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THE EFFECT OF AGING TESTS ON GLOSS AND ADHESION OF LACQUER COATINGS ON WINDOW ELEMENTS FROM PINE WOOD

The aim of this work was to investigate the formation of gloss and adhesion in accelerated thermal aging test selected lacquer systems formed on pine wood. Window elements were finished with lacquer systems, including impregnate, primer, and inter- and top lacquer layers in two colour versions white and cypress respectively. The range of investigations included gloss measurement and adhesion (pull-off method) of coatings to a substrate. Based on the contact angle, the values of surface free energy (γ_S), work of adhesion (Wa) and surface tension at the interface (γ_{SL}) were calculated, together with their dispersion and polar shares. On the basis of the experimental results it was stated among others, that tested finishing's were characterized by a semi-gloss effect, which was stable under thermal aging cycles. The lacquer coatings showed good adhesion to the substrate. The values of the Wa parameter remained at a high level as well. In turn the γ_S parameter indicated the occurrence of strong adhesion interactions in the substrate and particular layers of the coating systems. Aging processes had no significant effect on the manner of the obtained relations.

Keywords: pine wood, window element, coating, aging test, gloss, contact angle, parameter of adhesion

Introduction

Wooden window joinery is exploited under extremely unfavourable conditions, radically different from those in which the majority of other products operate. Their external part is exposed to changing weather conditions, while the other side is exposed to agents present in certain rooms [Hora 2003; Mateńko--Nożewnik and Proszyk 2004a, b; Grüll et al. 2005]. Functional properties and the durability of windows mainly depend on the applied wood species, accepted technical solutions, precision of their manufacturing, and above all, on the protection of the surface from the influence of biological and atmospheric

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factors. It is done through the impregnation and application of lacquer coatings [Graystone 2003; Dawson et al. 2005; Ozgenc et al. 2012]. Apart from the destructive effect of biotic factors, abiotic factors also cause significant changes [Bulcke et al. 2008]. A decisive impact on the stability of windows is found in the following factors of corrosive atmospheric attack:

- variable humidity and direct contact with water due to rain and pollution e.g. acid rains
- interaction of solar radiation, especially UV
- significant changes in temperature [Williams et al. 2000; Custódio and Eusébio 2006].

These factors may be characterized by short or long-term, accidental or periodic fluctuations of different intensity [Roux et al. 1988; Creemers et al. 2002]. One of the most important factors determining the durability, cost of production and the aesthetic qualities of wooden door and window joinery is, as already mentioned by the type and quality of the coating forming a specified varnish system [Budakçi and Taşçioğlu 2013]. The most important in this respect are different combinations based on acrylic polymers and acrylic copolymers, because of their valuable properties, e.g. low toxicity, high resistance of UV-light and temperature and other aggressive factors as well as aging processes. The varied range of monomers and acrylic copolymers exhibit numerous properties which enable the production of a wide range of solvent and waterborne products with various film-forming substances intended for application by different methods [Proszyk 1999; Baumstark and Tiarks 2002; Hora 2004]. Wood surface finishing systems for window joinery usually consist of several layers. Manufacturers offer both covering systems and transparent sets, available in a wide range of colours. Impregnation formulations usually contain biocides, however, it is the topcoat that determines aesthetic-decorative and resistance properties of finishes [Ozgenc et al. 2012; Baysal et al. 2013].

Lacquer coatings on wood during the service life of building joinery are subjected to aging processes. The natural aging process of lacquer coatings is mainly caused by the above mentioned climatic factors. A significant role in the aging processes of coatings is played by chemical processes occurring in the film-forming substances, which are manifested in:

- brittleness due to progressive polymer decay, oxidation, or crosslinking
- migration of wood components through coatings, especially extractive substances and of components of lacquer products (chalking, bloom) [Pecina and Paprzycki 1995].

Those processes often lead to deterioration of the aesthetic-decorative and protective advantages of finishes, primarily gloss. Moreover, scratches and cracks of different types occur on the molecular level and one by one in the nano, micro and macro scale. All of them may cause reduced adhesion of coatings to the substrate [Ahola 1995; Williams et al. 2000; Custodio and Eusebio 2006).This parameter is decisive to the functional characteristics and

durability of the finished surface [Bardage and Bjurman 1998]. In this context, it was decided to determine the formation of adhesion of coatings for wood, based on the normative pull-off methods and taking into account the assumptions of the adsorption theory of adhesion.

The aim of this work was to investigate the formation of gloss and adhesion selected lacquer systems formed on pine wood under an accelerated thermal aging test.

Materials and methods

Semi-finished products obtained under industrial conditions from Scots pine wood (*Pinus sylvestris* L.) were the experimental material. The products were connected longitudinally with finger joints and glued together into three-layer elements with a one-component PVAC adhesive (D3 durability class). The elements were improved with four layers of lacquer systems including the impregnate, primer product in interlayer and top layer in two colour versions, cypress and white respectively. Detailed information concerning the products may not be published as they are covered by a clause of total confidentiality.

Figure 1 is a simplified block diagram of the production of windows from which test samples were obtained.

Investigations were performed on the thermal aging of lacquer coatings under artificial conditions according to the standard [PN-88/F-06100/07:1988] in the function of the number of cycles of changing temperatures, after three, six and nine cycles respectively. The aesthetic-decorative values of sand adhesion relative to the control samples was evaluated.

The investigation of gloss of coatings using the photoelectric method with a PICO GLOSS apparatus, model 503, were based on ten measurements taken, along the grain at three angles of incidence 20°, 60° and 85°, respectively. Gloss degrees (expressed in gloss units GU) were determined for coatings at the angle of incidence of 60°.

Adhesion of coatings to the substrate was tested by the pull-off method, and was performed, based on the procedure described in the respective standard [PN--EN ISO 4624:2004] , which allowed the determination of the minimum strength necessary to the tear off, of the coating perpendicular to the substrate surface. Aluminium dollies were bonded with the two-component silane-epoxy adhesive. After seven days of conditioning (20 ±2ºC, RH 65 ±5%), adhesion was tested with a *PosiTest AT* apparatus. The surfaces of both the dolly and the sample after de-lamination were assessed.

The test of contact angle (θ) of coatings was performed according to the procedure described in the respective standard [PN-EN 828:2000]. A microscope (magnification ×56), equipped with a goniometric head was used. Ten drops of

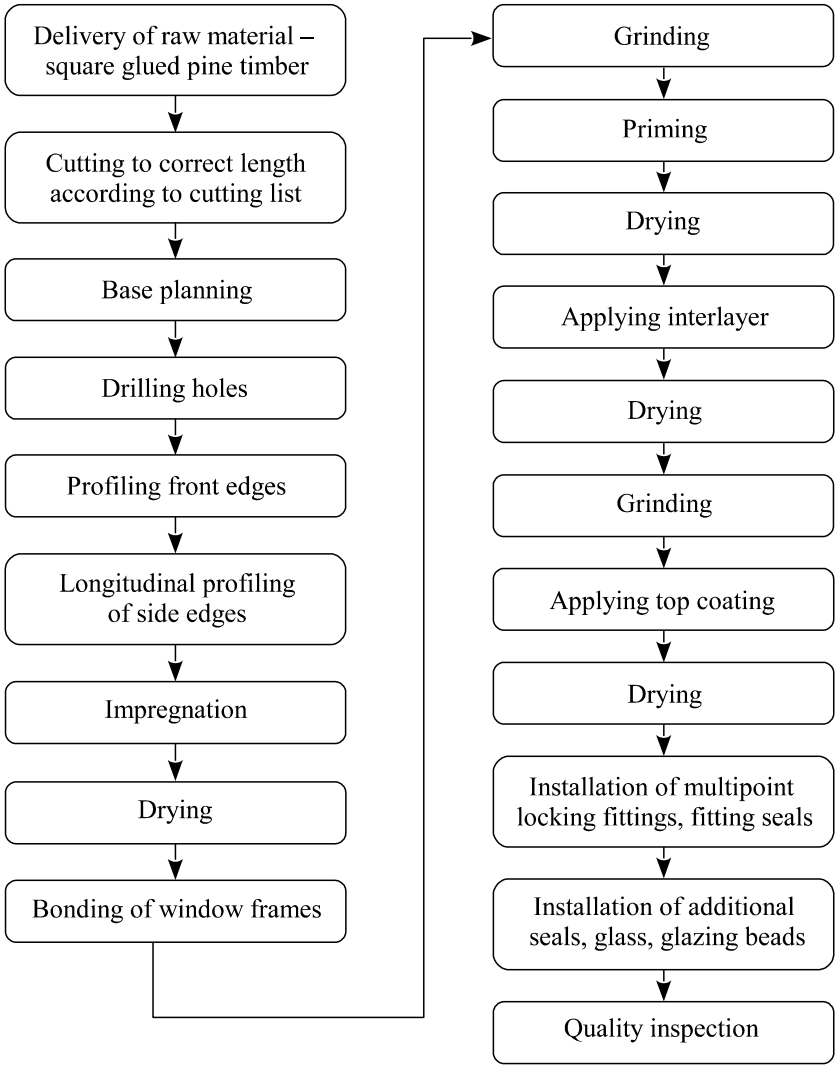

Fig. 1. The diagram of wooden window production technology

redistilled water (3.5 µl) were applied to tested surfaces with a chromatographic syringe. The θ angle was measured statically, five seconds after the application of drops. Investigations were performed, versus the number of aging cycles. Based on the θ angle, the values of surface free energy (γ_S), work of adhesion [Wa] and surface tension at the interface [γ_{SL}] were calculated, together with their dispersion and polar shares, according to formulas given in literature [Kloubek 1974; Neumann et al. 1974; Nguyen and Johns 1978; Liptáková 1980].

Results and discussion

The assessment of the appearance of different coating colours described in the study revealed that they were characterised by high aesthetic-decorative values. Thermal aging of the coatings in cycles of fluctuating temperatures did not have a negative impact on their appearance. The tests proved that the lacquer coatings under study were characterised by high stability and resistance especially thermal stress, as well as the accompanying humidity and shrinkage stress in the wood-coating system.

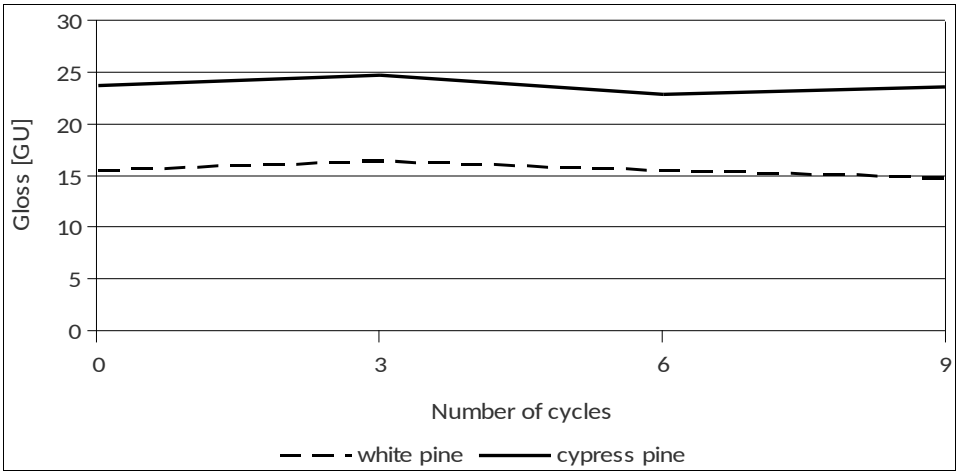

Fig. 2. Change of gloss as a function of the number of aging cycles

The tested surfaces in the form of control samples, for both coating colours: white and cypress, had gloss values from 15 to 25 GU, classified in the descriptive assessment as semi-gloss. There was no significant effect on the number of cycles of temperature changes, on the level of gloss of tested lacquer finishings.

An elementary statistical estimation of the development of adhesion for coatings of various colours to the pine wood surface as a function of the number of cycles of thermal aging and evaluation of disconnection mechanisms at destructive loadings are shown in table 1.

The overall assessment of the research findings indicates very high repeatability of the results. The values of the variation coefficient v for the options under consideration were low, and did not exceed 10%. The average adhesion values of the reference samples were in the range of 0.84-0.87 MPa for the considered colour variants.

The aging tests of all the test samples resulted in increasing adhesion to the substrate. Delaminations of the tested systems, with the destructive loadings

Table 1. Adhesion of coatings to the substrate

Colour of finishings	Number of cycles	Statistical data*)				
		$x_{min.}$	$x_{av.}$	$x_{max.}$	v	disconnection mechanism**)
		[MPa]			[%]	[%]
White pine	0	0.76	0.84	0.94	7.43	5A, 85B/C, 10n/m
	3	1.14	1.22	1.29	4.71	85B/C, 10n/m, 5-/Y
	6	1.12	1.23	1.41	4.04	90B/C, 5n/m, 5-/Y
	9	0.99	1.05	1.11	4.75	90B/C, 5n/m, 5-/Y
Cypress pine	0	0.81	0.87	0.95	6.09	70A, 30-/Y
	3	1.02	1.12	1.25	9.21	60-/Y, 30A, 10n/m
	6	0.97	1.03	1.16	7.42	85A, 10n/m, 5-/Y
	9	1.09	1.20	1.34	8.09	90A, 5n/m, 5-/Y

*) $x_{min.}$ – minimum value, $x_{max.}$ – maximum value, $x_{av.}$ – arithmetic average, v – coefficient of variation.

**) A – cohesive in substrate, B/C – adhesive between first and second coating, n/m – cohesive between n-layer and m-layer of the coating system, -/Y – adhesive of last coating and adhesive.

were quite varied. For the version pine wood cypress, the cohesional mechanism was dominant in the substrate, whereas in case of white pine wood mainly an adhesive destruction between the first and second layers was recorded. An elementary statistical estimation of the contact angle formation the two lacquer systems as a function of the number of cycles of thermal aging are summarized in table 2.

Table 2. Contact angle of tested systems before and after thermal aging with elementary statistical estimation

Colour of finishings	Number of cycles	Statistical data*)			
		$x_{min.}$	$x_{av.}$	$x_{max.}$	v
		[deg]			[%]
White pine	0	65.13	71.49	82.23	7.89
	3	65.50	70.45	77.03	4.43
	6	62.00	65.84	72.07	4.27
	9	60.48	64.20	71.03	7.26
Cypress pine	0	66.01	69.07	70.28	1.66
	3	66.01	68.06	70.07	2.07
	6	64.00	68.77	75.42	9.77
	9	65.27	66.90	68.05	1.15

The values of the coefficients of variation within the range of 1.15-9.77% indicate good repeatability of measurements. There was a slight decrease in the Θ angle values with an increasing number of aging cycles. More dynamic changes in the values were recorded for white pine wood finishing. The absolute value of this parameter decreased by 1.04-7.29 deg. As far as cypress-coloured coatings are concerned, these relations amounted to 1.01-2.17 deg. On the basis of the Θ angle and the theoretical formulas, which are based on the concept of adsorption theory of adhesion of polymers to the wood, values of γ_S, Wa and γ_{SL}, together with the dispersion and polar shares were calculated. Based on the literature data [Liptáková and Paprzycki 1983], γ_S for pine wood was adopted as 64.9 mJ/m^2, with the polar share (γ_S^p) of 38.9 mJ/m^2 respectively.

Figure 3 illustrates the formation of the γ_S parameter and γ_S^d and γ_S^p shares.

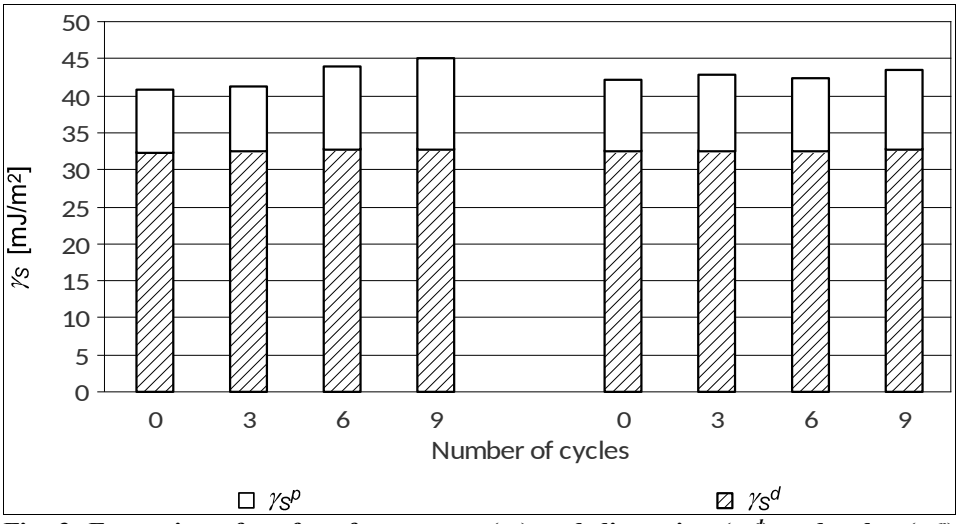

Fig. 3. Formation of surface free energy (γ_S) and dispersion (γ_S^d) and polar (γ_S^p) shares for surface of various coloured lacquer systems formed on the pine wood as a function of the number of cycles of thermal aging

The overall assessment of the γ_S parameter proved that they were similar for individual finishes, ranging within 40.76-42.15 mJ/m^2. In the function of the number of carried out aging tests generally, a slight increase in this parameter was found. The γ_S^p share affected the volume of the observed changes in γ_S. In turn, the values of the γ_S^d component in the tested samples were stable at 32 mJ/m^2. This indicates, therefore, that the conditions included in the thermal aging test experiments of coatings, the occurring changes influenced physico--chemical interactions of polar groups. Figures 4 and 5 present the results of Wa and γ_{SL} for the considered systems of substrate lacquer coating and their formation as a function of the number of thermal aging cycles.

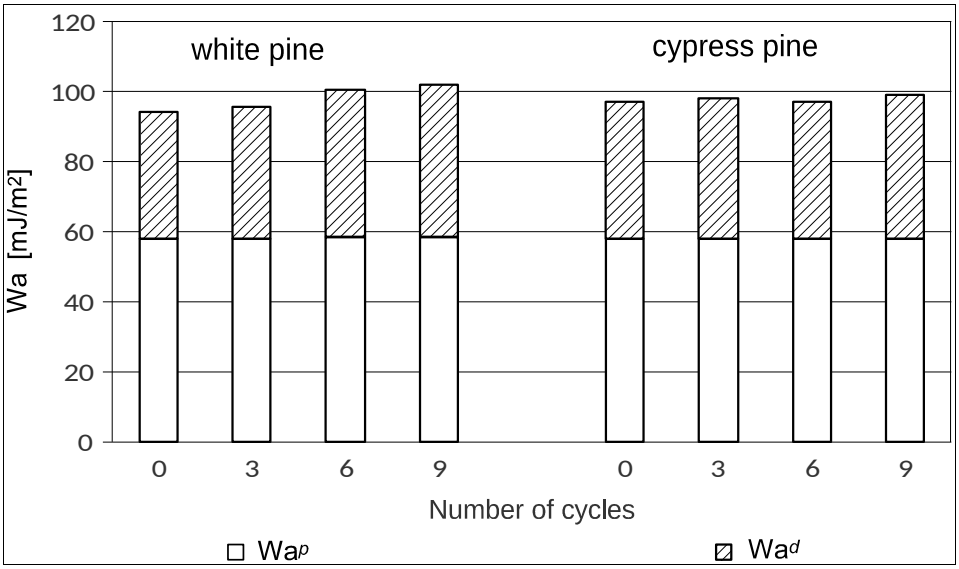

Fig. 4. Formation of the work of adhesion (Wa) and dispersion (Wa^d) and polar (Wa^p) shares for surface of various coloureds lacquer systems formed on pine wood as a function of the number of thermal aging cycles

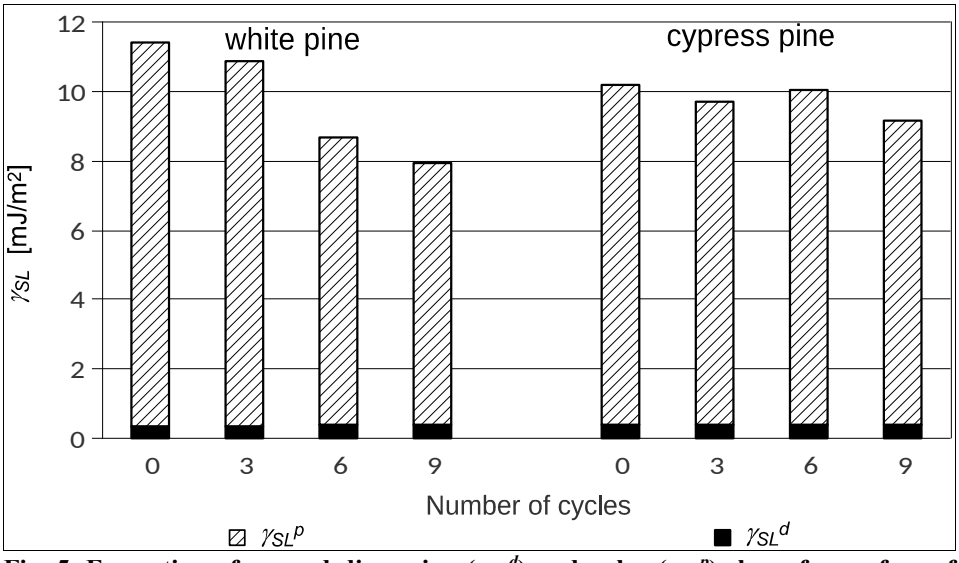

Fig. 5. Formation of γ_{SL} and dispersion (γ_{SL}^d) and polar (γ_{SL}^p) share for surface of various coloureds lacquer systems formed on pine wood as a function of the number of thermal aging cycles

It was found that the tested coating systems were characterized by high values of Wa, exceeding 90 mJ/m^2. According to the literature data, these were good relations. The values of Wa^d ranked at a relatively similar level of 58 mJ/m^2, while Wa^p values were varied. An interdependence was observed in its relations modifying the values of Wa in the tested lacquer coatings and γ_{SL}. Higher values of Wa were recorded at lower γ_{SL}. This finding confirms the theoretical assumptions included in the criterion of minimizing energy on the boundary interfaces. The formula assumes that the criterion claiming that maximum adhesion can be achieved if the system minimises γ_{SL} at the interface, where the materials are in contact with each other. It is the principle of the minimisation of surface tension on the interface of contacting materials. Some authors report that this value should range from 1 to 3 mJ/m^2 [Hellwig et al. 1968; Potente and Krüger 1978; Pirmasens 1983; Pecina and Paprzycki 1995]. It should be noted, however, that none of these studies of lacquer systems met this criterion. It proves that the physicochemical interactions considered in reference to this criterion are not fully adequate to the theoretical possibilities.

Conclusions

1. Tested finishes including lacquer coating systems in the colour versions of white and cypress formed on pine wood, were characterised by a degree of gloss, determined in the descriptive evaluation as semi-gloss. This property was stable in thermal aging cycles in the version of changing temperature cycles.

2. The finishing coatings showed good adhesion to the substrate. The aging tests revealed an upward tendency for values of this parameter.

3. The tests on adhesion of lacquer coatings to pine wood revealed that the cypress colour system was characterised by better relations in the delamination mechanisms.

4. The value of surface free energy (γ_S) in the tested coatings was approx. 42 mJ/m^2, where the dispersion share was predominant. The γ_S of coatings increased in function of the number of aging tests cycles. The γ_S^p share influenced variation in the γ_S value.

5. The value of the γ_S parameter in the coating systems was lower than this value in pine wood. It indicates strong adhesive interactions between the substrate and lacquer coating systems.

6. The values of the adhesion work (Wa) remained high, exceeding 90 mJ/m^2. Values of this parameter were increasing in the function of the number of aging tests cycles.

7. The tested lacquer coating systems did not meet the principle of minimisation of surface tension at the interface of materials in contacting materials.

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PN-EN 828:2000 Adhesives – Wettability – Determination by measurement of contact angle and critical surface tension of solid surface

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