Effect of Thermal Modification on the Surface Quality of a Coating Applied to Wood *via* the Electrostatic Spray Deposition Technique

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The surface properties of thermally modified ash wood with a powder coating were investigated, and the results were compared to the unmodified wood. The wood specimens were sanded with 80 grit sandpaper and then pre-heated at 80 °C for 5 min in an infrared oven. The surface of the unmodified and the modified wood specimens were coated with an epoxy/polyester (1 to 1 ratio) hybrid coating using an electrostatic corona spray gun at the pilot plant established in the laboratory. The coatings on the wood specimens were cured at different curing conditions in an infrared oven, i.e., 120 °C/15 min, 140 °C/10 min, and 160 °C/10 min. The results showed that the thermal modification (TM) of the wood caused a slight decrease in the mechanical performance of the surface system (wood substrate and coating film). For example, the scratch and abrasion resistance of the unmodified specimens at the curing temperature of 120 °C were 3.33 N and 135 revolutions but were 3.12 N and 120 revolutions after the TM. However, the average surface roughness (1.26 mu) and contact angle (60.8°) of the distilled water on the cured coatings on the modified wood were lower than those on the unmodified wood (1.86 mu and 80.8°, respectively).

Keywords: Electrostatic powder coating; Thermally modified wood; Surface quality; Ash wood

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INTRODUCTION

The thermal modification (TM) of wood for outdoor applications has a long history in the wood industry, due to its unique properties of thermally modified wood, *e.g.*, improved dimensional stability and biological durability, compared to unmodified wood. Although there are some effective wood modification techniques, *e.g.*, acetylation and furfurylation, these processes use chemicals, which are harmful to the environment after the disposal of wood. Among the wood modification techniques, the most accepted one is the TM, which is an environmentally friendly technique that does not use any toxic chemicals. Despite the positive features of the TM, it affects the surface properties of the wood, which generally results in surface inactivation, reduced surface roughness, enhanced hydrophobic behavior, *i.e.*, a decrease in the amount of free hydroxyl groups, and a darkened surface (Hill *et al.* 2021). Some chemical reactions occur in the wood during the thermal treatment, *e.g.*, hydrolysis and oxidation, which cause the reduction of free hydroxyl groups and surface inactivation (Hakkou *et al.* 2005; Petric *et al.* 2009; Miklečić and Jirouš-Rajković 2016). Previous studies reported that the surface inactivation caused by the TM influences the wettability, which plays a role in the bonding mechanism between wood and certain coatings (Jarusombuti *et al.* 2010; Hubbe *et al.* 2015; Zhang and Yu 2015). Coating systems need to spread and penetrate the surface of the wood for good bonding to occur.

Although electrostatic powder coating technology has been used in the metal industry in the USA and Europe since the early 1960s, it is still relatively new for the wood industry. In the early 2000, electrostatic powder coating for non-conductive substrates, especially medium density fibreboard (MDF) panels, was introduced to the furniture industry in order to benefit from the advantages of this technology. The use of powder coatings in the panel-type furniture industry has considerably increased over the last decade, due to its five considerable advantages, *i.e.*, its efficiency in powder use, excellent finish, economic benefits, energy savings, and being an environmentally friendly material. Today, two-thirds of the coatings used in the wood and wood-based panels industries are still based on solvent-based paints, because of their considerable advantages, e.g., low cost, easy supply, highly durable finish, and high gloss finish, when compared to the waterbased paints and powder coatings. Solvent-based paints are potentially hazardous to human and environment health because they contain some harmful volatile organic compounds (VOCs) such as formaldehyde, toluene, ethylbenzene, and xylene. An increasing awareness of environmental issues has increased the use of environmentally friendly and cost-effective coating systems. At this point, powder coatings present a good opportunity for the furniture industry. The most used thermosetting powders are epoxy, polyester, epoxy/polyester hybrids, polyurethane, acrylic, and hybrid compositions. The powder coatings are applied to the wood material surface using electrostatic forces. Then, the thermosetting powder coating melts and flows out on the surface of the substrate when the heat is increased in a curing oven. After a while, the coating is cured to the surface, which results in a network structure of cross-linked polymers with a higher molecular weight (Akkuş et al. 2021). The utilization of thermally modified wood for indoor or outdoor applications has progressively increased due to its above-mentioned advantages. Previous studies have reported that the surface of the wood after the TM shows some changes when compared to unmodified wood (Hakkou et al. 2005; Zhang et al. 2015; Miklečić and Jirouš-Rajković 2016; Hill et al. 2021). These changes include a decreased wettability and an inactivation of the surface. The presence of hydroxyl groups on the wood surface are affected by the thermal treatment. The hydroxyl groups react with the functional groups of the adhesives such as urea-formaldehyde, melamine formaldehyde. When the wood is exposed to elevated temperatures, the number of the hydroxyl groups decreases, which negatively affect the bonding performance between the wood and resin. These properties should be also considered before the overlay coating. The curing mechanism of epoxy/polyester thermosets involves reaction of the acid functionality with epoxy followed by esterification of the epoxy hydroxyl groups with the acids. Compounds such as amines and phosphonium salts catalyze these reactions (Petriccione 2011).

Wood-based panel producers, furniture producers, and other users are still new to this technology, and they need to be informed about the details of this process and given key precautions to ensure a successful finishing with powder coatings. The curing step is critical for the powder coatings. When the powder coating is applied on the surface of MDF surface at low temperatures, it is essential to understand the curing behavior of the powder coating. Specifically, the curing behavior of the powder coatings is primarily affected by the curing conditions, *e.g.*, air flow, the temperature, and duration. The present study was aimed at understanding the effect of the thermal treatment of wood on the curing behavior and surface quality of the powder coatings at elevated temperatures in the curing oven. Although the coating properties of thermally modified wood coated with solvent or waterbased paints have been investigated in previous studies by Miklečić *et al.* (2011), Jirouš-Rajković and Miklečić (2019), Nejad *et al.* (2019), and Slabejová *et al.* (2019), these studies did not explore the influence of the TM on various properties of coatings applied *via* the electrostatic spray deposition technique.

EXPERIMENTAL

Wood Specimens

Thermally modified ash wood specimens (*Fraxinus excelsa* Thunb.) with 320 mm x 135 mm x 20 mm dimensions were obtained from the Novawood Company (Duzce city, Turkey) (Fig. 1a). The wood specimens were thermally modified at the Novawood factory (Duzce city, Turkey) based on the Thermowood[®] process under Finnish patent (Fig. 1b). The average moisture content of the thermally treated wood specimens was 7% while the average moisture content of the unmodified wood specimens was 12%. The thickness of the specimens was reduced to 10 mm using a thickness planer machine so that the specimens were easy to place in the abrasion and scratch test equipment.



Fig. 1. The (a) unmodified ash wood specimens; and (b) thermally modified ash wood specimens

Powder Coatings

An epoxy/polyester hybrid (at a 1:1 ratio) powder coating was used in the experiments (Table 1). It was supplied by a commercial producer of electrostatic powder coatings (Mikroton Chemical Company, Istanbul, Turkey). The code of the coatings was RAL 9016, which is used for interior applications.

Application of Powder Coatings to the Surface of Wood

The top and bottom sides of the unmodified and modified wood specimens were sanded with 80 grit sandpaper to achieve a smooth surface before the powder coating application. The wood specimens were placed into the infrared oven at 80 °C for 5 min. One of the primary challenges of wood and wood-based panels in terms of coating applications is that the electrostatic coating application needs an electrically conductive substrate.

Test type	Unit	Standard Number	Value	
Physical tests		Standard Number	value	
Gloss	(60°)	EN ISO 2813 (2014)	5 to 95	
Impact resistance	kg∙cm	ISO 6272 (2011)	80 to 100	
Flexibility	mm	EN ISO 1520 (2006)	7 to 8	
Buchholz indentation test	-	EN ISO 2815 (2003)	90 to 100	
Cross-cut	Gt	EN ISO 2409 (2020)	0	
Density	g/cm ³	ISO 8130-3 (1992)	1.30 to 1.70	
Flow property	-	ISO 8130-5 (1992)	120 to 175	
Chemical tests				
Corrosion test (salt test)	Hour	ISO 7253 (1996)	1000 +	

Table 1. Physical, Mechanical, and Chemical Properties of the Epoxy/Polyester (1:1) Hybrid Powder Coating (Code: RAL 9016)

Following the pre-treatment, the wood specimens were immediately transported to the hangers of the powder coating application system (Fig. 2). The pneumatic control section of the corona type gun is shown in Fig. 3. Single layer powder coating (epoxy/polyester hybrid at a 1:1 ratio) was uniformly applied to the surface of the wood specimens via the corona spray gun for 2 min (Fig. 4). The electrostatic powder coating was applied using a pilot scale coating system manufactured by Boysan Electrostatic and Wet Coating Application Systems (Istanbul city, Turkey). The specimens were placed into the infrared oven in order to cure the powder coatings. To understand the effects of the curing conditions, *i.e.*, the temperature and duration, three different curing temperatures were used during the experiments. The curing conditions of the specimens are shown in Table 2. The coated specimens were conditioned to a constant weight at a temperature of 20 °C and a relative humidity of air 65%. The dry powders were made electrostatic by charging the powder particles at the tip of the corona spray gun. The electrical current and voltage applied by the corona spray gun were 80 µA and 50 kV, respectively. A total of 18 specimens, 3 for each treatment, were used in the experiments (Table 2). The mean density of the unmodified and modified specimens ranged from 0.52 to 0.57 g/cm³.

Determination of the Coating Thickness

The coating thickness of the wood specimens was measured with a PosiTector 200 Coating Thickness Gage (DeFelsko Inspection Instruments, Ogdensburg, NY), which measured the coating thickness between 20 and 300 μ m (Fig. 4).

Determination of the Scratch and Abrasion Resistance

The abrasion and scratch resistance tests were carried out according to the EN 438-2 EN 438-2:2016+A1 (2018) standard (Fig. 4). Both tests were carried out on the fully cured coatings on the unmodified and modified wood specimens. To carry out the scratch and abrasion resistance tests, a 6 mm diameter screw hole was opened on the diagonal center of the specimens, which had dimensions of 100 mm x 100 mm x 10 mm. Then, the specimen was fixed to the rotating table. The scratch resistance of the coatings was determined with a universal motorized scratch tester (Taber 551 Shear/Scratch tester, Taber Industries, North Tonawanda, NY). Three specimens were used for each treatment given in Table 2.

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Fig. 2. Pre-heating of the wood specimen in the infrared oven



Fig. 3. Pneumatic control section of the corona type electrostatic powder coating gun



Fig. 4. Application of the powder coatings on the wood specimens using the corona spray gun

The test specimens were firmly fixed through their central holes on the rotating table of a Taber® rotary abraser (Taber 5131 Abraser, Taber Industries, North Tonawanda, NY). The top surface of the coated specimens was exposed to two rotating abrasive discs coated with S-42 sandpaper and additional weights (500 g for each disc). The specimens were then subjected to rotation. The total number of cycles required to wear out three-fourths of the coated specimens was recorded as the abrasion resistance. Three specimens were used for each treatment given in Table 2.



Fig. 4. The (a) measurement of the coating thickness; (b) shear/scratch resistance test; and (c) abrasion resistance test

Determination of Surface Roughness and Wettability

The roughness of the 100 mm x 100 mm x 10 mm coated specimens was determined *via* a stylus roughness tester (Mitutoyo Surftest® SJ-301, Mitutoyo Europe GmbH, Neuss, Germany). The surface roughness measurement was carried out according to EN ISO standard 4287:1997/AMD 1 (2009). The average roughness (R_a), maximum height of the measured profile (R_z), and the root mean square deviation of the measured profile (R_q) were taken. Ten surface roughness measurements were recorded for each type of wood. Five 100 mm x 100 mm specimens were used for each wood treatment type. In addition, 10 total measurements for each treatment were taken (2 measurements for each specimen). The measurement points on the specimens were selected to represent all the surface of the wood specimen. The tracing speed of the stylus pin was 5 mm/s and the total tracing length was 12.5 mm.

The wettability behavior of the coated specimens was determined using a contact angle instrument with image analysis software (KSV-101, KSV Instruments Ltd., Helsinki, Finland). The digital images of a 5 μ L distilled water droplet were recorded with a video camera at 1 s intervals up to 5 s. The contact angles recorded at 5 s was reported. A total of 10 measurements were recorded for each type of treatment given in Table 2, two measurements for each specimen.

RESULTS AND DISCUSSION

The abrasion and scratch resistance of the specimens are shown in Table 2. The unmodified specimens showed slightly higher abrasion and scratch resistance at all the curing temperatures. For example, the scratch and abrasion resistance of the coatings on the non-modified specimens at a curing temperature of 120 °C were found to be 3.33 N and 135 revolutions, respectively, while they were found to be 3.12 N and 120 revolutions

for the modified wood specimens. Similar results were seen at curing temperatures of 140 °C and 160 °C. The dry film thickness of the coatings on the unmodified wood were higher than the dry film thickness of the coatings on the thermally modified wood at all curing temperatures. A higher coating thickness on the unmodified wood may be one of the reasons why its mechanical performance was better than the mechanical performance of the surface system (wood substrate and coating film) on the thermally modified wood. The results of the coating thickness measurements showed that the amount the coating penetrated thermally modified wood was greater than the amount the coating penetrated the unmodified wood. This can be explained by lower surface roughness of the thermally modified wood (Jirouš-Rajković and Josip Miklečić 2019). Ayrilmis and Winandy (2009) reported that the TM of the wood reduced the wettability and adhesive bond strength of wood. When wood is exposed to thermal treatment at a temperature of approximately 200 °C, its surface may experience inactivation, which results in a decrease in its wetting capability. The wettability affects the penetration of adhesives and coatings into the surface of wood, which is needed for the good mechanical interlocking. Particularly, hemicelluloses are degraded at high temperature thermal treatments, *i.e.*, approximately 200 °C, which explains the lower mechanical interlocking of the coatings on the modified wood when the coatings are cured (Hakkou et al. 2005; Hill et al. 2021).

The presence of hydroxyl groups on the wood surface significantly affects the bond with the OH groups of the epoxy. The decrease in the amount of the hydroxyl groups of the modified wood reduces the hydroxyl functionality of the epoxy resin. This negatively affects the crosslink possibility through the reaction between hydroxyl functionality and an epoxy ring (Petriccione 2011). It was thought to be a major reason for lower abrasion resistance and scratch resistance of the coatings on the modified wood.

Specimen Type	Infrared Oven	Conditions	Dry Coating	Abrasion Resistance of Coating (Rev.)	Scratch Resistance of Coating (N)		
	Curing temperature (°C)	Curing time (min)	Thickness (mu)				
Coating on	120	15	71.4 (10.8)	135 (26)	3.33 (0.28)		
the unmodified wood	140	10	72.6 (15.7)	187 (0)	3.45 (0.35)		
	160	10	85.0 (17.3)	195 (77)	3.53 (0.40)		
Coating on	120	15	56.4 (8.0)	120 (26)	3.12 (0.20)		
the modified wood	140	10	67.0 (16.4)	173 (65)	3.14 (0.24)		
	160	10	61.4 (5.8)	188 (19)	3.25 (0.26)		
Note: * The values in the parentheses are the standard deviations							

Table 2. Film Thickness, Abrasion, and Scratch Resistance of the Coatings on

 the Unmodified and Modified Wood Specimens

Note: * The values in the parentheses are the standard deviations

The thickness of the coating film on the unmodified and modified wood surfaces are given in Table 2. It was clearly shown that the TM of the wood caused a lower film thickness at all curing temperatures. This may be explained by the lower surface roughness of the thermally modified wood, as shown in Table 3. Furthermore, the equilibrium moisture content of the thermally modified wood in a standard atmosphere considerably decreases (Prieto and Kiene 2018). The lower moisture content (7%) of the modified wood may be another reason for the lower abrasion and scratch resistance of the coating, because the dielectric constant of wood decreases with decreasing moisture content, particularly in

the face layers. The decrease in the dielectric constant negatively affects the powder deposition onto the wood substrate, which results in the poor adhesion of the powder coating to the substrate. The sorption and diffusion properties of heat-modified wood decrease after heat exposure (Kymäläinen *et al.* 2018). The moisture content and temperature of wood are two important factors affecting its electrical conductivity, which affect the retention of the powder on the wood surface, and the resulting coating film thickness (Schmidt *et al.* 2013).

When the curing temperature was increased from 120 to 160 °C, the mechanical performance of the coating on the unmodified and modified wood specimens improved. The increase in the curing temperature decreased the improved surface smoothness and wettability of the wood specimens. This result revealed that the increased curing temperature provided the coating film better resistance to the abrasions and scratches. In addition, these results were consistent with previous studies (Lee *et al.* 1999; Andrei *et al.* 2000; Barletta *et al.* 2007). In a previous study, Akkuş *et al.* (2019) reported that the scratch resistance of particleboard and MDF coated with an epoxy/polyester hybrid powder coating temperature did not have a major effect on the visual appearance of the specimen, but the adhesion of the coating and the abrasion resistance were strongly related to the curing conditions in the IR oven. This was because a fully cured thermosetting coating showed its full potential regarding its abrasion and scratch resistance, which allowed greater resistance to the action of the scratch indenter.

The contact angle values of the water measured at 5 s are given in Table 3. The wettability of the cured coatings on the thermally modified wood specimens was found to be higher than the wettability of the coatings on the unmodified wood specimens. The difference in the coating wettability of the modified and unmodified specimens decreased as the curing temperature was increased from 120 to 160 °C (25% to 4%). The highest contact angle (with a value of 80.8°) was found from the coating on the unmodified specimen. The contact angle values of all the coatings on the modified and unmodified wood specimens were lower than 90°, which denoted that the coating surface was hydrophilic. The increase in the curing temperature decreased the surface roughness and improved the wettability of the coatings. This may be explained by the cross-linking mechanism of the coating, which corresponds to an increased curing temperature.

The coatings on the modified wood specimens showed a lower surface roughness than the coatings on the unmodified wood at all curing temperatures. For example, the average roughness of the coatings on the modified wood at a curing temperature of $120 \,^{\circ}C$ was 1.28 µm while it was found to be 1.95 µm for the unmodified wood coatings. The surface roughness of the coatings improved as the curing temperature increased. The decrease in the surface roughness of the coatings cured at elevated temperatures was primarily attributed to the viscosity drop of the coatings due to the increased temperature during the curing of the coatings. Ayrilmis (2020) investigated the surface roughness of epoxy/polyester hybrid powder coatings on oriented strandboard (OSB). A decrease in the surface roughness of the coatings occurred when the curing temperature was increased from 120 to 160 °C in the infrared oven.

Table 3. Surface Roughness and Wettability of the Coatings on the Unmodified and Modified Wood Specimens

Specimen Type	Infrared Oven Conditions		Surface Roughness Parameters (mu)			Contact Angle of Distilled Water (°)	
	Curing temperature (°C)	Curing time (min)	Ra	Ry	Rz	Duration (5 s)	
Coating on the unmodified wood	120	15	1.95 (0.4)	11.69 (2.7)	8.38 (2.8)	80.8 (6.3)	
	140	10	2.64 (0.7)	14.88 (4.5)	10.25 (3.5)	75.9 (6.3)	
	160	10	1.85 (1.2)	7.96 (5.3)	6.25 (4.1)	60.5 (14.4)	
Coating on the modified wood	120	15	1.28 (0.5)	8.07 (4.1)	5.42 (3.0)	69.1 (10.8)	
	140	10	1.86 (0.8)	12.4 (4.6)	8.33 (4.0)	63.3 (5.4)	
	160	10	1.60 (0.8)	6.53 (5.9)	4.64 (4.6)	58.0 (11.1)	
Note: *The values in the parentheses are the standard deviations							

CONCLUSIONS

- 1. The scratch and abrasion resistance of the coatings on the thermally modified ash wood were slightly lower than the scratch and abrasion resistance of the coating on the unmodified wood. The dry film thickness of the coatings on the unmodified wood was higher than the dry film thickness of the coatings on the modified wood at all curing temperatures. This may be one of the reasons for the lower mechanical performance of the coatings on the modified wood.
- 2. The thermal modification (TM) reduced the number of the hydroxyl groups of the modified wood, which adversely affects the crosslink possibility between the hydroxyl groups of the modified wood and epoxy ring (Petriccione 2011). It was thought to be a major reason for the lower abrasion resistance and scratch resistance of the coatings on the modified wood.
- 3. The coatings on the modified wood specimens had a lower surface roughness and water contact angle values than the coatings on the unmodified wood specimens at all curing temperature.
- 4. Increasing the curing temperature improved the abrasion resistance and scratch resistance of the powder coatings on both the unmodified and modified wood specimens.
- 5. The curing temperature of the powder coating can provide useful indication to powder coaters to obtain better surface quality.

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