generally decreased with heat treatment, nevertheless the decrease was much smaller when two layers coating is used.

No significant differences were found for the surface roughness. Although there is no difference between R_a parameter of treated and untreated wood, this parameter is higher for wood with two layers coating. Maximum roughness (R_y) increases slightly with heat treatment and with the number of layers in the coating. This parameter represents the distance between peak and valley points of the profile and can be used as an indicator of the maximum defect height within the assessed profile (Mummery 1993). Similar variation was found for R_z and somewhat for R_q parameters. Roughness measurements by the stylus method with uncoated untreated and heat treated Turkish river red gum showed that surface roughness values decreased with increasing treatment temperature and treatment times (Unsal and Ayrilmis, 2005). Similar results were reported by (Gunduz *et al*., 2008) with Camiyani Black Pine (*Pinus nigra* Arn. subsp. pallasiana var. pallasiana), with Turkish Hazel (*Corylus colurna* L.) (Korkut *et al.,* 2008a) and by (Korkut and Guller, 2008) with red-bud maple (*Acer trautvetteri* Medw.).

Table 3. Statistical data for surface hardness and adhesion values in untreated and heat treated wood coated with one and two layers Scots pine.

Heat treatment	Layer Thickness	N	Pendulum hardness			Adhesion test (MPa)			
			Mean	*HG*	Std. Dev.	N	Mean	*HG*	Std. Dev.
Control	1 coat	30	43,20	D	7,237	10	1,487	AB	0,36
	2 coats	30	55,17	C	11,271	10	1,606	AB	0,61
190°C - 2 hours	1 coat	30	51,83	C	9,337	10	1,107	CD	0,23
	2 coats	30	69,63	B	7,872	10	1,735	A	0,35
212°C - 1 hour	1 coat	30	44,63	D	6,184	10	0,967	D	0,18
	2 coats	30	75,80	A	8,794	10	1,365	BC	0,29
212°C - 2 hours	1 coat	30	41,80	D	6,499	10	0,931	D	0,20
	2 coats	30	55,07	C	6,868	10	1,562	AB	0,20

HG: Homogeneity Group, Std. Dev.: Standard Deviation, Mean: Average, N: Number of Measurements

Table 4. Statistical data for surface roughness values in untreated and heat treated wood coated with one and two layers Scots pine.

Heat treatment	Layer Thickness	N	Surface roughness R_a			Surface roughness R_y		
			Mean	*HG*	Std. Dev.	Mean	*HG*	Std. Dev.
Control	1 coat	30	1,80	F	0,11	14,65	D	1,47
	2 coats	30	2,08	CD	0,16	16,15	C	2,27
190°C - 2 hours	1 coat	30	2,10	C	0,12	17,43	C	1,97
	2 coats	30	2,36	B	0,19	19,23	B	3,58
212°C - 1 hour	1 coat	30	2,00	DE	0,14	17,22	C	1,80
	2 coats	30	2,48	A	0,35	21,04	A	3,18
212°C - 2 hours	1 coat	30	1,97	E	0,12	16,73	C	1,90
	2 coats	30	2,17	C	0,14	16,88	C	2,04

Heat treatment	Layer Thickness	N	Surface roughness R_z			Surface roughness R_q		
			Mean	*HG*	Std. Dev.	Mean	*HG*	Std. Dev.
Control	1 coat	30	12,18	F	0,71	2,29	F	0,14
	2 coats	30	13,59	DE	1,44	2,63	CDE	0,23
190°C - 2 hours	1 coat	30	14,28	C	1,06	2,67	CD	0,19
	2 coats	30	14,97	B	1,12	3,00	B	0,32
212°C - 1 hour	1 coat	30	14,23	CD	1,64	2,60	DE	0,20
	2 coats	30	15,97	A	1,61	3,23	A	0,47
212°C - 2 hours	1 coat	30	13,42	E	1,00	2,53	E	0,19
	2 coats	30	14,02	CDE	0,94	2,75	C	0,16

HG: Homogeneity Group, Std. Dev.: Standard Deviation, Mean: Average, N: Number of Measurements

CONCLUSIONS

Overall, lightness and glossiness decreased and red colour tone increased with heat treatment and no significant differences were found between one or two layers coating. In relation to pendulum hardness there was an increase initially, decreasing afterwards with the intensity of the heat treatment. This decrease was smaller if two layers coatings are used. Although adhesion strength generally decreased with heat treatment the use of a double layer coating improved the adhesion. No significant differences were found for the surface roughness although a slight decrease was observed.

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