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ADHESION PROPERTIES OF WOODS TREATED WITH COPPER BASED WOOD PRESERVATIVE CHEMICALS

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ABSTRACT

In this study, four wood species, pine (*Pinus sylvestris* L.), beech (*Fagus orientalis* L.), sessile oak (*Quercus petraea* Liebl.) and sapelli (*Entandrophragma cylindricum*), commonly utilized in furniture industry and window and door manufacturing were impregnated with two copper based wood preservative systems (CCA and ACQ). After preservative treatments, wood surfaces were coated with three different varnish systems (polyurethane, acrylic and water-based). Effects of wood preservative chemicals on adhesion strength of varnish films were determined with a pull-out test technique according to the ASTM-D 4541. The highest adhesion strength was recorded with the polyurethane varnish system on sapelli sapwood surface. According to the results, the wood preservative systems used showed significant interference and reductions in adhesion strength of the varnish systems. In general, ACQ pretreated surfaces showed limited impact on adhesion strength when compared to CCA treated surfaces.

KEYWORDS: adhesion strength, wood preservative, impregnation, CCA, ACQ, varnish

INTRODUCION

With increasing use of wood for exterior applications the concerns about fungal degradation, insect attack or weathering lead to the treatment of wood with various wood preservatives or application of varnishes and paints for protection. In general, varnishes and paints can provide short term protection to untreated wood surfaces (Evans et al. 1992). As a result, these failed finishes cannot protect wood surfaces they applied on. It has been reported that chemical degradation of varnish layers has been followed by discoloration and fading on wood surfaces. According to previous literature, reapplications of finishing on these faded surfaces were also failed (Baysal 2004). UV light and surface temperature was considered as the major factors for discoloration and chemical degradation (Futo 1974, Ayati 2003). According to Grantham et al. (1976), varnish layers applied on wood preservative treated wood lasted 2-8 years under outdoor exposure (Gratham et al. 1976). When varnish applications are made on wood preservative treated wood surfaces, the service life of wood products should be expected much longer while improving aesthetic characteristics. The combinations of wood preservative chemical application followed by varnish finishing applications introduce new challenges for adhesion properties. Furthermore, compatibility issues between pre-treated wood surfaces and varnish films need to be assessed for optimal performance. Most of the literature in these area deals with interference of wood preservative chemicals with adhesives. The presence of contaminants such as wax, oil and inorganic materials hampers the development of cohesive adhesion bonds between wood substrate and adhesive (Lee at al. 2006). The insoluble metallic depositions of wood preservatives, for example, impede the formation of interfacial adhesion between wood and adhesives leading to a poor adhesive strength in products (Vick and Kuster 1992).

Previous literature reports that adhesion strength of applied varnish layers highly depends on varnish type and wood species while varnish film thickness and measurement type had limited effect. In general, hardwood surfaces showed higher adhesion strength than softwood surfaces and reactive varnishes (polyurethane, acrylic e.g) resulted in higher adhesion strengths (Budakci 2003). Untreated and CCA pre-treated pine and Douglas fir specimens were coated with varnish and exposed to outdoor conditions for two years. The varnish layer on the CCA pre-treated specimens reflected minimal effects of outdoor weathering and erosion. It has been reported that chromium played a key role in UV adsorption on CCA treated wood surfaces contributing increased degradation of resistance (Feist and Williams 1991). Varnish layers showed reductions in adhesion strength, hardness and weight when applied combined (varnish followed by wood preservative application) and exposed outdoors in East Black Sea region in Turkey. Changes in color and microscopic features have also been reported (Peker 1998). Another study highlights reductions in adhesion strength of synthetic stains and varnish layers applied on preservative treated wood surfaces (Sonmez and Budakci 2001).

Rain, humidity, UV radiation, and temperature fluctuations throughout seasons or days are considered major factors accelerating wood degradation. Wood protection against these factors requires proper preservative treatment, varnish application or a combination of both.

Recently, the concerns regarding environmental impact of combustion of preservative treated wood have been increased in European Union. It has been reported that characterization of Cr (approx. 16%), Cu (approx. 9%) and As (approx. 8%) as well as percent levels of Si, Ca, Fe, Al, magnesium and potassium in fly and residual ashes of openy burned CCA treated lumber. Comparison of fly ash and residual ash compositions suggests that while over 99% of Cr and Cu partitions to residual ash, between 11 and 14% of the As partitions to the fly ash. This supports with thermodynamic predictions which indicates higher air emissions of both trivalent and pentavalent arsenic (As) species (Wassen et al. 2005, Helsen and Bulck 2005)

Therefore, this study attempts to evaluate the adhesion performance of polyurethane, acrylic and water-based varnish films on four wood species, pine (*Pinus sylvestris* L.), beech (*Fagus orientalis* L.), sessile oak (*Quercus petraea* Liebl.) and sapelli (*Entandrophragma cylindricum*), pretreated with CCA and ACQ preservative chemicals.

MATERIAL AND METHODS

Wood material

Four different wood species, pine (*Pinus sylvestris* L.), beech (*Fagus orientalis* L.), sessile oak (*Quercus petraea* Liebl.) and sapelli (*Entandrophragma cylindricum*), commonly utilized in furniture industry and window and door manufacturing in Turkey, were used in this study.

Specimens were cut 110x110x12 mm in size from randomly selected first grade pine, beech, oak and sapelli sap wood lumber showing no spiral grain, knots, splits or discoloration with minimal variation in density according to Turkish Standard 2470 and ASTM D-358 (TS 2470, 1976, ASTM D-358, 2003) The specimens' annual ring orientation was perpendicular to the surface. The specimens were kept in a conditioning chamber at 20 ± 2°C and 65 ± 3 % relative humidity until they reached stable weight. Then the specimens were cut into final size of 100x100x10 ±1 mm before their surfaces were sanded with grit 80 and 100 sand papers, respectively. The sanded surfaces were cleaned with a soft brush and vacuum technique. A total of 360 specimens were prepared according to 4x3x3x10 experimental design, 10 specimen for each wood species, preservative chemical and varnish type, respectively.

Wood preservative chemicals

Two waterborne wood preservative chemicals, chromated copper arsenate (CCA) and ammoniacal copper quat (ACQ) were chosen in this study since they are commonly utilized by the wood protection industry in Turkey. Recent environmental concerns have resulted in a phase out process for the CCA thus increasing the ACQ's importance as a replacement or alternative wood preservative. The treating chemicals were representative of commercial material. Some chemical and physical characteristics of these preservatives are shown in Tab. 1.

Tab. 1: Ingredient compositions of the wood preservatives used

Preservative	Active ingredient	Composition (wt %)	pH value
ACQ	Copper oxide (CuO)	66.7	9.2
	Didecyldimethyl ammonium chloride (DDAC)	33.3	
CCA	Chromium oxide (CrO_3)	47.5	2.5
	Copper oxide CuO	18.5	
	Arsenic pentoxide (As_2O_5)	34.0	

Treatment schedule and target retention

The specimens were full-cell pressure impregnated by submerging them under the designated treating solutions in plastic trays in a vacuum-pressure retort and applying an initial vacuum of 6 KPa absolute pressure for 30 minutes, followed by 1060 KPa pressure for 60 minutes which resulting in an average of 90 minutes contact between wood specimens and the preservative chemicals. To achieve uniform distribution of preservative chemicals and avoid warping and cupping the specimens' cut ends were sealed with a two-component epoxy resin before the treatment. After the treatment, the specimens were removed from the retort and weighed to determine gross solution uptake. Retentions were calculated from the solution uptake and solution concentration. A preliminary study was performed to obtain water uptake characteristics of wood species which demonstrate wide range of anatomical features. This data is used to calculate solution concentrations. Target retention was 6.4 kg.m^3 for both preservative chemicals. The American Wood Preservers' Association (AWPA) recommends this value for furniture used in exterior environments above ground and ground contact exposures (Use Category System, UC-3B and 4A) for both, ACQ and CCA (AWPA 2006). Tab. 2 shows average preservative retentions for wood species used.

Tab. 2: Mean retentions (kg.m^{-3}) of wood samples treated (average of 10 specimens) (values in parentheses are standard deviation)

Wood Species	ACQ	CCA
Pine	6.5 (±0.4)	6.9 (±0.3)
Beech	7.1 (±0.9)	7.6 (±1.1)
Oak	5.9 (±0.8)	6.0 (±0.5)
Sapelli	5.7 (±0.6)	5.3 (±1.2)

The treated specimens were reconditioned in a conditioning chamber at 20 ± 2°C and 65±3 % relative humidity for five weeks.

Varnishes

Polyurethane, acrylic and water-based varnishes were used for coating application on the preservative treated specimens. Preparations and applications of the varnishes were made according to the manufacturer's recommendations and ASTM D-3023 standard (ASTM D-3023, 2003). Technical specifications of the varnishes and application systems used are given in Tab. 3.

Tab. 3: Technical specifications of varnishes and application systems used

Varnish type	pH	Density (g. m^{-3})	Solid content (% weight)	Amount applied (g. m^{-2})	Nozzle gap of spray gun (mm)	Air pressure (bar)
Polyurethane (filler)	5.95	0.98	48	125	1.8	2
Polyurethane (finishing)	4.01	0.99	44	125	1.8	2
Acrylic (filler)	4.3	0.95	42	125	1.8	2
Acrylic (finishing)	4.6	0.97	44	125	1.8	2
Water-based (filler)	9.30	1.015	34	67	1.3	1
Water-based (finishing)	8.71	1.031	32	67	1.3	1

Varnish amounts were determined with an electronic balance to the nearest 0.01 g and applied to the treated specimens' surfaces with a pressurized spray gun. The coated specimens were conditioned at 20 ± 2°C and 65 ± 3 % relative humidity for three weeks.

Determination of adhesion strength

The specimens were conditioned according to ASTM D-4541 at 23 ± 2°C and 50 ± 5% relative humidity for 24 hours prior to the adhesion strength test (ASTM D- 4541, 2003). Stainless steel experimental cylinders (20 mm in diameter) were attached to the conditioned surfaces at ambient temperature (20°C) to perform a pull-out test as outlined in the standard. A double component high strength epoxy with no dissolving effect on varnish layers was used 150±10 g.m^{-2} rate as specified in ASTM D-4541. The adhesion strength of varnish layers were determined with a standard adhesion device shown in Fig. 1.

Adhesion strength (X) was calculated in (MPa) according to the following equation.

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

F=Force at the time of failure (N)
d= diameter of experimental cylinder (in mm) (ASTM D-4541)

Fig. 1: Determination of adhesion strength with a standard test device

Statistical Analysis

MSTATC software package was used to evaluate data statistically. A multiple analyses of variation "ANOVA" test was used to examine the effects of wood species, wood preservative chemicals and varnish types and their mutual interaction on adhesion strength. Duncan test and LSD (the smallest important difference) critical values were used to determine the significant difference between the groups.

RESULTS

Adhesion strength results based on wood species, varnish type and preservative chemicals are given in Tab. 4.

Tab. 4: Mean adhesion strength values based on wood species, varnish type and preservative chemical (all values are MPa)

Factors ABC*	Polyurethane			Acrylic			Water-based		
	CCA	ACQ	Control	CCA	ACQ	Control	CCA	ACQ	Control
Pine	2.336	2.292	2.674	2.229	1.847	2.611	2.101	1.464	1.656
Beech	2.802	4.203	3.948	2.293	2.356	2.929	2.993	2.420	2.483
Oak	3.121	4.522	5.095	2.738	3.503	4.267	2.866	3.184	2.675
Sapelli	4.140	5.987	7.579	2.738	3.949	3.949	3.758	3.439	3.057

*: A: wood species B: Varnish type C: Preservative chemical

According to the results adhesion strength values differ based on wood species, varnish type and preservative chemicals. In order to determine individual and /or combined effects of these factors an ANOVA analysis was performed. Results of ANOVA test is given in Tab. 5.

Tab. 5: Multiple variance analysis for the effect of wood species, varnish type and preservative chemicals on adhesion strength

Source	Degrees of Freedom	Sum of squares	Mean square	F value	P value (a =0.05)
Wood species (A)	3	225.471	75.157	723.1294	0.0000*
Varnish type (B)	2	129.037	64.518	620.7692	0.0000
Preservative chemical (C)	2	32.417	16.209	155.9535	0.0000
Interaction (A*B)	6	46.217	7.703	74.1139	0.0000
Interaction (A*C)	6	20.895	3.483	33.5074	0.0000
Interaction (B*C)	4	51.061	12.765	122.8223	0.0000
Interaction (A*B*C)	12	22.243	1.854	17.8341	0.0000
Error	324	33.674	0.104		0.0000
Total	359	561.015			

* : significant

At α=0.95 confidence level, the effects of wood species, varnish type and preservative chemicals were found statistically significant. A detailed comparison of average adhesion strength values were given in Tab. 6 when wood species considered alone for LSD ±0.9458 value.

According to Tab. 6, the highest adhesion strength was recorded on sapelli while pine wood surfaces showed the lowest adhesion strength. The results of Duncan test when varnish types are compared are also given in Tab. 6 (LSD ± 0.8191).

While polyurethane varnish surfaces displayed the highest adhesion strength, the water-based coatings proved to have the weakest adhesion. When preservative chemicals are considered (LSD ± 0.0891), the Duncan test results illustrated in Tab. 6.

Tab. 6: A comparison of mean adhesion strength values for wood species, varnish type and preservative chemicals (all vales are MPa)

Factors		$\bar{x}$	HG	LSD±
Wood species	**Pine**	2,137	D	0.9458
	Beech	2,936	C	
	Oak	3,552	B	
	Sapelli	4,288	A*	
Varnish type	**Polyurethane**	4.060	A*	0.8191
	Acrylic	2.951	B	
	Water-based	2.675	C	
Preservative chemical	**CCA**	2.845	C	0.8191
	ACQ	3.264	B	
	Control (Untreated)	3.557	A*	

$\bar{x}$: mean value HG: Homogeneity group *: The highest adhesion strength value

According to Tab. 6, the highest adhesion strength was recorded on untreated (control) wood surfaces while CCA pre-treated wood surfaces demonstrated approximately 20% reduction in adhesion strength. ACQ pre-treated surfaces, on the other hand, showed approximately 8.5 % reduction which suggests a lower level interference with adhesion. Results of additional Duncan tests examined the interactions between wood species and varnish type, wood species and preservative chemicals, varnish types and preservative chemicals are given in Tab. 7, 8, and 9, respectively.

Tab. 7: A pair-wise comparison of adhesion strength values among wood species and varnish types used. (all values are MPa)

AB**	Polyurethane		Acrylic		Water-based	
	$\overline{x}$	HG	$\overline{x}$	HG	$\overline{x}$	HG
Pine	2.441	G	2.229	H	1.740	I
Beech	3.651	C	2.526	FG	2.632	F
Oak	4.246	B	3.503	CD	2.908	E
Sapelli	5.902	A*	3.545	CD	3.418	D
LSD± 0,1638						

**: A: wood species, B: varnish type
$\overline{x}$: Average value HG: Homogeneity group *: The highest adhesion strength value

Tab. 8: A pair-wise comparison among wood species and preservative chemicals. (all values are MPa)

AC**	CCA		ACQ		Control (Untreated)	
	$\overline{x}$	HG	$\overline{x}$	HG	$\overline{x}$	HG
Pine	2.229	I	1.868	J	2.314	I
Beech	2.696	H	2.993	FG	3.120	F
Oak	2.908	G	3.736	D	4.012	C
Sapelli	3.545	E	4.458	B	4.862	A*
LSD± 0,1638						

**: A; wood species, C; preservative chemicals
$\overline{x}$: Average value HG: Homogeneity group *: The highest adhesion strenght value

Tab. 9: A pair-wise comparison among varnish type and preservative chemicals (all values are MPa)

BC**	CCA		ACQ		Control (Untreated)	
	$\overline{x}$	HG	$\overline{x}$	HG	$\overline{x}$	HG
Polyurethane	3.105	D	4.251	B	4.824	A*
Acrylic	2.500	FG	2.914	E	3.439	C
Water-based	2.929	E	2.627	F	2.468	G
LSD± 0,1419						

**: B; varnish type, C; preservative chemical
$\overline{x}$: mean value HG: Homogeneity group *: The highest adhesion strength value

Tab. 10: A comparison summary of all three factors (wood species, varnish type and preservative chemicals (all values are MPa)

ABC**		Polyurethane		Acrylic		Water-based	
		$\overline{x}$	HG	$\overline{x}$	HG	$\overline{x}$	HG
Pine	**CCA**	2.536	R-U	2.229	TU	2.101	UV
	ACQ	2.292	STU	1.847	VW	1.464	X
	Control	2.674	N-R	2.611	O-S	1.656	WX
Beech	**CCA**	2.802	L-P	2.293	SU	2.993	K-N
	ACQ	4.203	EF	2.356	R-U	2.420	Q-U
	Control	3.948	FG	2.929	K-O	2.483	P-T
Oak	**CCA**	3.121	KL	2.738	M-Q	2.866	K-O
	ACQ	4.522	D	3.503	HI	3.184	JK
	Control	5.095	C	4.267	DE	2.675	N-R
Sapelli	**CCA**	4.140	EF	2.738	M-Q	3.758	GH
	ACQ	5.987	B	3.949	FG	3.439	IJ
	Control	7.579	A*	3.949	FG	3.057	K-M
LSD± 0,2837							

**: A; wood species, B; varnish type, C; preservative chemical
$\overline{x}$: Average value HG: Homogeneity group *: The highest adhesion strength value

Tab. 9 shows that the polyurethane coatings exhibited the highest adhesion performance on untreated wood surfaces while the water-based coatings showed approximately 49% lower strength on the same type of surface. A general comparison table including all factors (wood species, varnish type and preservative chemical) based on a Duncan test with a critical value of LSD± 0.2837 is given in Tab. 10. According to the results, the untreated sapelli control surfaces presented the highest adhesion strength while ACQ treated pine wood had the lowest.

DISCUSSION

According to the results obtained and statistical analysis performed, the wood preservatives used in this study resulted in reductions on adhesion strength of varnishes used for the wood species tested.

In general, the highest adhesion strength was obtained on sapelli sapwood surface while the lowest strength was recorded on pine sapwood surface. This can be attributed anatomical features of these wood species. Sapelli wood can provide better mechanical interlocking for the varnish applied due to its diffuse porous wood and larger tracheids. Pine sapwood, on the other hand, is lacking this homogeneous distribution and has different sizes of tracheids since it contains early wood and late wood bands (Panshin and de Zeeuw 1980).

When softwoods and hardwoods compared, pine wood showed lower adhesion strength. In general, pine surfaces also showed flaking after the pull-out tests regardless of varnishes applied (Fig. 2). These wood failures can be attributed to the relatively low density of pine wood.

Fig. 2: Varnish flaking and wood failures on pine wood surface

When the applied varnishes compared, the highest adhesion strength obtained from polyurethane varnish while water-based varnish displayed the lowest strength. These findings are found parallel with the previous literature indicating polyurethane varnishes are classified in reactive finishes. The reactive varnishes (polyurethane and acrylic) complete their chemical reaction on wood surface contributing towards better adhesion on molecular level (Budakci 2003). Water-based varnishes, on the other hand, are considered in coalescing finishes group which follows a different chemical pathway during the curing. This difference also makes the water-based varnishes prone to interference with wood preservatives chemical films on pre-treated wood surfaces. Similar findings were reported in the literature when polyurethane and water-based varnishes compared in terms of adhesion strength (Yakin 2001).

CONCLUSIONS

In this study, the highest adhesion strength was recorded in untreated control specimens while CCA treated wood species gave the lowest strength. This can be explained with negative interference between varnish layer and CCA treated surface. It has been reported that the lumen surfaces of CCA treated wood is completely covered with hemispherically shaped deposits of high surface energy chromium, copper and arsenic metallic salts (Tascioglu 2007, Tascioglu et al. 2004, Vick and Kuster 1992, Maldas and Kamden 1988 a, b). These nano scale deposition of metallic salts ranging in diameter from around 1.0 μm to essentially invisible at a magnification of 5000x theoretically causes very little opportunity existed for the varnish to make molecular-level contact with lignocellulosic constituents of cell walls without physical blocking by the chemical deposition. The ACQ treated surfaces, on the other hand, showed lower level of interference since ACQ is lacking chromium and arsenic and fallows a different chemical reaction pathway to be fixed with the wood constituents. In general, ACQ treatments showed a lower level of interference with the varnishes used.

In conclusion, when polyurethane, acrylic and water-based varnishes are considered for furniture and decorative applications sapelli, oak, beech and pine wood should be preferred respectively. In addition, the ACQ treated surfaces showed higher adhesion strength over CCA treated surfaces at 6.4 $kg.m^{-3}$ retention level indicating limited interference of ACQ chemical with the varnishes used.

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