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Adhesion of water-borne acrylic and hybrid paint on wood treated with primers[2]

Prianjanje vodotopljivih akrilnih i hibridnih boja na predpremazima obrađeno drvo

Original scientific paper • Izvorni znanstveni rad
Received – prispjelo: 28. 10. 2000 • Accepted – prihvaćeno: 05. 12. 2000
*UDK 630 * 829.1*

SUMMARY • *Spruce substrates of radial and tangential texture have been successfully treated with isocyanate-based and resorcinol-based primers to improve the adhesion of water-borne paint in dry and wet condition. Microscopic evidence shows that the wet adhesion of the acrylic or hybrid base-coats was severely impaired by the water ingress into these porous and brittle layers. Direct application of the top-coat on wood,especially on that treated with primers, proved advanageous over base-coat / top-coat system in terms of wet adhesion.*

Key words: *spruce, durability, adhesion, primers, water-borne paints, hybrid paints, SEM microscopy*

SAŽETAK: • *Postojanost ugrađenog drva uvelike ovisi o cjelovitosti i dobrom prianjanju (adheziji) zaštitnih i dekorativnih premaza. Trajnost premaza je određena fizikalnim svojstvima filma, ali podjednako je važna i veza premaza s drvom. Moderni vodotopljivi premazi, a naročito debeloslojne neprozirne boje, često iskazuju slabo prianjanje na mokrom drvu tj. popuštanje međusobne veze u uvjetima visoke vlažnosti i povišenog sadržaja vode drva u uporabi.*

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[2] Rad je predstavljen na Drugom europskom kongresu o površinskoj obradi drva u Hagu u Nizozemskoj u listopadu 2000. Ovdje donosimo, s dozvolom organizatora, malo izmjenjeni oblik članka radi interesa šireg čitateljstva.
The paper has been presented at the Second wood coatings congress in The Hague, The Netherlands, in October 2000. Whe publish here, with the aproval of the organizer, a slightly altered version of the text in order to present it to broader readership.

Smrekove daščice radijalne i tangentne teksture su uspješno površinski obrađene dvjema vrstama predpremaza (primera) da bi se poboljšalo prianjanje vodotopljivih boja u suhom i mokrom stanju. Na predpremazane podloge je nanešena temeljna boja (dvije vrste) i završni nalič, ili je pak završni nanešen izravno (bez temeljnog) u dva sloja. Kakvoća prianjanja je ispitana mjerenjem sile odvajanja premaza (otkidanjem zalijepljenih kružića) na suhim i namočenim uzorcima. Mikroskopska analiza sljubnice i premaza je načinjena da se dobije uvid u strukturne i fizikalne razloge promjena kakvoće prianjanja.

Obje vrste predpremaza su se pokazale uspješnima u poboljšanju prianjanja gornjih premaza, pogotovo na mokrom drvu i na površinama kasnog drva, poznatima po problematičnom prianjanju. Pokazalo se da svojstva substrata bitno određuju rezultate mjerenja prianjanja, pa su se vizuelna i mikroskopska analiza lomnih slojeva pokazale presudnom u interpretaciji čvrstoće prianjanja.

Prianjanje na mokrom drvu je oslabljeno u sloju temeljne boje, u čiju poroznu strukturu prodire voda i oslabljuje joj kohezijsku čvrstoću. Prianjanje završnog naliča je bolje u suhom, a izrazito bolje u mokrom stanju, ako je nanešen izravno na predpremazanu podlogu, tj. bez temeljne boje.

Ključne riječi: *smrekovina, postojanost, prianjanje, predpremazi (primeri), vodotopljive boje, hibridne boje, SEM mikroskopija*

1. Introduction
1. Uvod

The main scope of this investigation was to study the effect of primer application on the adhesion of acrylic and hybrid exterior paint, and to look into the possible penetration of the wood-impregnating chemicals into the cell wall. It was supposed that the penetration and fixation of the isocyanate primer and phenolic primer within the cell wall can modify the substance in such a way that

a) the coherence and cohesive properties of the substrate are enhanced, or/and

b) the adhesion (chemical fixation) of subsequently applied polymers, namely acrylic or hybrid water-based coatings, and especially their wet adhesion, is improved.

The adhesion of non-penetrating materials is a surface phenomenon, and depends on surface forces, i.e. on the physics of the liquid-S3 interface. The impregnating chemical should either enhance the affinity of the coating to the surface of modified wood, or consolidate and strengthen the substrate, so that fixation of the coating is still great, but that the delamination between the cell wall layers is reduced. The micrographs of the adhesion failures from the 1997 EU-AIR investigation indicate that the adhesion failure of painted and exposed wood may originate between the surface of the wood and the paint film (adhesion failure at the interface), but also as a cohesive failure which occurs either within the wood or in the layer of the paint (Turkulin et al. 1998a & 1998b).

Attempts have been previously made to force the polymer substances of various chain length and molecular weight into the cell wall in order to modify the "wood substance", i. e. to cause the penetration of the polymer into the cell wall. Interpretation of the results have been dubious, since some authors claimed that the cell wall was modified, and other argued that big organic molecules cannot pierce through densely packed cell-wall (especially S2 layer) substance. Finally, the work by Rapp et al. (1999) surveyed the work in the field and clearly confirmed that melamine resin can be forced into the cell wall structure, but under conditions where small wood specimens were vacuum impregnated with very weak resin solution (7.5% solids).

During the last decade water-borne paints for exterior wood have been introduced and ever more widely accepted as an environmentally friendly alternative to the application of traditional solvent–borne systems. They come either as acrylic emulsions or alkyd – acrylic "hybrid" paints. The commercially available alkyd emulsion consists of alkyd droplets about 0.5 to 1.0 μm in diameter, emulsified in water by surfactants. This dimension is about 50 – 100 times larger than the size of an alkyd molecule dissolved in an organic solvent. The same problem concerns the application of water-borne acrylic paints for exterior wood. Therefore the ability of an acrylic or alkyd emulsion to penetrate into wood, and possibly into cell wall structure, has been questioned. The work by Côté and Robinson (1968), Schneider (1970) and Nussbaum (1994) showed by various microscopic methods that the application of paints results in no resin penetration, but only solvent penetration into the cell wall.

The adhesion of water-borne finishes in wet condition is notoriously poorer than is the case with solvent-borne paints. Ahola (1995) has shown that the adhesion of emulsion paints can be improved by application of pigmented stain. Vick et al. (1996) patented a resorcinol-based primer which was reported to improve the strength of adhesive bonds which also proved successful in wet conditions (Vick 1997). De Meijer (1999) has recently published an extensive literature survey on the subject, hence this report mainly covers the experimental details and results of a limited-scale research programme.

2. Methodology
2. Metodologija

Wood panels were chemically treated with low-viscous impregnating chemicals and subsequently the water-based acrylic /hybrid coating system was applied. Adhesion testing and SEM (Scanning Electron Microscopy) analysis of the cross-section fracture surfaces were performed.

Materials

Wood: Spruce (*Picea abies* Karst.) was the substrate. The wood was straight grained, without visible defects, cut into panels, quarter-sawn, 300 (L) X 100 (R) X 20 (T) mm. The panels were fine-machined (planing), the coating surface sanded (grit size 180) on a belt sander using minimal belt pressure and finaly fine-scraped prior to chemical treatment. Panels of average density (0.390 - 0.420 g/cm^3) were conditioned at 50%. r.h. and 20 ^{o}C before treatment. Ring width (mean values of 13 readings per board) ranged from 2.2 to 3.6 mm, the average value being 2.7 mm with the latewood proportion of 13.8%.

A number of panels was prepared as flat-sawn specimens from the material of similar physical characteristics. These were used for testing the adhesion on latewood bands, since it is known that the adhesion of viscous finishes is lower on latewood surfaces than on earlywood bands due to the poorer penetration into latewood lumina.

Impregnating primers

Variant I: Isocyanate primer was prepared as a 10 % solution of the *SUPRASEC DNR* in toluene. *Suprasec* is an ICI Polyurethane product - adhesive for particle-board production (Diphenylmethan-Diisocyanat - MDI). The colour is transparent. Code: **IC primer.**

Variant II: Hydroxymethylated resorcinol *phenolic primer* is a resorcinol coupling agent for enhancing the adhesive bond strength on solid wood (patented, Vick et al. 1996). This is an aqueous solution of resorcinol-formaldehyde binder, with less than 7 % solid content. The colour is dark reddish-brown. Code: **HMR primer.**

Finishing

Impregnation

Panels (with end-grain open) were half-immersed for 30-minutes at atmospheric pressure into impregnating chemicals of the Variants I and II; after that period the excess liquid was removed (wiped off with a tissue paper) and the panels were left to dry naturally (20^{o}C, 50% r.h.) for three days. Control panels were left without impregnation.

Finishing systems

All conditioned panels (controls and those impregnated with primers) were finished using the following system:

Two water-soluble types of base-coat were applied by brush on the panels' testing surfaces, and dried naturally (20^{o}C, 50% r.h.) for 24 hours. An acrylic water-based base coat (Wessco-acryl *Tauchgrund*, further code **WB**) makes a standard finishing system with the subsequently applied top coat. A comparative base material was an hybrid base-coat (*Pentol TG Hybrid*, further code

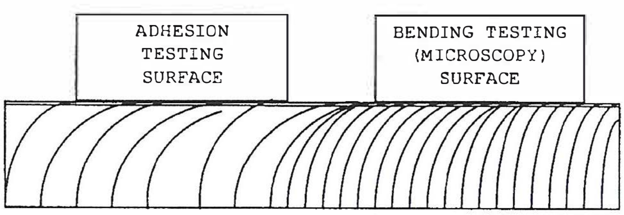


Figure 1. *Shematic presentation of the cross-section of the panel showing ring width and orientation, and portions of the painted surface used in particular tests.* • *Shematski prikaz poprečnog presjeka ispitne daščice s prikazom položaja i širine godova te dijelova bojane površine izložene određenim ispitivanjima.*

PB). On an number of panels the water-borne top coat was applied directly on wood to compare its adhesion with the properties of the base-coats. It was obvious that the surface was somewhat "blocked" by the fixation of the impregnating primers, since the application rate of the base coat on raw wood was significantly greater than on the modified surfaces. The spreading rates of the base coats were

Acrylic (WB) base-coat
90 g/ m^2 on the IC primer,
85 g/ m^2 on the HMR primer,
140 g/ m^2 for controls (raw wood surface).

Hybrid (PB) base-coat
60 g/ m^2 on the IC primer,
75 g/ m^2 on the HMR primer,
90 g/ m^2 for controls (raw wood surface).

Two coats of water based, high build, opaque, white acrylic coat *Wessco-acryl* (further **WT**) were applied by brush to obtain a dry film thickness of app. 100 m. The base coat and the first top coat were sanded (grit size 180) prior to application of the final coat. Drying time (at 20°C, 50% r.h.) of the first coat was 24 hours, and of the second coat 21 days. The spreading rates of the top coats were

1 st coat
95 g/ m^2 over the IC primer,
80 g/ m^2 over the HMR primer,
120 g/ m^2 for controls (raw wood surface).
170 – 190 g/ m^2 over base-coats

2 nd coat
160 – 190 g/ m^2.

Testing

Adhesion testing

Two general tests of the cured film adhesion to the substrate were performed: "dry" test and "wet" test. The former was an usual test method performed on specimens conditioned in the DIN normal climate (20°C, 65% r.h.). The latter test aimed at determining the effect of the impregnation on the adhesion of the film on moist or wet wood. The conditions of elevated moisture content frequently occur in exterior timber components during winter months even without liquid precipitation on the film surface, mostly due to the moisture flow from interior to the colder exterior surfaces.

The panels were cut into halves, 300 mm long, ca 57 mm wide sections or slabs on which all the surfaces, save for the finished face, were rough sanded and left unmodified. The size of the panels limited the number of tests to 12 per variable, and only 6 replicate tests could be done on wet sections. The size of these samples and the positions of testing surfaces can be seen on figures 2 to 4.

Adhesion testing was performed according to the EN 24 624 „pull-off" method. After curing the paint for 21 days, the screws (M8 - DIN 963 type) were glued by their heads to the paint surface using 2-component epoxy glue (AralditE 2011, curing period 24 hrs at 20°C, 50% r.h.). The excess cured adhesive at the glued bases of the studs was carefully removed by a cylindrical router immediately prior to testing. That created 15.4 mm diameter adhesive circular surface which was tested in tension perpendicular to the paint film by pulling off the screws like studs.

The panels for wet testing (with the glued studs, but the adhesive not routed) were immersed for 72 hours in water at room conditions prior to testing. In order to simulate the internal moisture flow to the paint interface, only the unfinished surfaces of the slabs were immersed, and the painted face was left to protrude ca 2 mm above the water level. That was supposed to ensure sufficient saturation of the interface without actual acting on the paint film with liquid water. Moisture content at end portions of the studs reached ca 40 – 70%, in the middle it amounted to about 20% (table 2).

Microscopic observation

Fractured transverse surfaces from the panels were gained by means of a bending test. Bending specimens were made by sawing the remaining halves of the panels into 220 (longitudinal) x 10 x 10 mm sticks. These were conditioned at 23±2°C, 50% relative humidity and tested in 3-point bending (constant speed 5 mm/min) so that the coated surface was in the tension zone. The tension zones of the tested specimens were separated and only the fractured transverse surfaces of these specimens were analysed. The small specimens were vacuum dried at 40±2°C for 24 h, and sputtered with platinum for microscopic analysis. Using the usual preparation method, it is estimated that the layer of platinum was approx. 15 nm thick (Zimmermann et al. 1994). The specimens were mechanically fastened to the mounting blocks so that the fractured edges were exposed for observation. The field-emission scanning electron microscope (FE-SEM) used was a JEOL JSM 6300 F situated at the EMPA. Images were recorded electronically and on the ILFORD FP4 PLUS b/w film.

3. Results and discussion
3. Rezultati i diskusija

Adhesion testing

The summary results of the adhesion testing are presented in tables 1 and 2, and on figures 2 to 4.

Dry adhesion properties

The analysis of the bond quality is based on both pull-off strength values and the failure characteristics, for the complete image about the finish condition can be made only if the origin of failure is taken into account. In dry testing, for example, the IC – primer treated specimens exhibited similar strength values on both base-coats, water-borne acrylic and hybrid. However, the smaller share of cohesive failure of the hybrid base-coat and larger proportion of wood failure shows that overall properties of this basecoat are better than those of acrylic base-coat.

Control specimens, i.e. non-primed wood finished with acrylic basecoat and top-coat (WB + WT), show good results: the strength values are some 20 % lower than wood substrate strength, the scattering of results is relatively low, and the failure occurs mostly as the fracture of the basecoat-wood bond at its interface. That means that the finishing system is well structured and that specimens are well prepared.

Application of the primers caused the failure mostly to occur either as a cohesive fracture within the basecoat, or in the wood. That shows that the primer application has contributed to the improvement of the base-coat/wood bond. HMR primed specimens yield lower strength values than radial-textured controls in all the three finishing systems (*index to control* in Table 1). However, a large proportion of wood failure makes it obvious that the results are controlled by the poorer substrate characteristics. The same is valid for the IC primed specimens, with even better strength values and larger wood failure proportions than with HMR primer. On tangential latewood surfaces the dry strength values are in the same order of magnitude or even better than on controls. That supports the finding that HMR primer improves adhesion, even on latewood bands where the penetration of the coating is relatively poor.

The fact that the strength values obtained on primed radial-textured specimens are lower than on controls, shows that the mechanical properties of the substrate may play an important role in overall strength results of such tests. The images of the pull-off tested surfaces (figures 2 to 4) clearly show that the strength results should be complemented and interpreted in terms of the type of failure in order to complete the argumentation about the differences in adhesion between the tested variables.

Control specimens (figure 2) exhibit the type of adhesion failure of the water-based top-coat and base-coat on dry, non-impregnated wood. The failure occurs mostly within the base-coat (i.e. its border to wood, darker areas), leaving traces of base-coat on the surface, and some white patches where the complete paint system remained on the substrate, but the epoxy was detached from the top-coat surface. The hybrid base-coat leaves dark surfaces on studs and on substrate (lower specimen on fig. 2). This indicates the following:

- Adhesion of top-coat to base-coat is better than is the link between the base-coat and wood.
- The cohesion of the hybrid base-coat is relatively weak, and the separation occurs mostly within this layer.

Isocyanate (IC) and resorcinol (HMR) primer impregnated specimens (fig. 3) show larger proportion of wood failure than control specimens, and the failure spreads mostly at the interface layer. Since HMR primer stains wood surface reddish-brown, it is easy to distinguish small areas of wood failure (pale), base-coat to interface failure (darker areas), and interface failure (predominant surface area).

The direct application of the topcoat on wood gives better strength results than the basecoat-including systems. The failure mainly appears as an adhesion fracture between the topcoat and wood, but this was expected knowing the high cohesive properties of the topcoat.

Wet adhesion properties

Wetting has drastically reduced the strength of the bond between the finish and the wood (*Index to dry* in Table 2). The ranking of the finishing systems applied is similar as in dry testing, only the differences in the order of magnitude are better pronounced in wet than in dry testing.

When base-coats are applied over primers , 60 – 80 % of the failure surface shows cohesion failure of the basecoat (figure 4). In dry testing the PB basecoat also failed predominantly in cohesion, but WB showed some adhesion-to-wood fractured surfaces. In wet condition the ingress of water molecules into the basecoat layer, and the substitution of its cohesive bonds by

Table 1.
DRY TESTING RESULTS - Cumulative results of the adhesion testing (pull-off test according to EN 24 624) • *REZULTATI SUHOG ISPITIVANJA – zbirni rezultati ispitivanja prianjanja otkidanjem zalijepljenih kružića prema EN 24624)*

TEST METHOD *NAČIN ISPITIVANJA*	FINISHING SYSTEM *SUSTAV POVRŠINSKE OBRADE*				PULL-OFF STRENGTH *ČVRSTOĆA PRIANJANJA*				FAILURE CHARACTERISTICS *ZNAČAJKE LOMA*		COMMENTS *PRIMJEDBE*
	PRIMER *PREDPREMAZ*	BASE COAT *TEMELJNI SLOJ*		TOP COAT - *ZAVRŠNI*	Nr OF TESTS *BROJ MJERENJA*	$\bar{X}$	S	INDEX TO CONTR. *INDEKS PREMA KONTROLNIM*	FRACTURED SURFACE *LOMNA POVRŠINA*		
		WB ACRL.	PB HYBR.			N/mm^2	N/mm^2		WOOD *DRVO*	FINISH *PREMAZ*	
DRY ON RADIAL TEXTURE *SUHO NA RADIJALNIM PLOHAMA*	CONTROL (no primer)	1X	-	ACRYLIC TOP COAT, 2X *AKRILNI ZAVRŠNI NALIČ 2X*	12	4,50	0,59	**1,00**	20 %	80% adhesion WB/wood	
		-	1X		12	5,21	0,62	**1,16**		90% cohesion PB	Good substrate and adhesion
		-	-		12	4,40	0,35	**0,98**	30 %	70% adhesion WB	
	HMR	1X	-		12	3,39	0,46	**0,75**		60% interface failure 40% cohesion WB	→ on earlywood zones → on latewood zones
	HMR	-	1X		12	3,73	0,23	**0,83**	50%	50% cohesion PB	
	HMR	-	-		12	3,79	0,38	**0,84**	100%		(Finish not tested)
	IC	1X	-		12	3,83	0,82	**0,85**		90 % Cohesion WB	Some interface failure
	IC	-	1X		12	3,44	0,35	**0,76**	80%	20% Cohesion PB	
	IC	-	-		12	5,14	0,91	**1,14**	80%	20% Cohesion WT	
DRY-TANGENT. *SUHO NA TANG.*	-	-	-		6	5,19	1,50	**1,15**		10% coh. WT 90% adh. WT/wood	
	-	1X	-		6	4,56	0,39	**1,01**		40% coh. WB 60% adh. WB/wood	
	HMR	-	-		6	6,13	1,32	**1,36**		10% coh. WB 90% adh. WT/HMR	HMR primer improves adhesion and strength of basecoat
	HMR	1X	-		6	4,43	0,65	**0,98**		90% adhesion WB	
SUBSTRATE *PODLOGA*		-	-	-	8	5,70	0,86	**1,27**	100%		

Table 2.
WET TESTING RESULTS - Cumulative results of the adhesion testing (pull-off test according to EN 24 624) • REZULTATI MOKROG ISPITIVANJA – zbirni rezultati ispitivanja prianjanja otkidanjem zalijepljenih kružića prema EN 24624)

TEST METHOD *NAČIN ISPITIVANJA*	FINISHING SYSTEM *SUSTAV POVRŠINSKE OBRADE*				PULL-OFF STRENGTH *ČVRSTOĆA PRIANJANJA*					FAILURE CHARACTERISTICS *ZNAČAJKE LOMA*		COMMENTS *PRIMJEDBE*
	PRIMER *PREDPREMAZ*	BASE COAT *TEMELJNI SLOJ* WB ACRL.	BASE COAT *TEMELJNI SLOJ* PB HYBR.	TOP COAT *ZAVRŠNI*	Nr OF TESTS *BROJ MJERENJA*	$\bar{X}$ N/mm^2	S N/mm^2	INDEX TO CONTR. *INDEKS PREMA KONTROLNIM*		FRACTURED SURFACE *LOMNA POVRŠINA* WOOD *DRVO*	FRACTURED SURFACE *LOMNA POVRŠINA* FINISH *PREMAZ*	
WET ON RADIAL TEXTURE *MOKRO NA RADIJALNIM PLOHAMA*	CONTROL (no primer)	1X	-	ACRYLIC TOP COAT, 2X *AKRILNI ZAVRŠNI NALIČ 2X*	10	1,20	0,74	**1.00**	0.27		50% adh. WB/wood 50% coh. WB	
		-	1X		11	1,78	0,67	**1,48**	0,34		100% cohesion PB	
		-	-		8	2,26	0,57	**1,88**	0,51		100% adhesion WT	4 studs failed in routing (end pos.)
	HMR	1X	-		10	1,77	0,46	**1,48**	0,52	20 %	70% coh. WB 10% WT/epoxy adh.	Wood = HMR interface failure
	HMR	-	1X		12	2,05	0,97	**1,71**	0,55		80% adh. WT/PB 20% coh. PB	Poor adhesion WT to PB
	HMR	-	-		12	2,80	1,02	**2,33**	0,74	50 %	(50% HMR interface) 50% epoxy adh.	Moist. content: end grain 40 - 70% middle 16 – 20 %
	IC	1X	-		10	2,00	0,42	**1,67**	0,52		60% coh. WB 40 % adh. WT/epoxy	
	IC	-	1X		12	3,17	0,86	**2,64**	0,92	30 %	60% cohesion PB	10% WT to epoxy adhesion
	IC	-	-		12	3,03	0,69	**2,53**	**0,59**	10 %	50% adhesion WT to IC	40% WT to epoxy adhesion
WET-TANGENT. *MOKRO NA TANG.*	-	-	-		6	1,25	0,68	**1,04**	**0,24**		100% adhesion WT on wood	Moist. content: end grain 40 - 75% middle 19 – 23 %
	-	1X	-		6	0,96	0,32	**0,80**	**0,21**		60% coh. WB 40% adh. WB/wood	
	HMR	-	-		6	1,44	0,38	**1,20**	**0,23**		50% adh. WB/HMR 50% adh. epoxy	HMR primer improves wood
	HMR	1X	-		6	1,70	0,28	**1,42**	**0,38**		60% coh. WB 40% adh. epoxy	strength and adhesion of subsequent coats

water infiltration leads to the swelling and the separation within the layer. It is suspected that the advantage of great micro-porosity of a basecoat during storage and transport of unfinished products becomes a major disadvantage i.e. the weakest point of the system. Liquid water can penetrate through cracks or migrate from deeper wood areas to its surface, nest in the voids of the porous base-coat and further reduce its coherence.

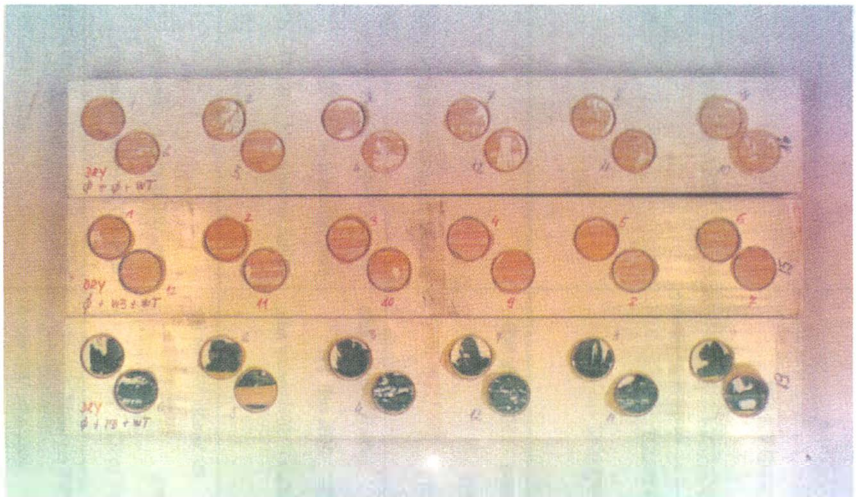


Figure 2. *Photograph of the surfaces tested dry for adhesion in pull-off test. Paler areas show wood failure, white areas show top-coat remnants, dark grey areas present base-coat deposit.* • *Fotografija ispitnih površina suhog prianjanja nakon testa otkidanjem kružića. Svijetle površine pokazuju lom po drvu, bijele su ostaci završnog naliča, a tamno sive su plohe slojevi temeljne boje.*

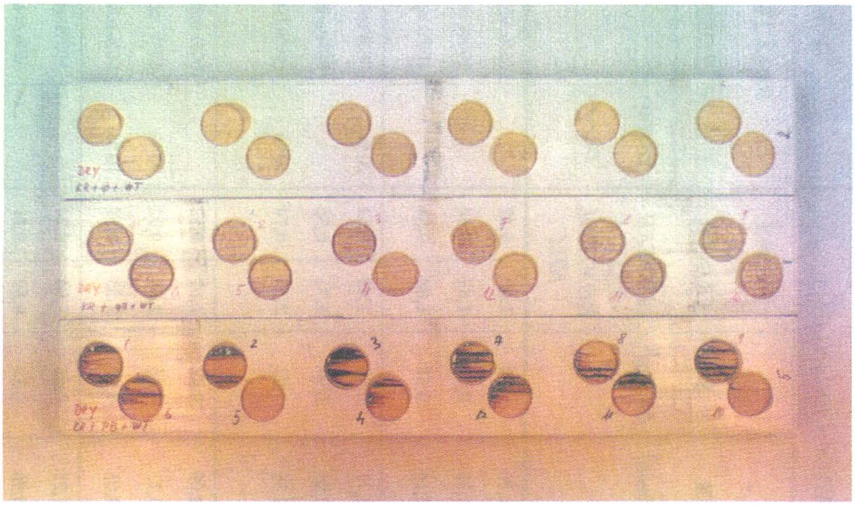


Figure 3. *Photograph of the surfaces tested dry for adhesion in pull-off test. Predominantly greyish surfaces depict failure at the border between the base-coat and primed wood surface.* • *Fotografija ispitnih površina suhog prianjanja nakon testa otkidanjem kružića. Prevladavajuće sive površine su posljedica loma na granici između slojeva temeljne boje i predpremazanoga drva.*

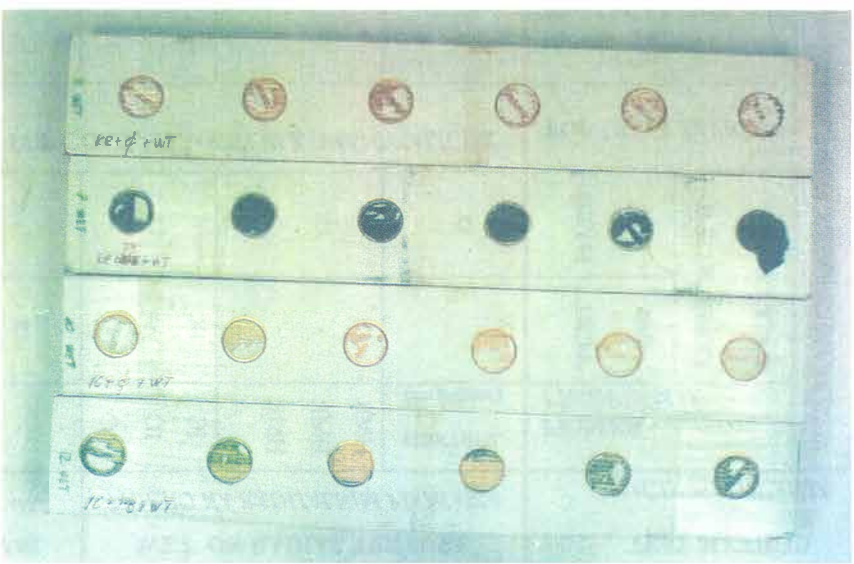


Figure 4. *Photograph of the surfaces tested wet for adhesion in pull-off test. Predominant base-coat failure on primed wood.* • *Fotografija ispitnih površina mokrog prianjanja nakon testa otkidanjem kružića. Prevladava lom u sloju temeljne boje nanešene na predpremazom obrađeno drvo.*

The poorest wet adhesion is achieved with WB basecoat (50 – 70% strength loss), somewhat better with hybrid basecoat, the best adhesion was again obtained with the direct application of the top-coat on wood (30-50 % strength loss). Primer-treated wood behaves in moist conditions better than non-treated substrate, increasing the control strength 1.5 – 2.5 times. The isocyanate primer was again yielding higher strength values and being somewhat more efficient than resorcinol HMR primer in preventing the reduction in wet strength. This was especially obvious with the HB basecoat.

Direct application of top-coat on wood yielded greater strength differences to control specimens in wet than in dry condition, further supporting the finding that the porous base-coat presents the weakest point in the system and creates the layer of greatest risk for water infiltration and swelling.

Strength loss and failure characteristics of tangential specimens clearly confirm that the adhesion on latewood shows the greatest reduction when wood is wetted (75 – 80 % strength loss). HMR primer proved efficient in improving the wet adhesion on latewood as in dry testing, but the overall values of wet strength tests and interface properties are still relatively poor (21 – 38 % retained dry strength).

Microscopic analysis

Micrographs of the tension-loaded zones near the paint surface of coated panels readily reveal the physical characteristics of the coating. A choice of microphotographs on figures 5 to 14 support the evidence about the physical behaviour of the coating system.

The two paint components, base-coat and top-coat, differ in their coating thickness on the spruce substrate (Fig 5) but also in appearance of their fractured transverse surfaces. The thin-layered water-borne base-coat is characterised by brittleness in its brash look on the fractured transverse surface (Fig 5). A number of small cracks and microvoids could be seen across the section of the base coat and it exhibits the brash and ground-looking layer near the wood surface (Fig. 6). The top-coat layer on the other hand fails in a tough mode and the fractured transverse surfaces are smooth (Fig. 5). Two separate coats of the top-layer are discernible neither on SEM micrographs nor in optical (UV or fluorescence light) microscopy, and the two top coats seem to have coalesced into one homogeneous layer (figures 5, 9 and 12).

The appearance of the base coat explains the relatively poor adhesion results and large proportion of the cohesive failure within this layer noticed on pull-off surfaces and commented earlier. This is particularly true for the results of wet adhesion testing, because it is easily perceivable that water ingress into the porous layer of the base-coat caused swelling and abatement.

The base coat covers the wood surface well and sticks to it, but the penetration is unexpectedly poor and only occasionally a cell or two beneath the surface in earlywood zones are filled with coating (Fig. 5 and 10).

The adhesion of the base coat to the genuine wood substrate is not sufficient (Fig. 6). The paint film sticks to the S3 layer and embed it (Fig 7) but does not penetrate the cell wall; the cell wall fracture shows characteristic radial agglomerations and the gen-

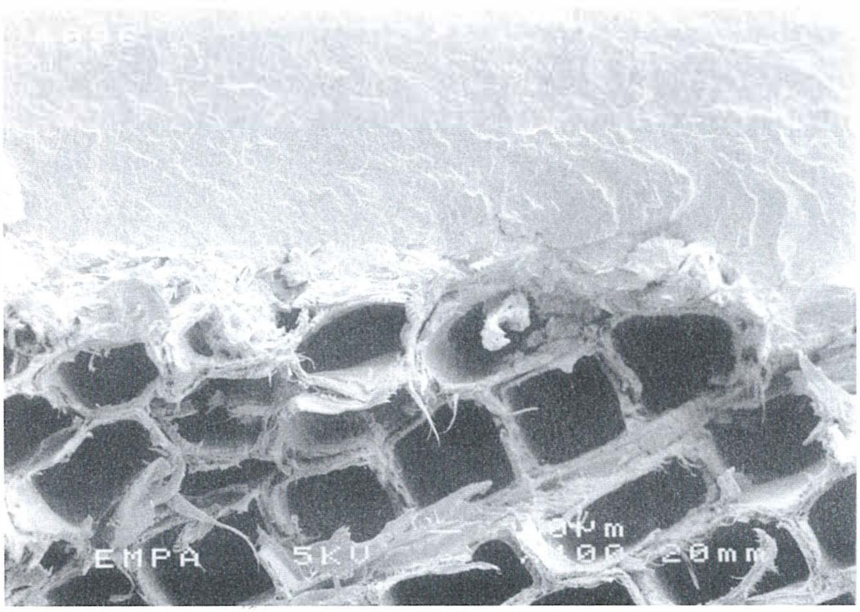


Figure 5. *Control specimen: fractured end-grain surface of the genuine, painted spruce wood. Thin, brash base-coat layer and thick top-coat layer are discernible. A number of microvoids in the base-coat layer is obvious. Two separate top coats present a coalescened homogeneous layer. 400:1 • Kontrolni primjerak: poprečna lomna površina smrekovine bojane bez predobrade. Razabiru se tanki, krti temeljni sloj i debeli završni nalič. Vidi se veći broj mikropukotina u sloju temeljne boje. Dva nanosa završnog naliča stopila su se u cjelovit sloj. 400:1.*

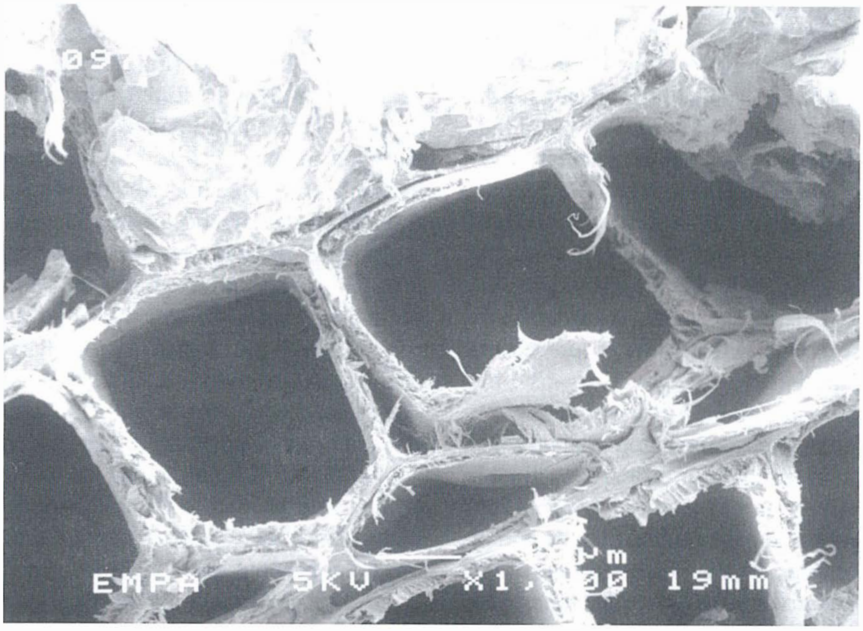


Figure 6.
Control specimen: fractured end-grain – surface of the genuine, painted spruce wood. Base-coat shows brash-like appearance. Earlywood tracheids fail in the same mode as unmodified wood. 1100:1 • Kontrolni primjerak: poprečna lomna površina smrekovine bojane bez predobrade. Sloj temeljne boje odaje krhka svojstva. Traheide ranog drva pucaju na isti način kao i neobrađeno drvo. 1100:1.

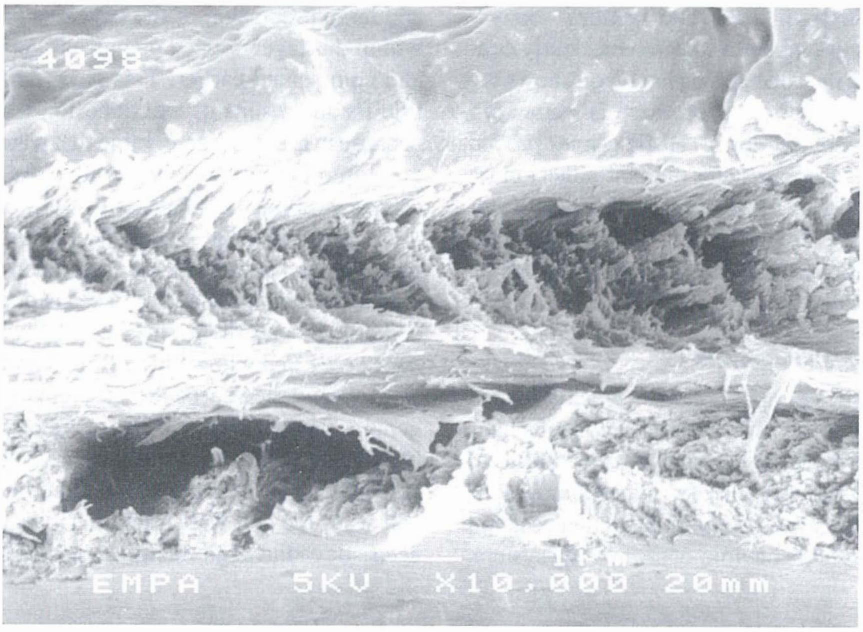


Figure 7.
Control specimen: fractured end-grain – surface of the genuine, painted spruce wood. Two adjoining cell walls show tough failure characteristics as in unmodified wood. Base-coat (upper) adheres good to the S3 layer of the upper cell wall. 10000:1 • Kontrolni primjerak: poprečna lomna površina smrekovine bojane bez predobrade. Dvije susjedne stanične stijenke pokazuju žilava lomna svojstva kao kod neobrađenog drva. 1000:1.

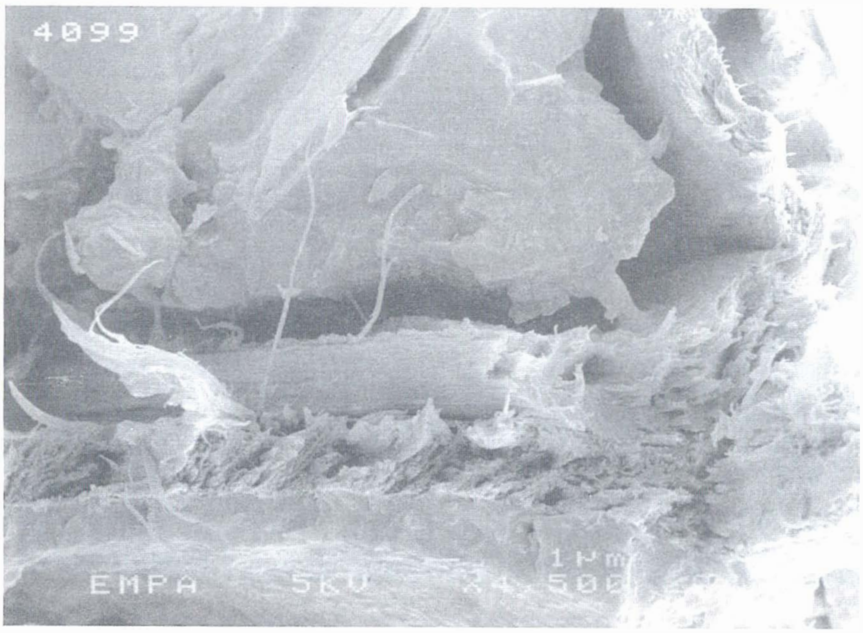
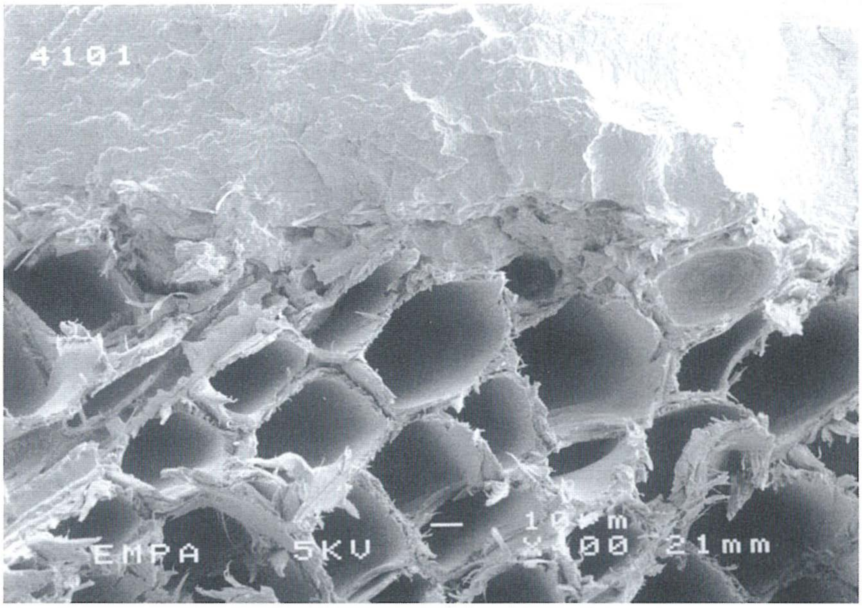
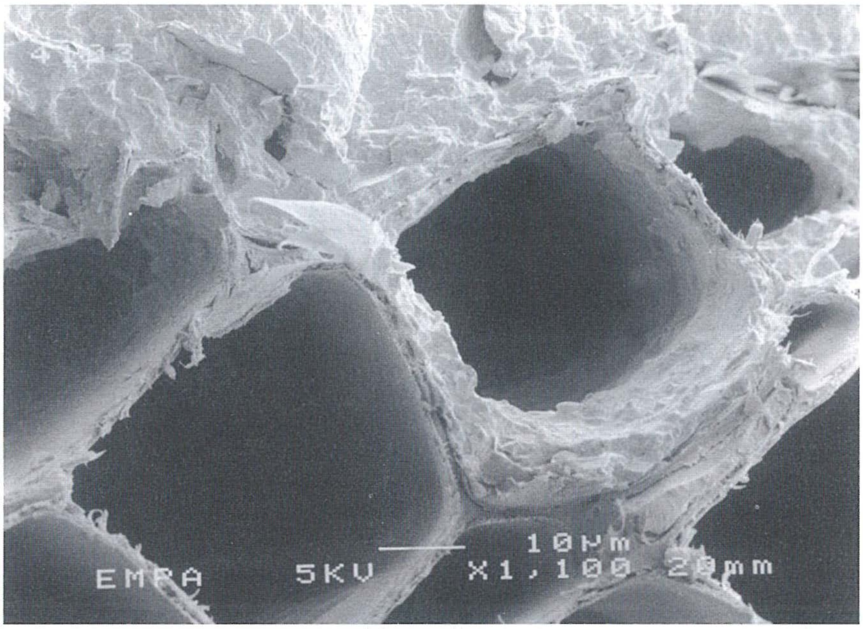


Figure 8.
Control specimen: fractured end-grain – surface of the genuine, painted spruce wood. Resin deposit within the lumen of the lower cell wall. Complete detachement of the paint in the upper lumen due to poor adhesion and/or testing stress. 4500:1 • Kontrolni primjerak: poprečna lomna površina smrekovine bojane bez predobrade. Naslage boje unutar lumena na doljnjoj staničnoj stijenki. Potpuno odvajanje boje u gornjem lumenu pokazuje ili slabo prianjanje ili(i) posljedice naprezanja pri lomu.





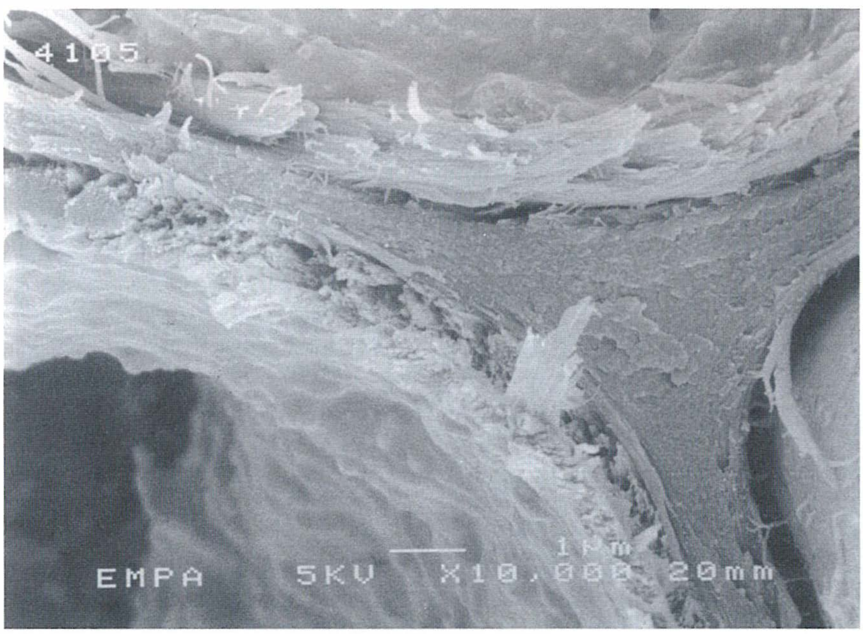


Figure 9.
HMR specimen: fractured end-grain – surface of the HMR primer impregnated and painted spruce wood. Earlywood failure mode closely resembles failure mode on control specimens. 400:1 • HMR uzorak: poprečne površine smrekovine obrađene rezorcinskim predpremazom i bojom. Vrsta loma ranog drva blisko sliči na lomne površine kontrolnih uzoraka. 400:1.

Figure 10.
HMR specimen: fractured end-grain – surface of the the HMR primer impregnated and painted spruce wood. Resin deposit within the lumen and fairly good attachement to the cell walls is obvious. 1100:1 • HMR uzorak: poprečne površine smrekovine obrađene rezorcinskim predpremazom i bojom. Vidljiva je naslaga smole u unutrašnjosti lumena i prilično dobro prihvaćanje uz staničnu stijenku. 1100:1.

Figure 11.
HMR specimen: fractured end-grain – surface of the the HMR primer impregnated and painted spruce wood. It is supposed that the upper lumen is filled with thick-layer forming base-coat, while the deposit in the lower lumen is primer's resorcinol resin. These two materials and the border between them can not be distinguished on SEM images. Structural appearance of the cell wall structure indicates that no penetration of the primer into the wall body, especially into S2 layer, has occured. 1000:1 • HMR uzorak: poprečne površine smrekovine obrađene rezorcinskim predpremazom i bojom. Pretpostavljamo da je gornji lumen ispunjem debeloslojnom temeljnom bojom, dok je naslaga u doljnjem lumenu rezorcinska smola predpremaza. Ova dva sloja tj. granica među njima se ne mogu raspoznati na SEM slikama. Strukturni izgled stanične stijenke naznačuje sda nije došlo do prodiranja predpremaza u staničnu stijenku, pogotovo ne u S2 podsloj.

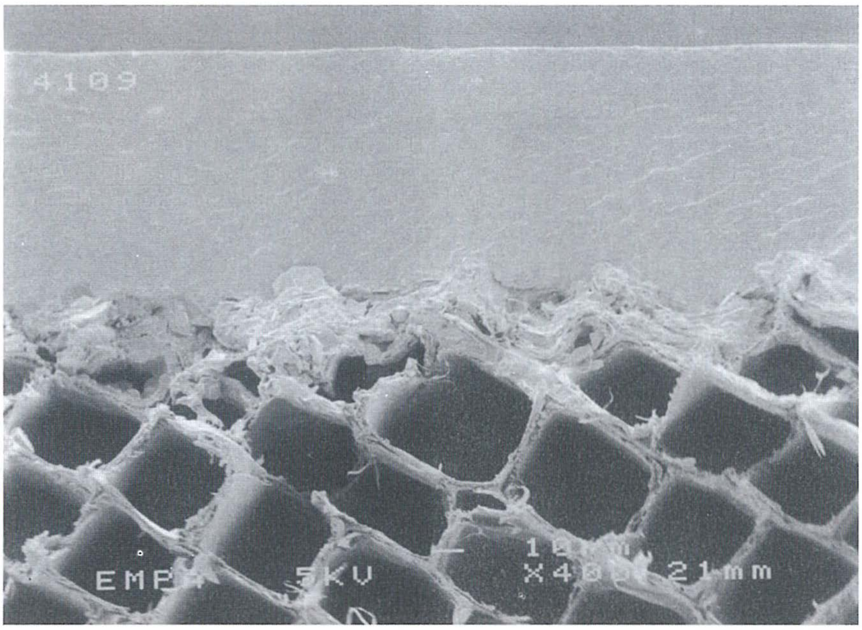


Figure 12.
IC specimen: fractured end-grain – surface of the the isocyanate primer-impregnated and painted spruce wood. Earlywood failure mode closely resembles failure mode on control and on HMR primer-impregnated specimens. 400:1 • IC uzorak: poprečne površine smrekovine obrađene izocijanatnim predpremazom i bojom. Način loma ranog drva blisko sliči onom na kontrolnim i na HMR uzorcima. 400:1.

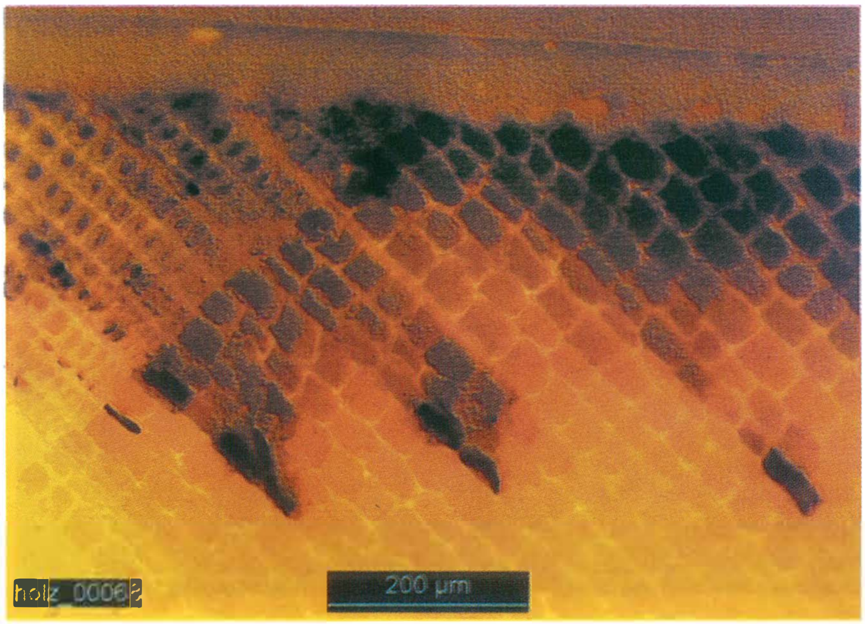


Figure 13.
Optical (reflected fluorescence light) micrograph of the end-grain surface of spruce wood impregnated with HMR primer and painted with water-based system. Deep penetration of the primer (dark areas) is obvious. Layers of the base-coat and two top coats could not be differentiated. • Optička mikrografija (odbijenim fluorescentnim svjetlom) površine poprečnog presjeka smrekovine obrađene HMR resorcinskim predpremazom i vodotopljivim sustavom boje. Jasno se vide tamne zone duboke penetracije predpremaza. Nije moguće razlikovati temeljni od dva sloja završne boje.

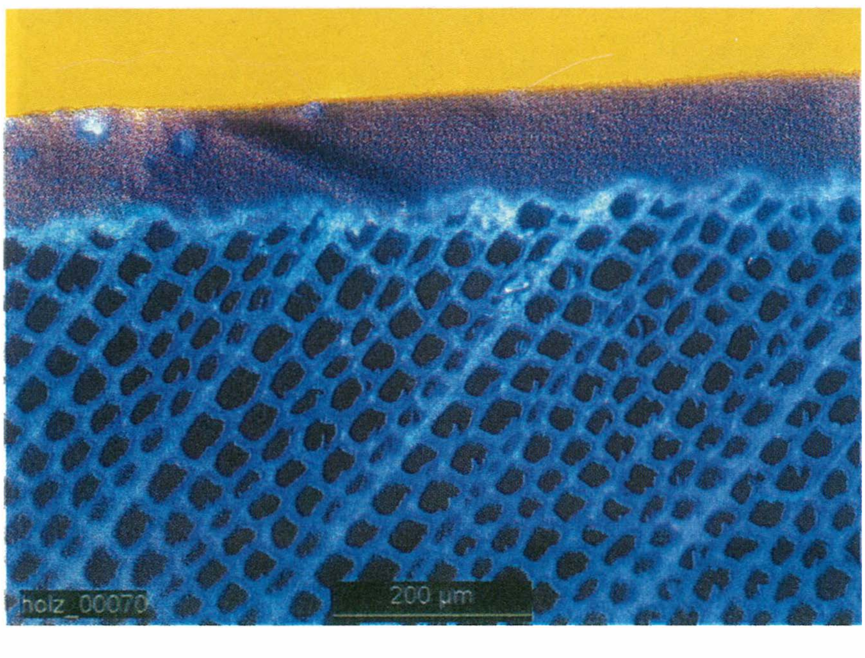


Figure 14.
Optical (reflected ultra-violet light) micrograph of the end-grain surface of spruce wood impregnated with isocyanate primer and painted with water-based system. Shallow penetration of the primer (glooming areas) can be seen. Layers of the base-coat and two top coats could not be differentiated. • Optička mikrografija (odbijenim ultra-ljubičastim svjetlom) površine poprečnog presjeka smrekovine obrađene izocijanatnim predpremazom i vodotopljivim sustavom boje. Može se vidjeti plitka penetracija predpremaza (sjajnija područja). Nije moguće razlikovati temeljni od dva sloja završne boje.

eral appearance of uncoated, unexposed wood (Fig. 7& 8). The water-borne base coat, however, seems to show signs of a weakening of the link with the wood at the interface (Fig. 6) where a series of cracks either in the wood or in the coating indicates the brittle character of the failure. The voids in a base-coat film are most probably due to the low spreading possibilities of the coat with such a high solids content (43 - 52 %, depending on the colour). It is also possible that some voids within the film, and some detachment of the base-coat from the wood cell wall happened during testing, where the stiff and brash coat showed brittle appearance and might have failed from wood (Fig. 8). There are examples, however, where the attachement of the base coat to the substrate seems very good (Fig. 7).

Generally the impregnating chemicals did not penetrate deeply into gross wood structure; and only in the earlywood regions some penetration occured beyond the surface layer of tracheids. More could be seen on the fluorescence micrographs (figs 13 and 14). The exception is the HMR resorcinol-phenolic primer, which coloured the deeper layer of the interface and whose traces could be seen even 10 tracheids from the surface (figure 13). The direct paths for impregnation penetration were the rays and they were often filled to a depth of several rows of tracheids from the surface and occasionally also a few neighbouring tracheids are filled in this process. Impregnating chemicals do not completely fill the lumina, but sometimes the interior cell wall surfaces are covered with a deposit of a resin (figures 10 and 11).

The consistent presence of radial agglomerations in S2 indicates that no cell-wall modification/penetration has taken place. Comparison of the figure 6 and 7 (control wood specimens, without impregnation) and figures 11 (HMR primer) and fig. 12 (IC primer) shows that the mode of failure of the substrate does not seem to be affected by the chemical treatment.

4. Conclusions
4. Zaključci

Impregnation of spruce wood prior to application of water-based acrylic and hybrid finishing system for exterior wood was performed using two types of primers. Resorcinol-phenolic aequous primer (HMR-primer), and solvent-borne isocyanate (IC) primer were tested for their effectiveness in improving the adhesion of base-coat in dry and in wet condition.

Both primers proved effective in improving the dry adhesion, although results did not show the best pull-off strength values. The reason mainly lays in the properties of the substrate which greatly influence the initiation and spreading of the cracks. The direct application of the top-coat (without base-coat) on wood, and especially on primed wood, proved advantageous in terms of dry adhesion.

Base-coat seems to be the weakest link in terms of wet adhesion. The water ingress into the micro-voids of the base-coat contributes to its swelling and abatement of its coherence. The application of the HMR and IC primers has improved the wet adhesion of subsequent coats because of the better spreading of the base-coat on the primed wood and better adhesion to a thin deposit of the primer. Wet adhesion of the top-coat, when applied directly on wood, was significantly better than the case with base-coat application.

FE-SEM analysis showed no indication whatsoever that any of the tested primers penetrated the cell wall, causing its physical modification. Combination of the adhesion testing and microscopic observation of the tested finishing systems proved very usefull, particularly as far as SEM is concerned. Microscopic analysis of the surface topography of fractured areas could support a better interpretation of the mechanical results of the pull-off adhesion testing.

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Obavjest

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