STSM REPORT

Finishing process of thermally modified wood and resistance of coatings

COST Action: FP1006

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Poznan University of Life Sciences

STSM Scientific Report Contents:

1. Purpose of the STSM

The aim of the STSM was to establish collaborations between Poznan University of Life Science and the University of the Basque Country to develop new projects and perform significant contributions to achieve the COST Action goals.

The specific purpose of this STSM to Poznan University of Life Sciences was to obtain theoretical and practical knowledge about finishing thermally modified wood with lacquer products, testing procedure and data analysis.

In addition to the obtained results during the STSM, complementary experiments are currently ongoing at the home institution in order to prepare a joint publication and establish basis for other studies.

2. Description of the work carried out during the STSM

Task 1: The outlines of the research were determined.

- Before start the STSM the wood thermally modified in Spain was shipped to the host institution to be conditioned.
- Comparison of thermally modified samples in Spain and Poland at industrial level, treatment features.
- Acquisition of knowledge on finishing of wood with UV lacquers and properties of lacquer coatings.
- To select appropriate application methods in technical centre of Sherwin-Williams Company and preparation of lacquer products in laboratory conditions.
- Determination of the appropriate tests to measure physical properties and resistance to mechanical, thermal and chemical factors.
- To prepare a plan for a future collaboration through research projects.

Task 2: Thermally modified samples and substrate preparation

The thermally modified samples were chosen based on availability and diversity of treatment conditions. On these grounds were used samples of Ash (*Fraxinus excelsior*) and Monterey pine (*Pinus radiata*), also in some specific tests were used samples of European beech (*Fagus sylvatica*). The features of samples and coating application types are described in the table 1.

Table 1. Thermally modified samples and type of finishing

Wood species	control	192[1]	200[2]	210[3]	**Finishing type** Brush[4]	Rollers[5]
Pine	•			•	•	•
Ash	•	•	•	•	•	•
Beech	•				•	

[1,2] Thermo treated in Poland in sawmill-Stefan company; [3] Thermo treated in Spain in Termogenik company
[4] Finishing of wood in laboratory conditions; [5] Finishing of wood in Sherwin-Williams Company

Before lacquer finishing is necessary to prepare the surface in order to improve adhesive bonding of wood. In laboratory was used silicon carbide (SiC) sandpaper with abrasive grain of 280, on the other hand, in Sherwin-Williams Company the samples were sanded with an orbital sander (abrasive grain of 320) and with a system t7 1350 planetary unit. The sanding process also was carried out after application of initial coat layer to enhance the lacquer adhesion.

Task 3: Finishing process: Applications of lacquer products

Thermally modified wood has been developed in order to improve some properties and thus extend the range of uses and the service life of modified products. The modified wood presents significant changes in surface properties and water-substrate interactions which affect application methods and curing, especially with water based products.

For the experiments different kinds of lacquer products and two different application systems were used. Table 2 summarizes features of products as well as parameters used for application in laboratory. In addition Table 3 contains a number of features of the products and application at industrial scale.

Table 2. Principal features of coating products and it parameters

Chemical group	Lacquer designation	Target	Gloss level	Giscode[1]	Components	Density (g/cm3)	Viscosity flow (DIN53211)	Application amount (g/m²)	Layer
Solventborne	OLI-KS Parkettsiegel 7600	Surface resistence	semi matt	KH1	Oxidative dryer, Alkyd resin, additives	0.884	25-35 s (4mm)	100	1st
Waterborne	OLI-AQUA TOP Parkettsiegel 51.20	Surface protection	semi matt	W2/DD+	Polyurethane disperser, water,additives	1.04	20-23s (4mm)	100	2nd-3rd
Waterborne hardener	OLI-AQUA Härter 13.1	Curing agent	-	-	-	-	-	10	2nd-3rd

[1] Gefahrstoff-Informationssystem der Berufsgenossenschaft der Bauwirtschaft

The lacquer mixtures were prepared following the producers recommendations. Lacquers were applied with a paintbrush in 3 layers, first layer was a solvent borne lacquer, and second as well as third layer was a waterborne product with a hardener. The thickness of coatings layers were measured with a disc micrometer in different points and the layer thickness average was calculated. Between layers applications the samples were dried according to the manufacturer's instructions and then conditioned in laboratory before the beginning of the tests.

Table 3. Principal features of coating products and it parameters at industrial scale

Chemical group	Lacquer designation	Target	Gloss level[1]	CEPE code[2]	Components	Density (g/cm³)	Viscosity (mPa)	Application amount (g/m²)	Layer
UV	Beckry Seal UL1117	UV Sealer	-	4	mixture	1.12	2700-330	20	1st
UV	Beckry Clear UM1115-0012	Industrial UV Clear Top	6-7	4	mixture	1.18	720-980	5	2nd
UV	Beckry Clear UM1115-0013	Industrial UV Clear Top	6-7	5	mixture	1.19	720-980	5	3rd

[1] Gloss Units measurement angle 60°; [2] European Council of Paint, Printing Ink and Artists' Colors Industry

In addition to other technologies, UV curable products were used at industrial scale in Sherwin-Williams Company. These products have an important role in coating industries, especially as a mean of overcoming solvent emission limitations. The UV curable lacquers were applied with roller coating machine (BÜRKLE system) in 3 layers, the first layer was a UV sealer product followed by UV curing process (LED bulbs). The second as well as the third layer was UV top sealer followed by UV curing process that combined LED bulbs with low pressure UV-C lamps.

Task 4: Curing and drying process

The next phases of coating process are drying and curing of samples. The drying process is used to eliminate solvents and water from the surface; and the curing process is used for polymerization of compounds to improve adhesion, breaking molecules and creating free radicals from the photoinitiators. The characterizations of drying and curing in different coats and the differences between methods are summarized in the Table 4.

Table 4. Characterization of drying and curing process in coats

	Coat	Chemical group	Lacquer designation	Application method	Drying			Curing		
					Temp. (°C)	RH[1] (%)	Time (h)	UVLamp (nm)	UV-dose (W/cm)	Speed (m/min)
A	1st	Solvenborne	OLI-KS 7600	Brush	20	50	18	-	-	-
	2nd	Waterbone	OLI-AQUA TOP 51.20		20	50	5-6	-	-	-
	3rd	Waterborne hardener	+ OLI-AQUA Härter 13.1		20	50	200	-	-	-
B	1st	UV low yellowing sealer	UL1117	Roller coating machine	-	-	-	Hg 280-320	80	10
	2nd	UV Clear Topcoat	UM1115-0012		-	-	-	Hg 280-320	80	10
	3rd				-	-	-	Ga 390-450	120	15

[1] Relative Humidity; **A**: Application in laboratory conditions; **B**: Application in Industry

Nowadays the coatings formulators are looking for ways to reduce the levels of volatile organic compounds maintaining high performance levels. The solvent based formulations are commonly used, however are being substituted by waterborne coatings which are considered safe and of efficient use for applicators together with UV curing systems which are formulated with very small amounts of solvents or even without any solvent at all.
In the curing process with UV-lamps and UV-LED technology, the development of the photoinitiators, lacquer formulations and UV-curing technologies, nowadays allows the application by roller coating and the use of 100% UV-Lacquers. Figure 1 shows stages industrial coating process.
The experiments were carried out in a wood-finishing line using a hybrid line mix of LED and arc lamps. The radiation used in the base coat was between 280-320 nm with mercury lamps for the cross-linking process, and in the topcoat were used special Gallium's UV-lamps in combination with medium pressure lamps in the radiation range of 390-450 nm.

Figure 1. Stages in industrial coating process. From left to right sanding, applying, curing and Mercury lamps

Task 4: Experimental test methods performed in the host institution.
After experiments on thermally modified wood samples several tests were performed in order to analyze obtained properties in wood after lacquering with products of different chemical groups using two application methods. The list of the tests performed is the following:

- The thicknesses of obtained coatings were determined with disc micrometer (0.1 µm) measuring in 10 points after each coat layer.
- Evaluation of resistance to selected mechanical factors: –Abrasion test was made with the TABER ABRASER 5130 apparatus model 352 according to the procedure presented in PN-88/F06100/04 standard, using strips of S-33 sanding paper. Records of mass loss (0.0001 g) were made after each 50 rotation until 200 rotations. – Impact test was performed according to the procedure described in PN-93/F-06001/03 standard using a prototype device PUD-6 (DOZAFIL POLIFARB Wrocław, Inc., Poland). Five tests were conducted for the following heights of weight's fall: 10, 25, 50, 100, 200, 400 mm and was done evaluation of failures. – Scratching resistance test was completed with Clemen's apparatus according to the PN-65/C-81527 standard. Samples were placed on device's table loading the graver within the range 1000, 1500 and 2000 g and moving it at the velocity about 40 mm/s. The measurements of scratched width were done using a microscope.
- Surface gloss characterization: – Values of wood coating gloss were done with photoelectric method using PICO GLOSS, model 503 (Erichsen firm). In accordance with DIN 67 530, ISO 2813 and PN-88/F- 06100/02, 10 measurements in parallel sense to wood fibers and 10 perpendiculars to the wood fiber of each sample were taken at 3 angles of incidence of the light 20°, 60° and 85°.
- Evaluation of adherence of coatings to substrates: – The adherence tests were performed with the pull-off method, basing on guidelines EN 24624 which allow determination of the minimum tension stretching necessary to the tear off the coating at the exertion of loading in perpendicular toward direction to the substrate. Aluminum stamps were glued to the surface of coatings with 2K silane-epoxy adhesive about the trade name Jowat 690.00. After conditioning, the adherence in testing-machine SCHOPPER of the ZDM 2.5/91 type set on the range 500 daN were determined. The velocity of loading caused delaminating in the system and the pull off force was determined. Each delaminated sample and stamp was evaluated in visual mode.
- Measurements of surface wettability: – Surface contact angles of lacquer coatings were conducted using distilled water as wetting liquid, in compliance with the procedure standard PN-EN 828. Water was applied with the chromatographic syringe (sessile drop) at volume 3.5 µl. Measurements of contact angle were taken (10 per sample) using an adapted microscope to the goniometric equipment.
- Evaluation of surface hardness: – A Persoz pendulum hardness tester was used to monitor the surface hardness of the coats films on glass. The hardness of coat surface was measured with respect to the pendulum oscillation time (at 20°C and 50% R.H). The Persoz pendulum consists of a triangular open framework with an adjustable counterpoise weight. The pendulum pivots on two bearings of 5 mm diameter that rest on the test surface. – The surface hardness of the coats films on wood was measured by Rocker method following the principles of oscillation in surface and time of oscillation (ASTM D 4366).

- Evaluation of resistance to selected thermal factors: – Resistance to high temperature *dry heat* test was done according to PN-EN 12722 at cycles of 20 min and increasing temperature until visible failures or color-off appeared. The block was taken off and the samples were conditioned during 24 h. Finally the evaluation of surface coatings quality was conducted using a numeric scale. – Resistance to high temperature *wet heat* test was done according to PN-EN 12721 with the specifications described above but with the block placed on fabric moist instead of direct contact. The fabric was from polyamide fiber of weight 50 g/m^2. – Resistance to steam action was tested according to PN-88/F-06100/06. The samples were put on the holes of a tank's cover filled with boiled water. Samples were set to the steam action during 1 h. After 24 h the surfaces were evaluated.

- Research on metameric effect: Metamerism of samples was performed in artificial conditions in a Minispectra chamber with multispring-system to compare and make estimations of visible features of surface after several cycles of thermal wet aging, according with 88/F-06100/07 standard.

3. Description of the main results obtained

3.1 Thickness of obtained coatings

Table 5 shows two different application methods at laboratory scale and at industrial scale. Several products with different chemical groups were tested and the thickness of each layer was measured.

Table 5. Coating methods and thickness of layers

Finish type	Product	Coat	Pine[1] (mm)	TI (µm)	P210[2] (mm)	TI (µm)	Ash[3] (mm)	TI (µm)	A192[4] (mm)	TI (µm)	A200[5] (mm)	TI (µm)	A210[6] (mm)	TI (µm)
BRUSH	Substrate	0	22,334 ±0.264		18.294 ± 0.173		20.098 ±0.023		19.957 ±0.027		19.884 ±0.074		21.297 ±0.031	
	Solvenborne	1st	22.414 ±0.333	80	18.303 ±0.052	90	20.184 ±0.017	86	20.021 ±0.026	64	19.917 ±0.066	33	21.344 ±0.021	47
	Waterborne	2nd	22.426 ±0.263	130	18.353 ±0.066	50	20.247 ±0.012	63	20.064 ±0.017	43	19.975 ±0.066	58	21.402 ±0.012	58
	Waterborne	3rd	22.534 ±0.329	108	18.373 ±0.004	20	20.301 ±0.026	54	20.116 ±0.011	52	20.037 ±0.069	62	21.448 ±0.021	46
	Total thickness	0-3rd		200		79		203		159		153		151
ROLLER	Substrate	0	-	-	18.621 ±0.181		21.286 ±0.158		21.162 ±0.137		21.330 ±0.033		21.271 ±0.080	
	UV Topcoat	3rd	-	-	18.678 ±0.120		21.351 ±0.064		21.203 ±0.128		21.372 ±0.024		22.306 ±0.046	
	Total thickness	0-3rd	-	-		57		65		41		42		35

TI= Thickness Increase; [1] ρ=588.926±72.958; [2] Pine 210°C ρ=486.453±26.132; [3] Ash 192°C ρ=695.436±79.691; [4] Ash 200°C ρ=696.839±78.116; [5] Ash 210°C ρ=626.533±27.135

3.2 Evaluation of resistance to selected mechanical factors

- Resistance of lacquer coatings to Abrasion test.

The abrasion experiments in two finishing systems in thermally treated wood and in control samples are showed in the figure 2. The number of cycles and the mass loss during the abrasion test showed significant differences between application systems, and unusual results were found in thermally modified wood at 192°C.

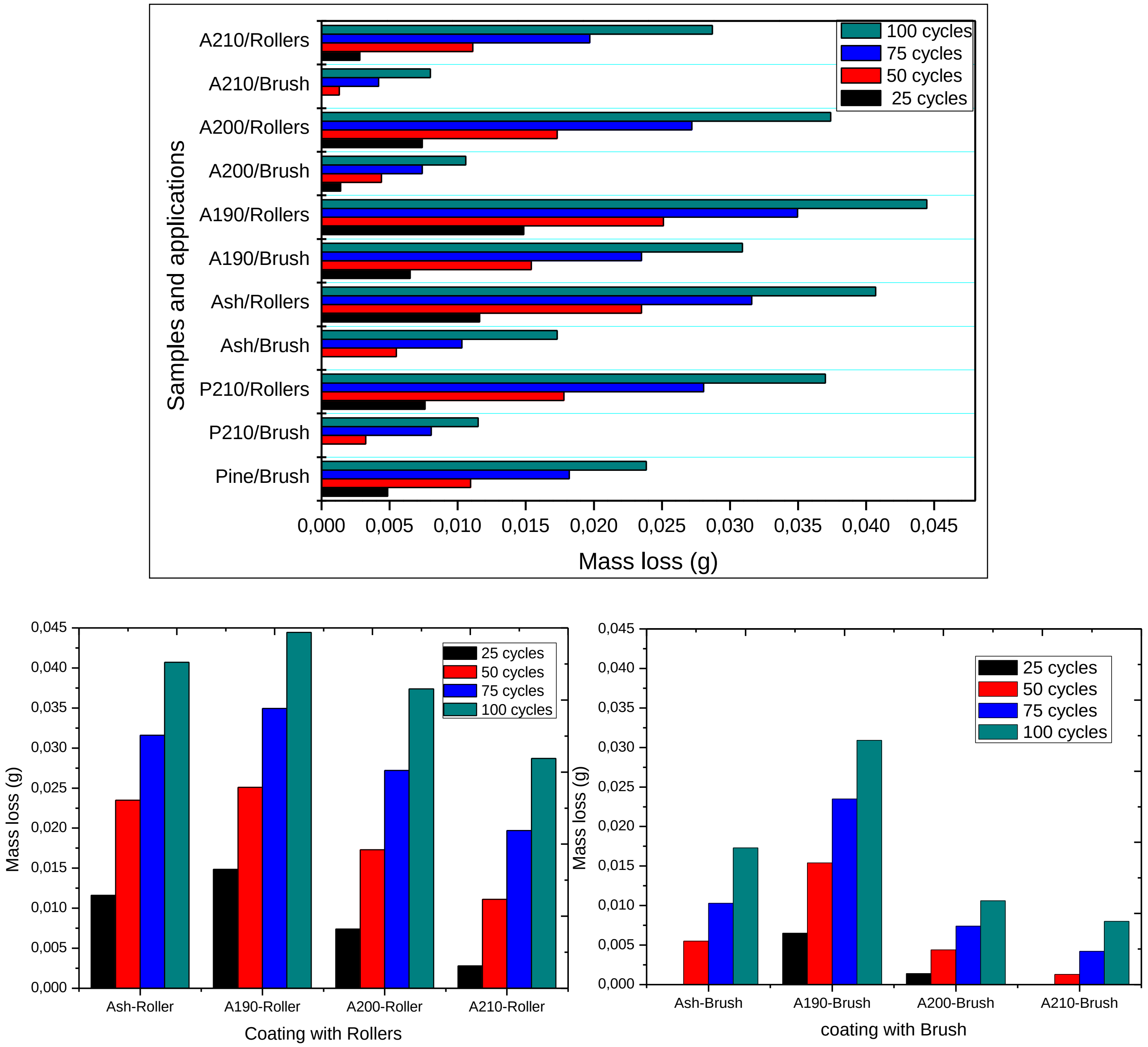

Figure 2. Results of resistance of lacquer coatings to Abrasion test. The figure in the top shows all samples and in the bottom shows tests with ash

– Resistance of lacquer coatings to impact and scratching resistance test.

The visual outcomes of mechanical tests are shown in the figure 3 and are ordered by wood specie and treatments. These images clearly showed distinct patterns between applications and admissible values of scratch test. Analyzing the results of impact resistance test of each lacquer coatings, a similar resistance for each group of coatings and a slight improve in UV lacquers were found.

3.3 Surface gloss characterization.

Results of gloss in lacquer coatings as well as the kind of denomination are shown in table 6. In addition, figure 3 represent the gloss values in the different coating types and the standard deviation.

Fundamentally the measured coatings in perpendicular to grain orientation were classified as half gloss. On the other hand, coatings measured in parallel to grain orientation showed diverse results.

Table 6. Gloss values and denominations

Sample	Incidence angle (°)						Degree of gloss[1]		Gloss type[2]	
	Perp. to grain			Parallel to grain						
	20	60	85	20	60	85				
Ash	1.54	1.47	1.08	8.86	15.5	5.86	<10	[10-35]	*Mat*	*Half mat*
Ash brush	37.63	37.87	72.1	69.75	84.15	81.53	[35-60]	>80	*Half gloss*	*Brillant gloss*
Ash roller	1.44	2.35	2.97	12.27	17.44	8.19	<10	[10-35]	*Mat*	*Half mat*
Ash 190°C	0.3	0.24	1.69	1.21	6.87	1.89	<10	<10	*Mat*	*Mat*
Ash 190 °C brush	36.81	35.75	69.66	66.32	86.48	80.78	[35-60]	>80	*Half gloss*	*Brillant gloss*
Ash 190 °C Roller	0.65	0.56	6.11	5.00	13.00	3.85	<10	[10-35]	*Mat*	*Half mat*
Ash 200°C	0.2	0.2	1.5	1.04	6.64	1.81	<10	<10	*Mat*	*Mat*
Ash 200 °C brush	35.36	32.04	69.37	63.87	84.51	77.33	[10-35]	>80	*Mat*	*Brillant gloss*
Ash 200°C Roller	0.61	0.59	7.47	5.8	17.47	4.55	<10	[10-35]	*Mat*	*Half mat*
Ash 210°C	0.4	0.32	3.24	2.13	16.04	5.88	<10	[10-35]	*Mat*	*Half mat*
Ash 210°C brush	31.75	25.3	67.7	54.42	84.65	70.93	[10-35]	>80	*Half mat*	*Brillant gloss*
Ash 210°C Roller	0.48	0.46	5.58	4.71	11.72	3.9	<10	[10-35]	*Mat*	*Half mat*
Pine	1.08	1.04	4.33	3.83	7.03	1.83	<10	<10	*Mat*	*Mat*
Pine brush	35.51	29.93	72.54	62.02	85.53	74.4	[10-35]	>80	*Half mat*	*Brillant gloss*
Pine 210°C	0.56	0.52	3.88	2.76	8.27	3.14	<10	<10	*Mat*	*Mat*
Pine 210°C brush	26.84	23.84	65.28	60.97	81.64	68.78	[10-35]	>80	*Half mat*	*Brillant gloss*
Pine 210°C Roller	0.61	0.6	6.64	5.48	16.06	4.08	<10	[10-35]	*Mat*	*Half mat*

[1] Degree of gloss of samples at incidence angle of the light 60°; [2] Gloss estimation in perpendicular and parallel measure

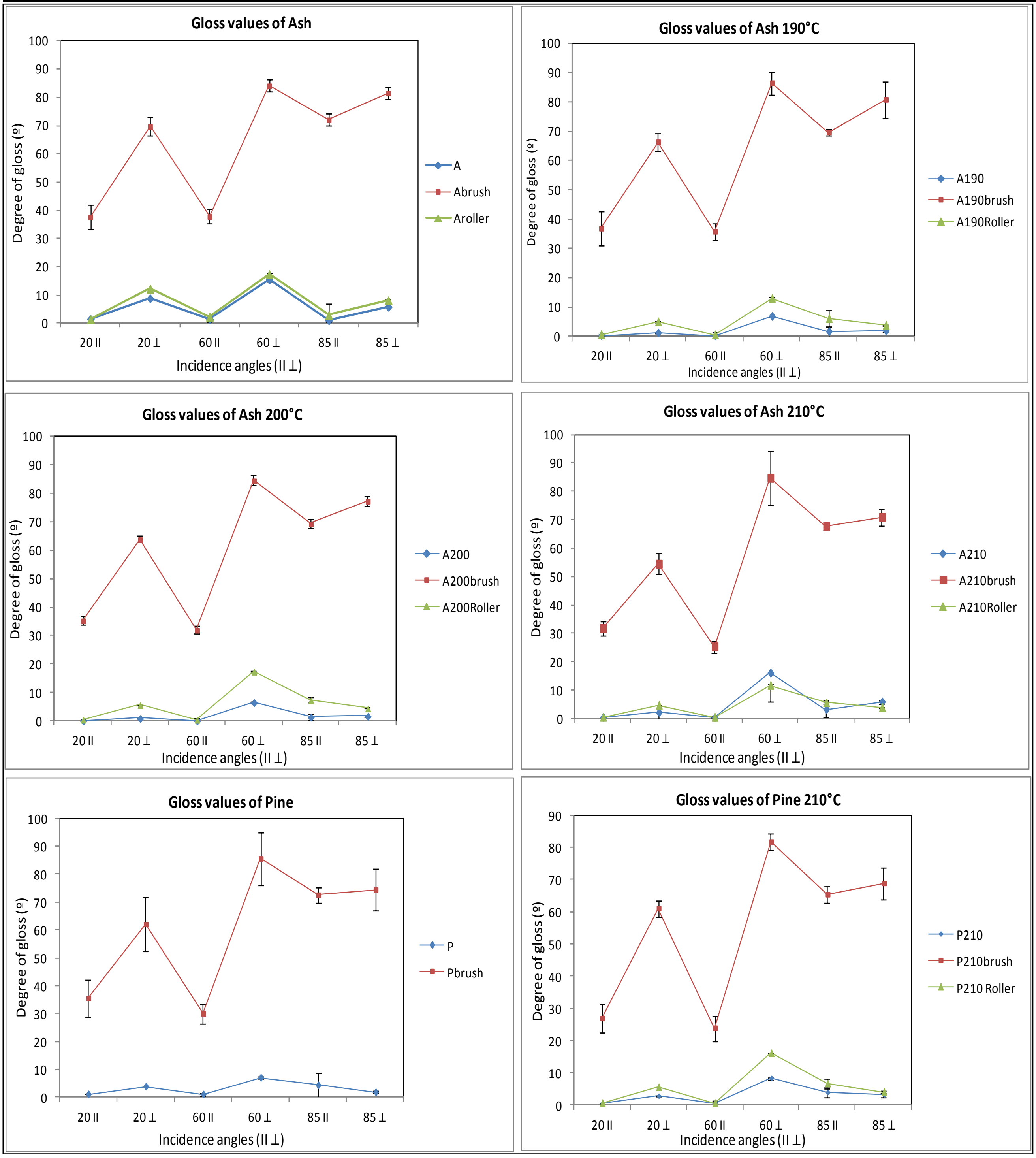

Figure 3. Gloss values in different coating-substrate and the standard deviations

3.4 Evaluation of adherence coatings- substrates.

A visual Analysis was done after the pull-off test. The table 7 shows the statistic values and the assigned delamination type in the adherence range. It was observed various shares of particular failure types at ultimate load. The coating system changes delamination type; and comparing coated control samples (not thermally-treated) with thermally-treated coated samples it was found out that the adherence is improving depending on the number of coats (layers) and temperature parameters.

Table 7. Evaluation of adherence coatings- substrates

SAMPLE	PULL OFF (MPa)			Surface Peel Off[4]									Stamp[5]
	m[1]	X_{MAX}[2]	X_{min}[3]	*A*	*A/B*	*B*	*B/C*	*n*	*n/m*	*-/Y*	*Y*	*Y/Z*	
ABrush	0.77	0.88	0.58	10	-	-	-	-	-	90	-	-	-
ARoller	0.857	0.99	0.76	-	-	-	-	-	-	62.5	2.5	-	42.2
A192°C Brush	0.82	0.88	0.74	7.14	2.86	-	-	-	-	90	-	-	-
A192°C Roller	0.924	1.02	0.86	24.29	3.57	-	-	-	-	15.71	9.29	-	47.14
A200°C Brush	0.81	0.87	0.72	20	75.71	-	-	-	-	4.29	-	-	-
A200°C Roller	0.913	1.02	0.87	30.71	-	-	-	-	-	-	7.86	-	61.43
A210°C Brush	0.86	0.96	0.77	57.14	24.29	-	-	-	-	15.71	-	-	-
A210°C Roller	0.934	1.06	0.82	37.86	-	-	-	-	-	-	-	5	57.14
PBrush	0.77	0.94	0.61	2.86	4.29	-	-	-	-	83.571	-	-	-
P210°C Brush	0.76	0.85	0.61	60	15.71	-	-	-	-	24.29	-	-	5.4
P210°C Roller	0.84	1.09	0.7	33.57	-	-	-	-	-	-	18.57	-	47.86

[1]Average;[2]Maxim value;[3]Minimum value;[4] Delamination type according to EN 24624;[5] Delamination on stamp

3.5 Measurements of surface wettability and surface hardness.

A study of the wettability and hardness of the different lacquers applied on glass and wood substrates was carried out. The study includes values for each separate coating layer, and adhesion between two and three layers (table 8 and table 9). Regarding these data is possible to obtain all feasible lacquer-substrate combinations and enhance the application system and technological parameters such as the application pressure and lacquer product quantity.

Table 8. Surface wettability and surface hardness

Layers	product	**Pressure** (MPa)	Hardness[1]	Wettability[2]		
				Glass	**Pine Wood**	**Beech Wood**
I	(1)Solvenborne	120	73.27	73.27	71.37	72.95
I	(1)Solvenborne	90	68.23	68.23	73.24	73.30
II	(2)Waterbone	120	70.07	70.07	63.20	65.33
II	(2)Waterbone	90	66.59	66.59	60.10	62.12
III	(1)+(2)	120	63.75	63.75	60.57	63.23
III	(1)+(2)	90	63.72	63.72	61.31	61.18
IV	(1)+(2)+(2)	120	62.99	62.99	60.14	60.19
IV	(1)+(2)+(2)	90	63.01	63.01	71.37	60.74

[1] Persoz pendulum cycles;[2] Wettability by contact angle

The results of wettability on wood surface are diverse, and it cannot be concluded which system is better so far. To obtain additional knowledge of interactions that occurs on the surface of the wood with applying lacquers, some experimental trials in the home institution are ongoing using techniques of high-resolution microscopy and X-ray.

Table 9. Evaluation of hardness-wettability on wood surface

Application method	SAMPLE	Hardness[1] II	Hardness[1] ⊥	Wettability[2]
BRUSH	pine	15.66	17.5	63.90
	P210	19	19	63.79
	Ash	18.38	16.72	58.99
	A192	15.66	15.33	62.67
	A200	15.66	16.17	60.10
	A210	16.83	15.17	55.11
INDUSTRIAL ROLLER	P210	25.17	25.28	63.30
	Ash	25.33	25.84	62.97
	A192	26.78	26.33	61.29
	A200	27.92	28.08	41.41
	A210	28.67	29.22	63.28

[1]Rocker cycles. Perpendicular to grain (II) and Parallel (⊥);[2] Wettability by contact angle

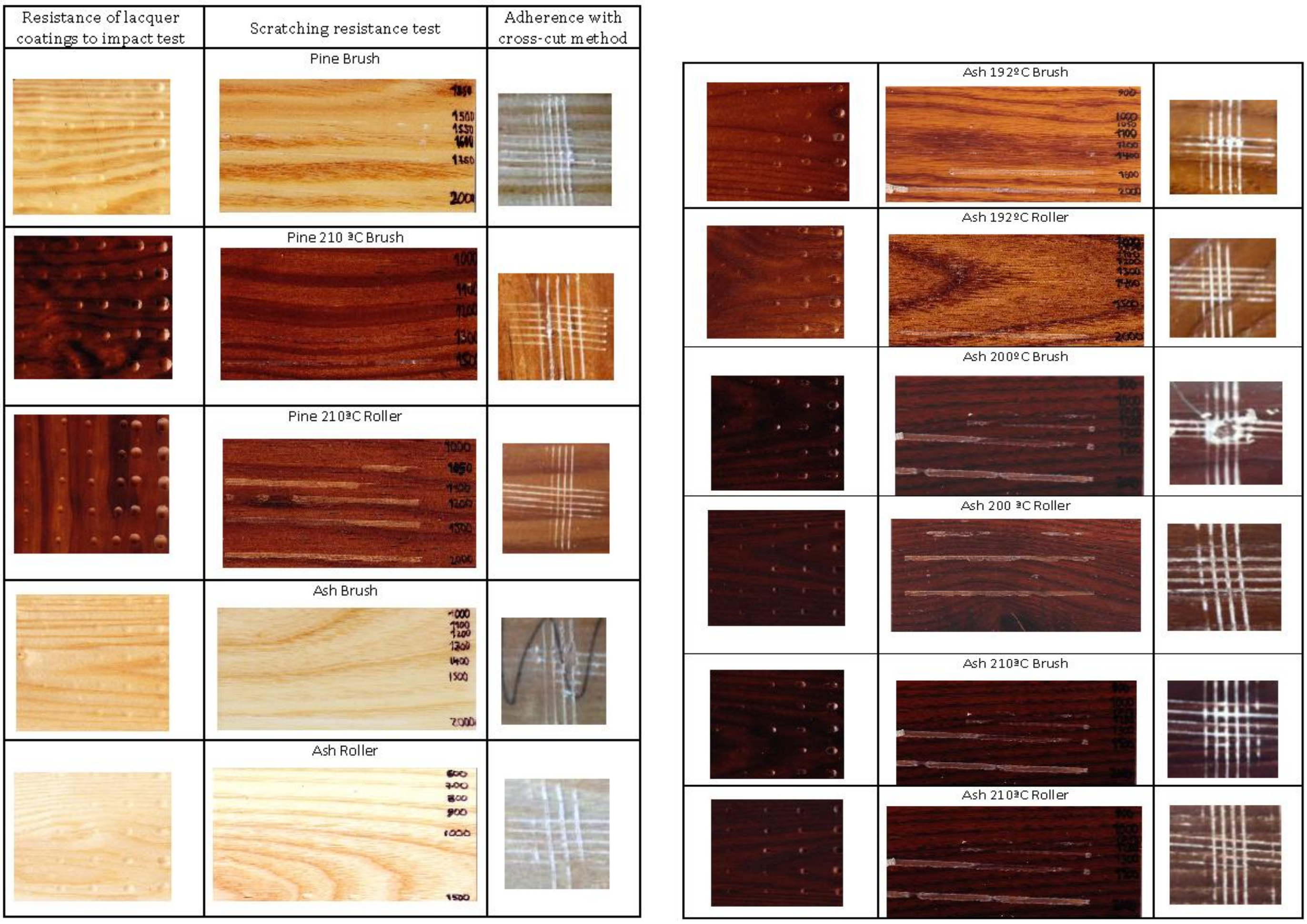

Figure 4. Resistance test of lacquer coatings to adherence, impact and scratching

3.6 Evaluation of resistance to selected thermal factors

Several tests on painted surfaces were performed in order to determine limits on the surface without apparent damage. The results of these tests are visual and are not included in the report. This data will be used in ongoing research in order to compare the results of these visual measurements with the results of the other analytic techniques.

4. Future collaboration with host institution

Proposals of collaborations were discussed during the STSM and as the result it was decided to collaborate through publications on impact factor journals, joint participations in international workshops and collaboration through specific research projects in the frame of COST Action or agreements in research projects related to wood modification.

5. Confirmation by the host institution of the successful execution of the STSM

It's attached in a separate file.

6. Other comments

I would like to thank the COST Action FP1006 committee for supporting this research experience, and I am very grateful to Dr. Tomasz Krystofiak my supervisor in host institution, to PhD student Monika Muszyńska for her cooperation, and to all the staff of Pozńan University of Life Sciences in Poland that kindly collaborated with me.

Special thanks to Dr. Jalel Labidi from my home institution that helped me in preparation for the STSM, the Basque Government (scholarship of young researchers training and project GIC12/55) and Termogenik Company.