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RESEARCH ARTICLES

EFFECT OF ARTIFICIAL WEATHERING AND TEMPERATURE CYCLING ON THE ADHESION STRENGTH OF WATERBORNE ACRYLATE COATING SYSTEMS USED FOR WOODEN WINDOWS

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ABSTRACT

The adhesion of coatings to wood is important for their long-term performance. In this study, the adhesion strength of water-based acrylate coatings used for wooden windows after exposure to artificial weathering (AW) and temperature cycling (TC) was investigated. The analysis of the adhesion quality of coatings was performed via a pull-off test and failure characteristics. The 3-layered and 4-layered white and brown acrylate dispersions from six different producers were compared and the effect of coating thickness on adhesion strength was investigated. The adhesion strength values proved to be very variable. After AW, the adhesion strength and its variability increased for all the samples. TC had no statistically significant effect on the adhesion strength values. White coating systems were initially characterized by lower adhesion strength, but after AW and TC, they reached higher adhesion strength values than brown ones. The overall highest adhesion after AW and TC was recorded for the coatings based on alkyd-acrylate hybrid basis (Producer 3), while the lowest adhesion variability after AW was measured for one type of tested acrylate coating (Producer 4). The effect of different layering on adhesion strength was not demonstrated in this study.

KEYWORDS

acrylate coatings, adhesion strength, artificial weathering, coating thickness, pull-off test, temperature cycling

INTRODUCTION

Wood is a widely preferred material in many structural and decoration areas, both for interior and exterior applications (Rijckaert et al. 2001). However, wood components are affected when wood is exposed to solar radiation and other factors such as water, moisture or temperature changes (Feist 1990). This can result in unfavourable physical, mechanical, chemical or biological changes in wood (Feist 1990; Pandey 2005; Nzokou et al. 2011). Finishing is still one

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of the most often used methods to enhance the service life and appearance of wood in exterior and interior applications. The coatings themselves are also subjected to decomposition caused especially by UV-light radiation, water and heat stress and their service life on the exterior is dependent on the polymer base (Gobakken and Lebow 2010), used pigmentation (de Windt et al. 2014) and UV-stabilizing agents (Forsthuber et al. 2013). Wood coating systems constantly evolve (Evans et al. 2015). The content of toxic compounds is reduced (Rijckaert et al. 2001; Stirling et al. 2011) and emphasis has recently been placed on the use of waterborne systems (Tesařová 2010; Moya et al. 2016). Wood stain coating systems (EN 927-2 2014), specifically the multilayer coating systems based on acrylate, alkyd, polyurethane or hybrid compositions (Evans et al. 2015; Gaylarde et al. 2011; Grüll et al. 2014) are most commonly used for the protection of exterior joinery products such as windows. Longer service life can be achieved by using the pigmented types (de Windt et al. 2014). Wood has a long tradition as a window joinery material in Europe and, until the end of the 1980s, was widely considered to be the best joinery material available (Ahola et al. 1999). Window coatings prevent the rapid moisture changes of wood and associated dimensional changes, stresses and cracking (de Meijer 2001). In addition, they protect wood against photochemical degradation, increase the durability against photochemical biological attack (bacteria, moulds, fungi etc.) (de Meijer 2001), enhance the appearance and provide easier cleaning solutions.

The aim of coating testing is to predict whether a given coating is or is not durable enough to satisfy the service life demand (Brunner et al. 2005). For the long-term performance of coatings, the sufficient adhesion strength of coatings to wood is very important (Bardage and Bjurman 1998), as it affects both the quality and durability of the coating. Adhesion strength can be expressed as a quality of finish, which depends on the type of finish and its interaction with the underlying material (Özdemir and Hiziroglu 2009; Ugulino and Hernández 2017). Numerous evaluation methods have been developed to investigate and improve finishing processes. Previous studies have shown that adhesion strength depends on several parameters such as coating thickness (Baek et al. 2009), surface roughness (de Moura and Hernández 2006; Cool and Hernández 2012; Ugulino and Hernández 2017) wood characteristics (Özdemir et al. 2015c) or wood moisture content (Landry and Blanchet 2012). Adhesion strength can change as a function of time. As for long-term behaviour, it is in particular the contact zone between the wood structure and coating that is of major importance (Rijckaert et al. 2001).

The material ageing on the exterior can be simulated by artificial weathering and temperature cycling (Kesik et al. 2015; Oberhofnerová et al. 2018). These test methods provide relevant results on the quality of coating systems in time in a considerably shorter period than natural weathering tests (Demirci et al. 2013; Özdemir et al. 2015a; Özdemir et al. 2015b; Söğütlü et al. 2016; Ugulino and Hernández 2017). Adhesion strength of coatings can be evaluated using several testing methods—the axial pull-off test with a dolly glued on the coating according to ISO 4624 (2016), shear measurements in torque mode or block-shear tests and the semi-quantitative cross cut or cross hedge according to ISO 2409 (2013) or ASTM D3359-17 (2017). However, the pull-off tests remain popular due to their practicality and precision (de Meijer and Militz 2000; Özdemir and Hiziroglu 2007).

In this study, the effect of artificial weathering and temperature cycling on the adhesion strength of white and brown waterborne acrylate coating systems used for wooden windows was determined. The effect of the type of coating, coating thickness and number of layers on the adhesion strength was investigated.

MATERIAL AND METHODS

Materials

Experiments were carried out using Norway spruce (*Picea abies* L.) with a density of 496 kg/m^3 (at an equilibrium 12% moisture content). A total of 144 defect-free samples with dimensions of 140±2 × 140±2 × 10±2 mm (longitudinal × radial × tangential) for temperature cycling test and 150 × 37 × 18 mm (longitudinal × radial × tangential) for artificial weathering with the 45 ± 10° inclination of the growth rings to the test surface were prepared and conditioned in T = 20 ± 2°C and RH = 65 ± 5%. The acrylate opaque dispersions from different producers (specification in Table 1) were subsequently applied according to the technical data sheets. The samples were coated with brown 3-layered and 4-layered and white 3-layered systems (Table 2).

TABLE 1. Specification of coating systems.

Producer	Impregnation	Base Coat	Middle Coat	Surface Coat
1	fungicides and insecticides (propiconazole, permethrin, iodopropynyl butylcarbamate (IPBC))	acrylate basis	acrylate basis	acrylate basis, UV-stabilizers (benzotriazole)
2	IPBC and propiconazole as fungicides	acrylate basis	acrylate basis, zinc oxide	acrylate basis, zinc oxide, phenyl-phosphates
3	IPBC and permethrin as biocides	alkyd-acrylate hybrid basis	acrylate basis	alkyd-acrylate hybrid basis with zinc oxide, siloxanes, fungicides, and UV-stabilizers (benzotriazole)
4	propiconazole and permethrin as biocides	acrylate basis	acrylate basis	acrylate basis, UV-stabilizers (benzotriazole), silicon dioxide and methylsiloxane
5	IPBC and permethrin as biocides	acrylate basis	acrylate basis, UV-stabilizers (benzotriazole), silicon dioxide and methylsiloxane	acrylate basis, UV-stabilizers (benzotriazole), silicon dioxide and methylsiloxane
6	propiconazole and IPBC as fungicides	acrylate basis	acrylate basis modified with fatty acids and tall oil, also containing UV-stabilizers (benzotriazole)	acrylate basis modified with fatty acids and tall oil, also containing UV-stabilizers (benzotriazole)

Note: Only informative specification of the tested coatings was provided by producers

TABLE 2. Technology of coating application.

Coating system	No. of coats	Colour	Impregnation	Base Coat	Middle Coat	Surface Coat	Pigmentation
1	3	brown	dipping	dipping	none	spraying	iron oxide, Tungstic acid
2	4	brown	dipping	dipping	dipping	spraying	iron oxide, Tungstic acid
3	3	white	dipping	dipping	none	spraying	titanium dioxide

Four specimens were prepared for each combination of coating system (1-3), producer (1-6) and test method (AW, TC). The samples were conditioned at T = 23 ± 2°C and RH = 50 ± 5% for 14 days before coating thickness measurements to achieve equilibrium moisture content.

Coating Thickness

The coating thickness of the samples was determined before and after AW and TC by a non-destructive ultrasonic thickness meter Sursonic Squirrel (Testima, Prague, Czech Republic) according to ISO 2808 (2007). The methodology was the same as previously described in the study by Hýsek et al. 2018b. The probe was manually put on the measured surface with a hydrogel as a coupling agent. The measurement readings from the device represented the average thickness of the coating in the measurement area (7 mm diameter). Two measurements were done per sample.

Adhesion Strength

The adhesion strength of samples was determined before and after AW and TC according to ISO 4624 (2016) using device Comtest OP1-P20 pull-off adhesion tester (ProInex, Czech Republic). Two measurements were performed per sample by gluing 20-mm-diameter dollies on the coating surface with epoxy resin (24 h of curing)—see Figure 1. A cut around the dolly in

FIGURE 1. Preparation of samples for pull-off test.

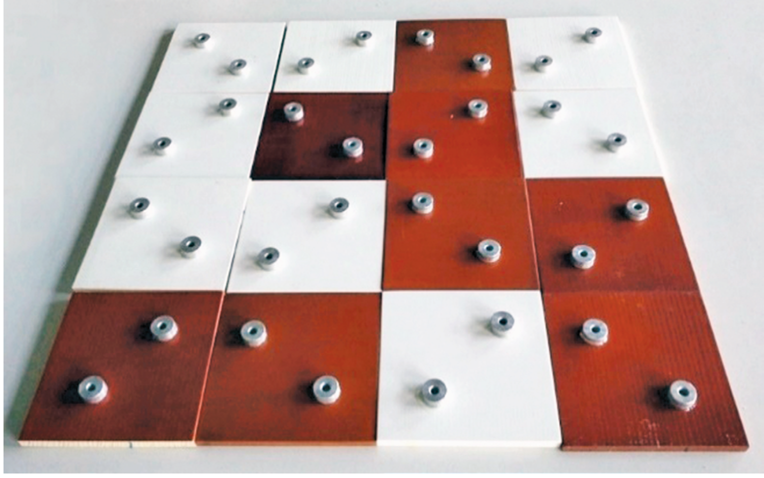
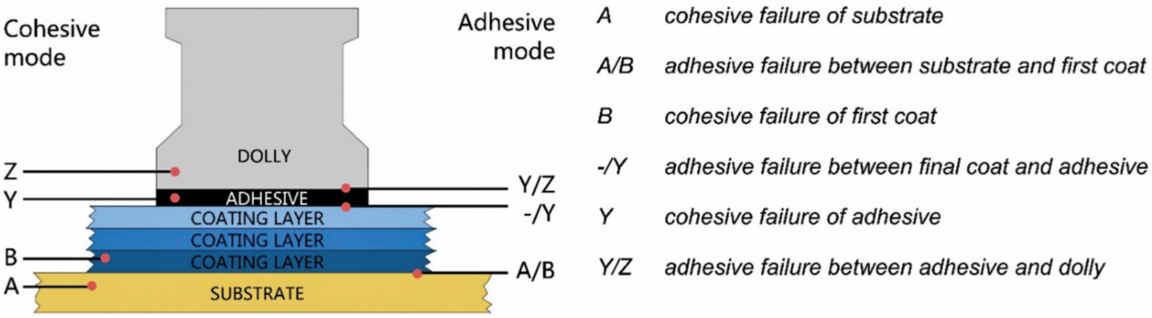


FIGURE 2. Types of failure during adhesion pull-off test (ISO 4624 2016).



the coating layer on each sample was performed. Pulling was done with 3 mm/min speed until separation of the dolly from the surface. The adhesion strength value of coating was determined in N/mm^2 from the display of the pull-off testing unit.

The nature of failure was estimated visually as a percentage damage of the total area covered by the dolly according to ISO 4624 (2016). The adhesive and cohesive types of failure are manifested in Figure 2.

Artificial Weathering (AW)

Prepared samples (72 pieces) were sealed with silicone to prevent moisture uptake through its cross-section and placed in a QUV tester (Q-Lab, USA) for 1008 hours (6 weeks). AW was performed on the basis of EN 927-6 (2006) and consisted of the phases specified in Table 3. After AW, the coating thickness and adhesion strength were remeasured.

Temperature Cycling (TC)

Temperature cycling was carried out according to ČSN 67 3098 (1988) in a climatic chamber Discovery My DM340 (ACS, Italy). Prepared samples (72 pieces) were exposed to the temperatures given in Table 4 and then conditioned in a climate chamber at 20 °C and 65% RH until they reached air dry moisture content. The coating thickness and adhesion strength were subsequently remeasured.

TABLE 3. Weathering phases and their specification.

Rank	Phase	Duration (h)	Temp. (°C)	UV irradiance (W/(m²nm))	Spraying (l/min)	Repetition
1	Condensation	24	45 ± 3	x	x	3×
2	UV+spray	144 (2.5 h UV + 0.5 h spraying)	60	0.89 (λ = 340 nm)	6.5	3×
3	Condensation	24	70	x	x	3×
4	UV+spray	144 (2.5 h UV + 0.5 h spraying)	60	1.25 (λ = 340 nm)	6.5	3×

TABLE 4. Specification of temperature cycling parameters.

Rank	Phase	Number of cycles	Temperature
1	Phase 1	10	T1 = 50°C (1 h); T2 = –30°C (1 h)
2	Phase 2	10	T1 = 70°C (1 h); T2 = –30°C (1 h)
3	Phase 3	5	T1 = 100°C (1 h); T2 = –40°C (1 h)
4	Phase 4	5	T1 = 115°C (1 h); T2 = –40°C (1 h)

Statistical Evaluation

The statistical evaluation of results was performed using an analysis of variance (ANOVA) and post hoc Tukey´s HSD multiple comparison test with an α significance value of 0.05. All of the statistical analysis was carried out using software Statistica 12 (StatSoft, USA) and MS Excel (Microsoft, USA).

RESULTS AND DISCUSSION

The results of adhesion strength and coating thickness measurements of unexposed and exposed (after AW and TC) samples are presented in Figure 3 and Table 5. In the statistical analysis of variance, a statistically significant difference was observed only between adhesion strength values before and after AW ($p = 0.00$), not before and after TC ($p = 0.64$). Using Tukey´s HSD multiple comparison test, the adhesion strength of unexposed and exposed coating systems did not statistically differ between the types of coatings (Table 2 and 5, Figure 3) or between individual producers (Table 1, Figure 3) ($p > 0.05$).

The values of adhesion strength of unexposed acrylate coatings were in the range of 0.4–1.1 N/mm^2. The adhesion strength of white coating systems was lower compared to brown ones for all of the coating types (Table 5, Figure 3). The highest values were obtained for producer 1, specifically for brown coloured samples 1-1 (= Producer 1—Coating system 1, specification from Table 1 and 2) and 1-2, the lowest for white samples 2-3 and 4-3.

The exposed samples were characterized by higher adhesion strength in the range of 0.1–1.6 N/mm^2 after AW (Table 5). Some coatings were characterized by statistically significant increase of adhesion strength after AW (Table 5). This is in the accordance with study of Yalcin and Ceylan (2015). The reason for the adhesion strength increase may be attributed to the continuous curing that occurs in the coating layers with further weathering (Çakıcıer 2007). On the other hand, the study by Ugulino and Hernández (2017) states that the solvent-borne coatings were characterized by approximately 50% of adhesion loss after an artificial aging test. The overall highest values of adhesion after AW were obtained for producer 3. The variability of adhesion strength values highly increased in most cases (Figure 3). The lowest variability of adhesion strength after AW was observed for one type of tested acrylate coating (Producer 4). The highest values of adhesion strength were observed for white coating systems for all of the producers, specifically for samples 2-3 and 1-3 (Table 5, Figure 3). It is in the accordance with the study of Oberhofnerová et al. (2018) wherein the white acrylate coatings achieved different results compared to the brown ones. In addition, in the work of Grüll et al. (2011), white coating systems showed the higher durability during exterior weathering compared to

FIGURE 3. The adhesion strength of samples before and after AW and TC (ANOVA results representing the 95% confidence interval in vertical bars).

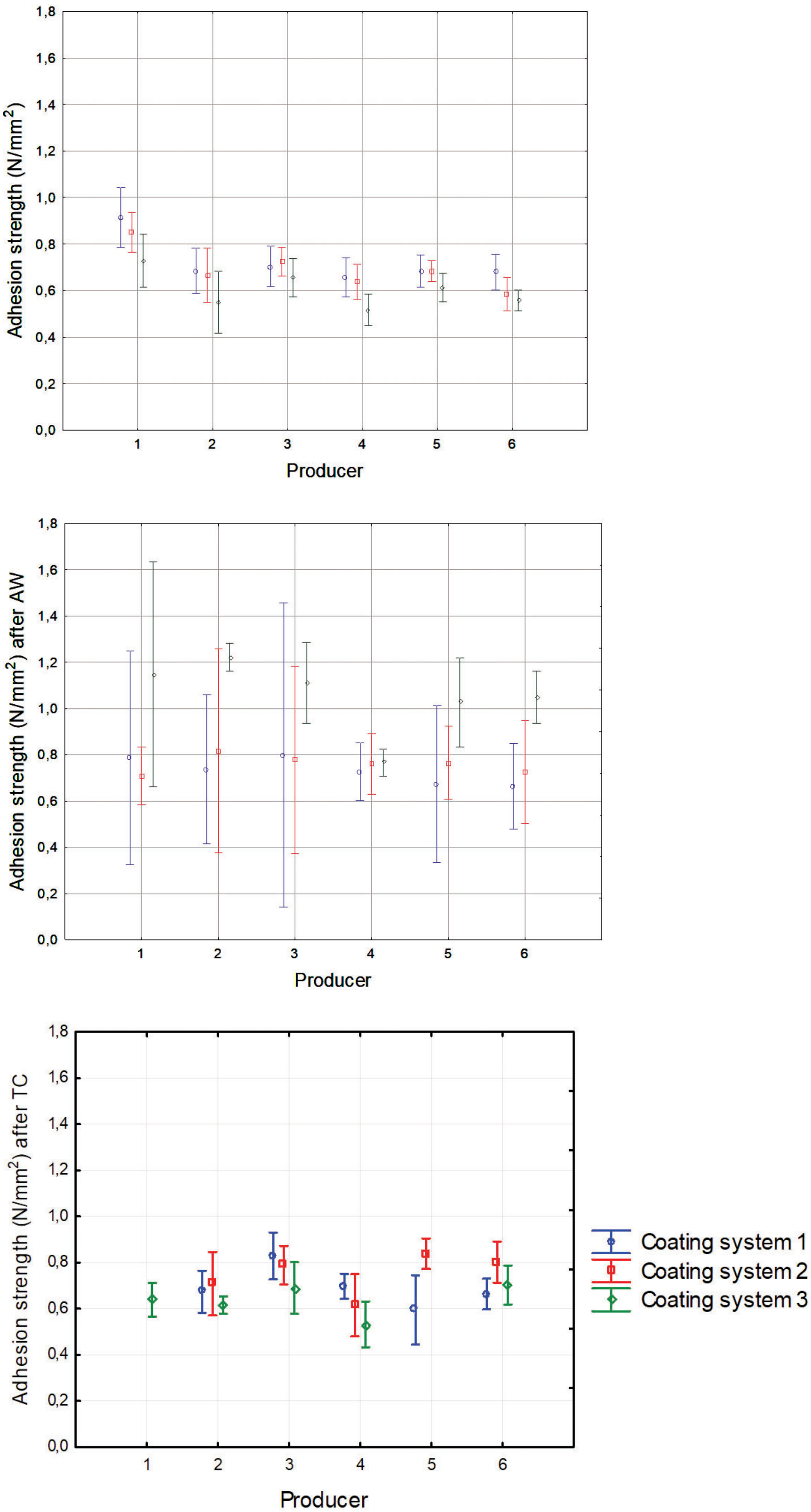


TABLE 5. Adhesion strength and coating thickness of samples before (X) and after AW and TC.

Prod.	Coating system	Adhesion strength (N/mm²)			Coating thickness (μm)			Adhesion change (%)	
		X	AW	TC	X	AW	TC	AW	TC
1	1	0.92	0.79 (N)	*	93.00	80.00		**–14.03**	
	2	0.85	0.71 (N)	*	93.38	79.33		**–16.67**	
	3	0.73	1.15 (S)	0.64 (N)	102.67	82.00	138.67	**57.74**	**–12.29**
2	1	0.69	0.74 (N)	0.67 (N)	112.25	104.20	114.33	**7.45**	**–1.70**
	2	0.67	0.82 (N)	0.71 (N)	111.33	105.40	107.00	**22.70**	**6.25**
	3	0.55	1.22 (S)	0.62 (N)	113.40	115.67	130.00	**122.12**	**11.82**
3	1	0.70	0.80 (N)	0.83 (N)	113.11	104.67	133.67	**13.56**	**17.59**
	2	0.73	0.78 (N)	0.79 (N)	104.90	105.33	100.33	**7.13**	**8.74**
	3	0.66	1.11 (S)	0.69 (N)	106.67	102.40	113.67	**69.32**	**5.25**
4	1	0.66	0.73 (N)	0.70 (N)	93.50	96.33	87.00	**10.77**	**6.20**
	2	0.64	0.76 (N)	0.62 (N)	94.50	103.67	99.33	**19.31**	**–3.45**
	3	0.52	0.77 (N)	0.53 (N)	108.40	111.20	93.33	**48.16**	**2.84**
5	1	0.68	0.67 (N)	0.59 (N)	100.50	103.33	100.60	**–1.46**	**–13.07**
	2	0.69	0.77 (N)	0.84 (N)	109.33	104.67	102.67	**11.68**	**22.38**
	3	0.61	1.03 (S)	*	96.00	78.20		**67.28**	
6	1	0.68	0.66 (N)	0.66 (N)	118.00	105.00	122.00	**–2.57**	**–2.45**
	2	0.58	0.73 (N)	0.80 (N)	142.86	120.25	134.00	**24.08**	**37.20**
	3	0.56	1.05 (S)	0.70 (N)	99.22	91.80	92.33	**87.51**	**25.84**

Note: After TC, these coatings released oil-based substances and it was therefore impossible to measure the adhesion strength because of poor bonds between coating surfaces and dollies. (*)

(S) signifies the statistically significant and (N) signifies NOT statistically significant difference between the adhesion strength before and after AW or TC (analyzed by Tukey´s test at 95% significance layer)

different coloured coatings. Hýsek et al. (2018a) also confirmed completely different performance between white and brown coating systems in other important evaluation characteristics of wood window coating. The brown coating systems were significantly less water-permeable than the white systems.

After TC, the exposed samples were characterized by the adhesion strength in the range of 0.4–0.9 N/mm². The highest adhesion strength was observed for samples 5-2 and 3-1 (Table 5, Figure 3). The best overall results were recorded for producer 3 as well as after AW. These findings are at variance with the studies of Demirci et al. (2013) and Kesik et al. (2015), where the temperature caused a decrease in the adhesion strength. However, there was no statistically significant difference between unexposed and exposed samples after TC ($p = 0.64$).

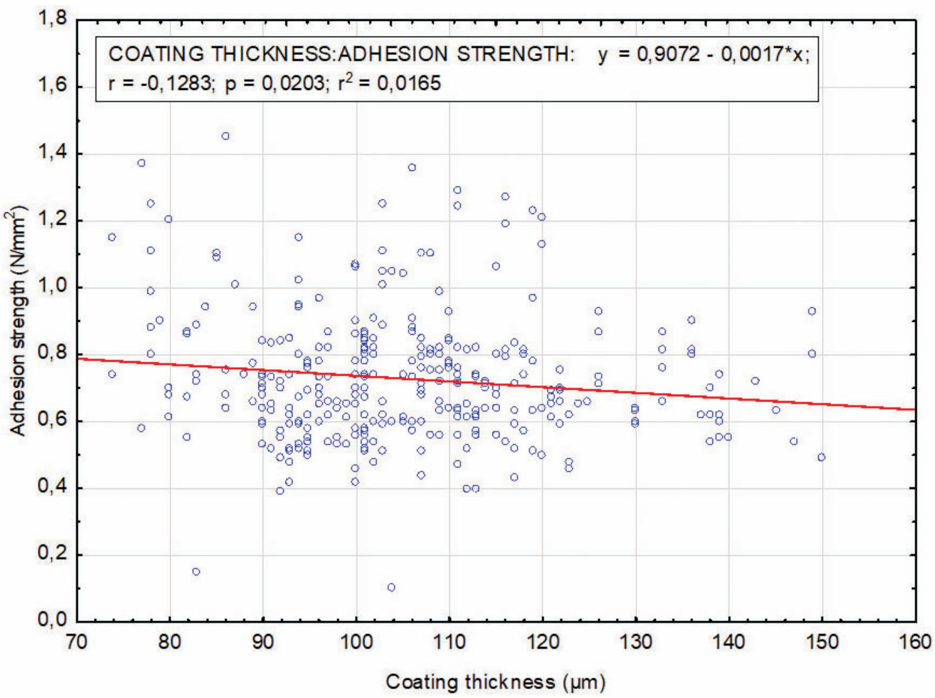
TABLE 6. Nature of failure in adhesion tests (mean values in %).

TEST	A	A/B	B	-/Y	Y	Y/Z
X	27.8	65.2	4.0	1.6	0.0	1.5
AW	25.9	50.2	11.1	12.2	0.0	0.5
TC	54.1	36.9	3.0	4.7	0.0	1.4

Further analysis of the adhesion was performed by evaluating the nature of failure caused by the pull-off test. Table 6 presents the mean values of failure type in percentages according to scale given in the Materials and Methods section (ISO 4624 2016).

The highest failure of samples was observed between the wood and the first coat (A/B) followed by the failure in wood (A) in the case of unexposed samples. After AW, the A/B value decreased and adversely, the failure in the first coat (B) and between the final coat and adhesive increased (-/Y). This effect was probably caused by the initial state of coating layer degradation after AW. After TC, the failure in wood (A) increased twice and the failure between the wood and the first coat (A/B) decreased. The tested coating systems were characterized by good quality in most cases because the failure was mostly in the wood or in the interface of wood/coating (adhesion character of failure). Cohesion type of failure in coatings was observed especially for samples 4-2 (29%), 5-2 (22%), 5-1 (12%), 6-2 (7%), 6-1 (6%). In addition, after TC, the cohesion type of failure in the coatings slightly increased.

FIGURE 4. Correlation between adhesion strength and coating thickness.



The different application technology resulted in different thicknesses of coating film (Table 5). The mutual relationship between adhesion strength and the coating thickness was investigated. The correlation was very weak with the coefficient of correlation $r = -0.13$; however, the effect of the film thickness on the adhesion strength of tested coatings was statistically significant ($p = 0.02$).

The measured adhesion strength of coated spruce samples was lower in the comparison with other studies focused on waterborne coatings (Bardage and Bjurman 1998; Sögütlü et al. 2016). The adhesion strength results seem to be significantly affected by minor variations in the test steps—the choice of adhesive, the preparation of the coating surface or the test dolly influence the adhesion strength results as well as the wood material itself (Demirci et al. 2013; Kesik et al. 2015). The mechanical anchoring due to different surface roughness of wood substrate is also very important for the adhesion of coatings to wood (Bardage and Bjurman 1998). The results are not easy to compare with previous studies because more factors can influence adhesion of coatings to wood substrate as mentioned above, but they can be suitable for comparing the quality of the tested coatings with each other.

CONCLUSIONS

The analysis of the adhesion quality of water-based acrylate coating systems was done using a pull-off adhesion test and failure characteristics. The samples were exposed to artificial weathering and temperature cycling in order to simulate the aging process in an external environment. After AW, the adhesion strength increased for all of the samples simultaneously with the variability of adhesion values. A slight increase in adhesion values was observed after TC as well, but it was not statistically significant. Based on the results, the adhesion strength did not significantly differ between tested acrylate coating systems. White coating systems were initially characterized by lower adhesion strength before aging, but after AW and TC, their adhesion strength was higher in the comparison with brown ones. Although there was no statistically significant difference between coating systems themselves after AW, the white coatings were affected the least. The overall best results were obtained for the coatings based on alkyd-acrylate hybrid basis (Producer 3) and lowest adhesion strength variability after AW was observed for one tested type of acrylic coating system (Producer 4). The tested coating systems were characterized by good quality in the most cases. The failure was mostly observed in wood substrate or in interface of wood/coating (adhesion character of failure). The effect of different layering on the adhesion strength was not demonstrated in this study. The adhesion strength of the tested coating was characterized by very weak correlation with the coating thickness.

The results of adhesion strength of the same type of coatings are very variable in the studies previously conducted. The exact values depend on many parameters of substrate and the coating itself. On the other hand, they allow for a comparison between tested variants of coatings mainly using artificial weathering in laboratory conditions. In addition, the nature of failure analyses provides a good idea about the coating quality.

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