Service life of low build hybrid wood treatment for tongue and groove cladding – FTIR study of chemical interaction between wood substrate and treatment formulation

8th Woodcoatings Congress

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SHR

Outline

- Introduction: Project Goals
- Previous results: Two step process on cladding parts
- Test setup: Natural weathering test
- Results after 2 years of natural weathering: FTIR results
- Outlook

Two step process

New approach to achieve covalent bonding of the finish coat to the wood by means of functional silanes in a sol-gel pre-treatment formulation.

Adhesion – Proof of concept

0% amino silane

3% amino silane

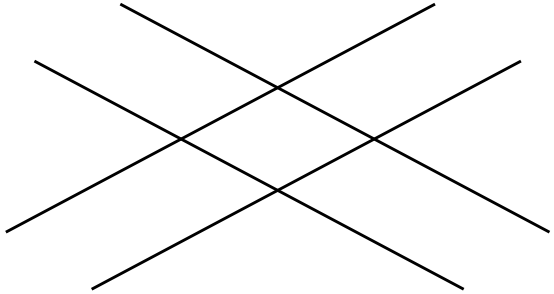
12% amino silane

Test setup

- Medium build paint systems
- Cladding parts: modified Radiata pine
- 8 weeks of artificial weathering EN927-6
- 2 years of natural weathering Southwest orientation
- Adhesion test SKH publication 05-01
- Visual assessment for cracking and flaking
- FTIR analysis for presence of silanes at interface wood - coating

Tape test

According to SKH Publication 05-01, adhesion is tested directly after weighing and on a clean (dried with a tissue) surface. Four cuts are made in the paint systems with the following pattern:



The distances between the cuts is about 1 cm and the angle between the cuts is about 30°. A tape (bond strength of 10N / 25mm) is placed over the cuts, pressed firmly and pulled off after one minute in an angle of 180° in about 1 second time. Adhesion is evaluated according to SKH Publication 05-01. An evaluation of 5 means no adhesion and 0 means excellent adhesion.
With this method it is assumed that the adhesion between the tape and the coating is of such quality that a viable assessment can be made of the adhesion of the coating to the wood.

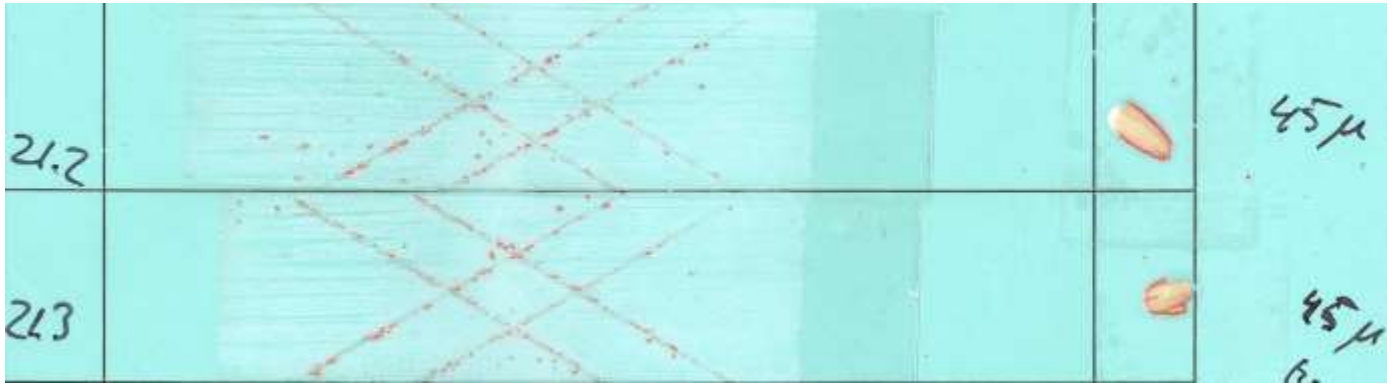
Upscaling - 8 weeks EN 927-6

Evaluation after 8 weeks of artificial weathering for planed Norway Spruce for the novel paint system:

Paint code	Cracking [Y/N]	Flaking [Y/N]	Adhesion [0 – 5] Tape test	Chalking [0 – 5] Tape test	Remarks
21	N	N	1,0	4,0	

In the figures below two individual test panels are shown after 8 weeks of artificial weathering for planed Norway Spruce along with the tape test results.

Sample code 21



Upscaling – IR and spray cycle

Accelerated weathering by IR heating and water spray

- no cracking or flaking
- increased heat in drying = improved adhesion

Upscaling – natural weathering

- Natural weathering
  - Water trap applied to panels
  - Started Summer 2010

Step 1: Pre-treatment formulation

Raw data for pre-treatment formulation G used in up scaled testing.

Component	**Amount [g]**	**Amount [wt %]**
Binder*	852.35	32.74%
Water (adjusted to pH 2)	852.17	32.73%
TEOS	287.49	11.04%
Ethanol	94.87	3.64%
ɣ-aminopropyltriethoxysilane	349.22	13.41%
Octyltriethoxysilane	42.41	1.63%
10% Hydrochloric acid	125.00	4.80%
Total	***2603.51***	***100.00%***

** Urethane modified co-solvent free short oil alkyd resin emulsion (32% oil).*

Step 1: Pre-treatment formulation

- Mixing of the binder/water part and the seperately prepared silane/ethanol part (24 hrs in magnetic stirred capped flask) was done in an overhead stirrer (open air).

- Remarkable difference with previous 100 ml volumes was that pH control to pH 5 was impossible. Stopped with adding 10% HCl solution at 5% on total formulation.

Step 1: Pre-treatment application

- Submerging of panels in pre-treatment formulation G for 30 seconds

- Drip drying before drying variations:
  AD: 24 hrs ambient conditions
  1h IR: 1 hr IR heating (70°C) followed by 23 hrs ambient conditions
  8h IR: 8 hrs IR heating (70°C) followed by 16 hrs ambient conditions
  24h 103: 24 hrs oven curing at 103°C
- Conditioning at 20°C / 65% RH for 24 hrs

Step 2: Paint formulation

Raw data composition of SHR formulated model paint.

Component	Amount [g]	
	Paint 1	Paint 2
Demineralised water	56.66	80.03
Ammonia 10% solution	2.02	2.16
Disperging agent	9.87	10.55
Wetting agent	0.62	0.66
Defoamer 1	0.51	0.44
Micronised TiO2	40.64	43.09
Binder*	229.26	243.07
Drier-solution	5.26	5.57
Defoamer 2	2.08	2.20
Co-solvent	9.00	7.54
Thickener 1	4.33	4.59
Thickener 2	1.37	1.46
Defoamer 3	0.72	1.62
Red pigment 1	1.06	1.30
Red pigment 2	0.45	0.56
Yellow pigment	0.11	0.13
Total	***363.95***	***404.98***

** Urethane modified co-solvent free short oil alkyd resin emulsion (32% oil).*

Step 2: Paint application

Radiata pine cladding parts for natural weathering test.

Drying Method	*Weathering Test*
	Natural weathering
AD	900mm: 4 panels (2x 1, 2x 2 paint layers)
1h IR	900mm: 4 panels (2x 1, 2x 2 paint layers)
8h IR	900mm: 4 panels (2x 1, 2x 2 paint layers)
24h 103°C	450mm: 6 panels (2x no paint, 2x 1, 2x 2 paint layers)
Reference	450mm: 6 panels (3x 2 paint layers SHR paint, 3x 2 paint layers Commercial paint)

2 years natural weathering

Code	*	Photos of the exposed cladding parts
1h IR A3	1	
1h IR D1	1	
1h IR C3	2	
1h IR F2	2	

** number of brush applied paint layers.*

2 years natural weathering

Code	*	Photos of the exposed cladding parts
8h IR B1	1	
8h IR F3	1	
8h IR A1	2	
8h IR E2	2	

** number of brush applied paint layers.*

2 years natural weathering

Code	*	Photos of the exposed cladding parts
AD C2	1	
AD E1	1	
AD B3	2	
AD D3	2	

** number of brush applied paint layers.*

2 years natural weathering

Code	*	Photos of the exposed cladding parts	
24h C4	0		32%
24h D4	0		45%
24h A4	1		33%
24h E4	1		27%
24h B4	2		17%
24h F4	2		47%

** number of brush applied paint layers.*

2 years natural weathering

Code	*	Photos of the exposed cladding parts	
Ref A2-1	2		43%
Ref C1-1	2		18%
Ref E3-1	2		16%
Ref A2-2	2		37%
Ref C1-2	2		28%
Ref E3-2	2		15%

** number of brush applied paint layers.*

FTIR study

- Shimadzu IR Prestige-21
  Mid-IR scanning range 4000 – 750 1/cm
- Shimadzu AIM-8800
  Automatic Infrared Microscope
  Liquid N2 cooled MCT detector
- Micro ATR crystal
  spot of diameter about 200 micrometer
  viewing dept up to 10 micrometer

FTIR study

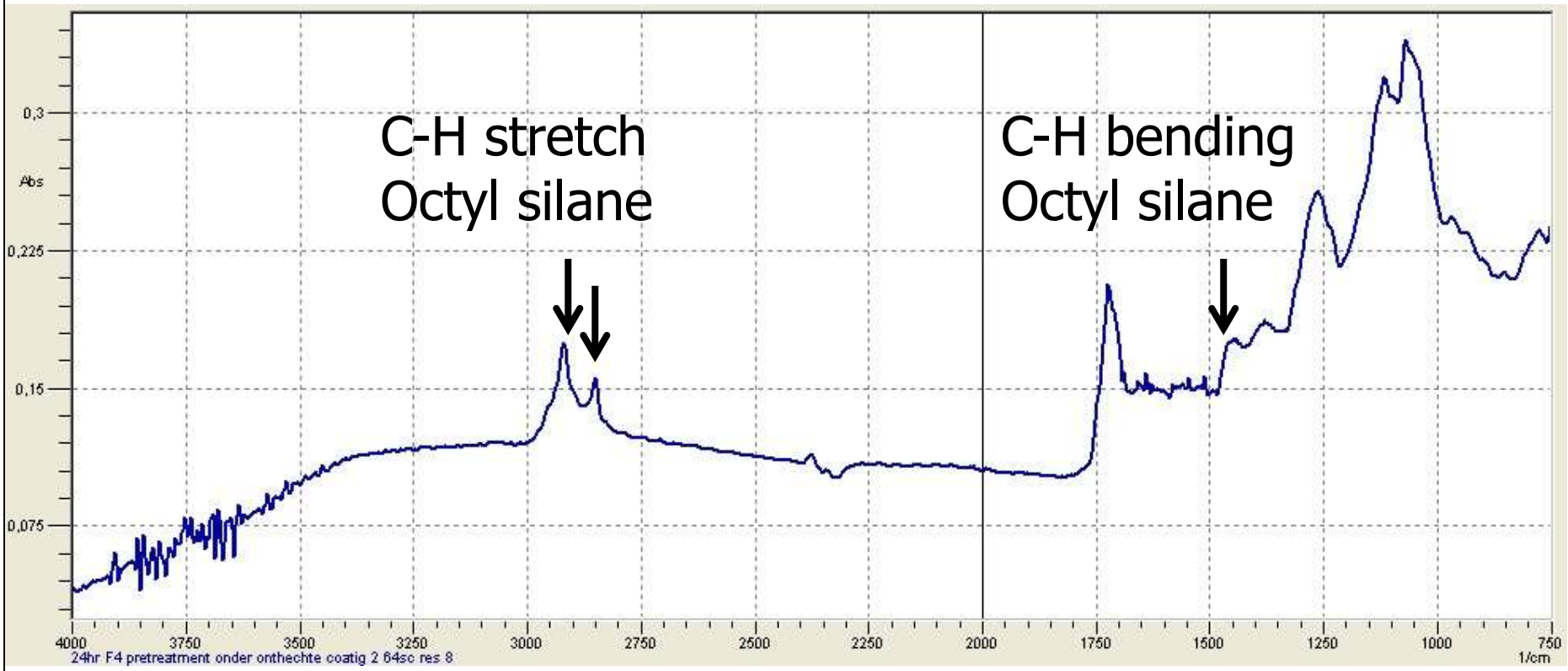
Thermally modified Radiata pine

FTIR study

Finish coat wood-paint interface at 2 years assessment (panel E3-1)

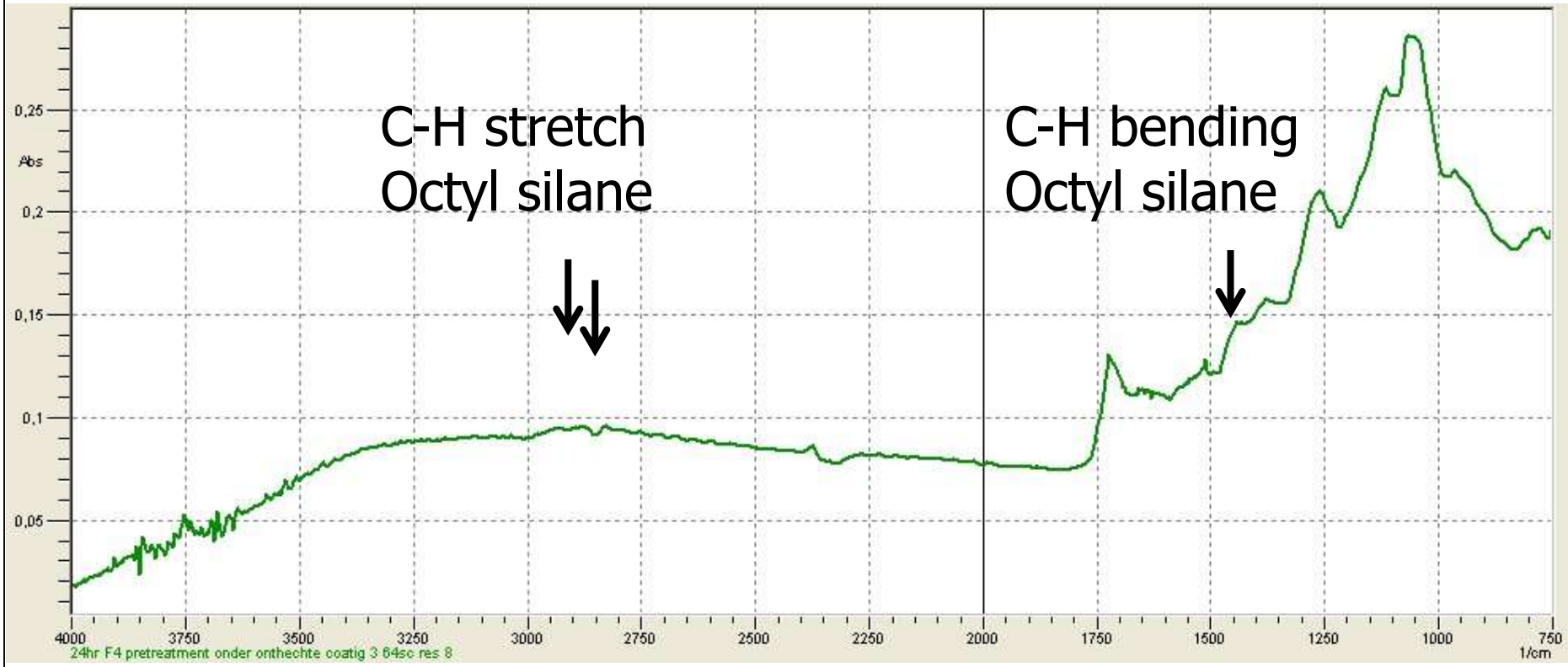
FTIR study

Pre-treatment interface with finish coat after adhesion test at 2 years assessment (24h F4)



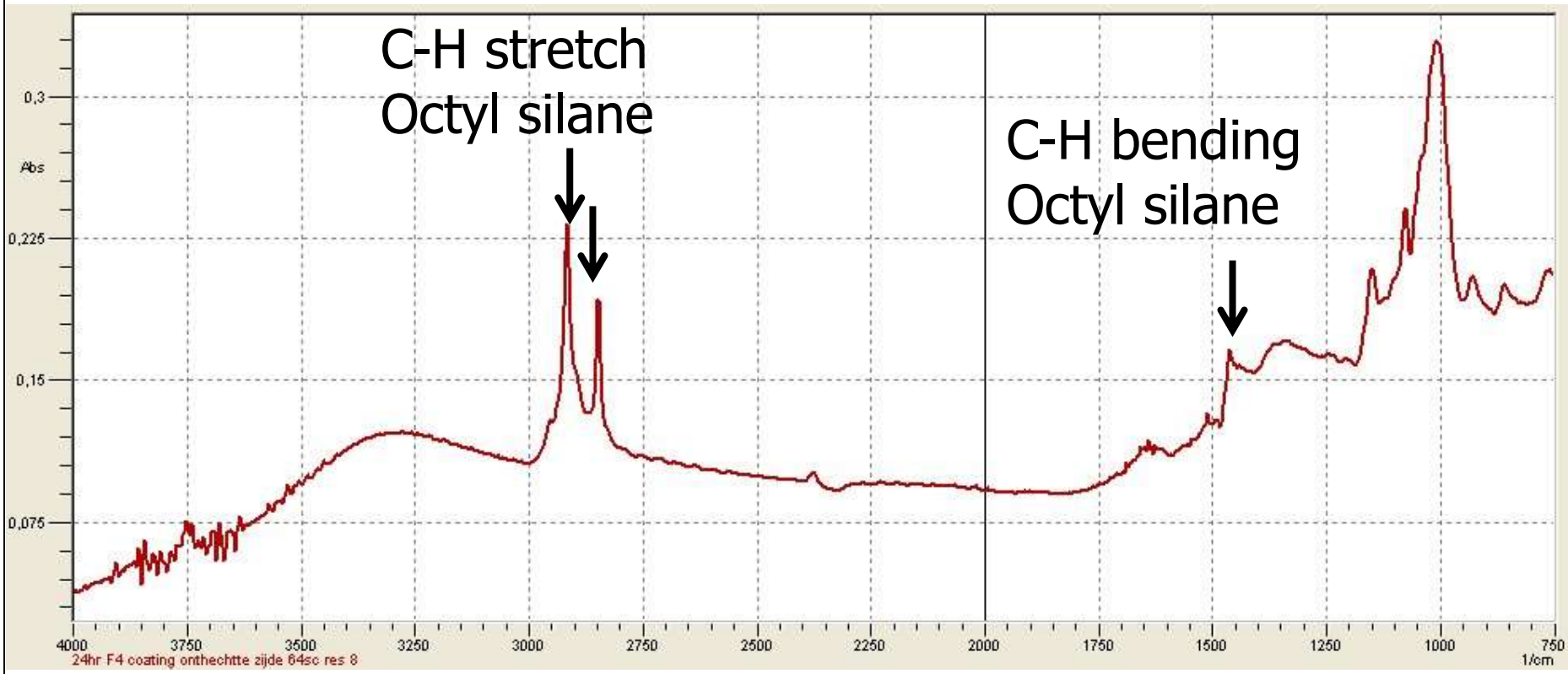
FTIR study

Pre-treatment interface with finish coat after adhesion test at 1 year assessment and subsequent weathering for 1 year (24h F4)



FTIR study

Finish coat interface with wood after adhesion test at 2 years assessment (24h F4)



Conclusions and outlook

- Small scale tests with 100 g pre-treatment formulation showed fast reactivity towards gelation and good adhesion of the system to the wood was seen
- Up scaling of the pre-treatment formulation to 2600 g showed much greater stability and therefore slower reactivity towards gelation
- This might have influenced the natural weathering test results.
- Visual assessment showed very little differences after 2 years natural weathering

Conclusions and outlook

- FTIR analysis showed the presence of the octyl silane at the wood interface
- FTIR analysis showed poor weatherability of the pre-treatment, therefor a protective finish coat is needed
- FTIR analysis showed good adhesion of the finish coat to the pre-treatment, the adhesion failure that was seen in the natural weathering test apparently originates from poor adhesion of the pre-treatment to the wood

Conclusions and outlook

- Later this year the opportunity will come to perform SEM-EDX analysis of some of the panels
- Natural weathering will be continued to see whether or not differences in durability will show between reference panels and test panels
- At appropriate time optimisation of both pre-treatment formulation and finish coat will be made

Thank you for your attention !

Problem identification

Blistering and flaking due to adhesion failure of paint

Aim

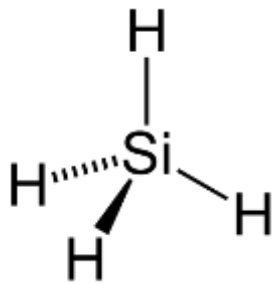
Develop a wood treatment based on silane sol-gel technology to:

- improve hydrophobicity of wood
- achieve persistent wet adhesion

Chemical Background

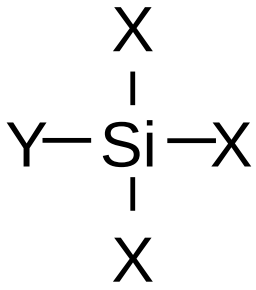
Silanes

Chemical compounds consisting of a central silicon atom with 4 constituent groups



Organofunctional silanes

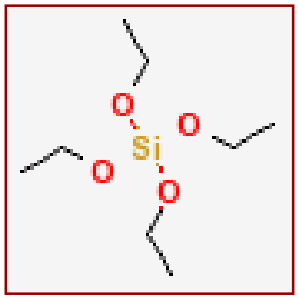
Silanes having both inorganic and organic groups



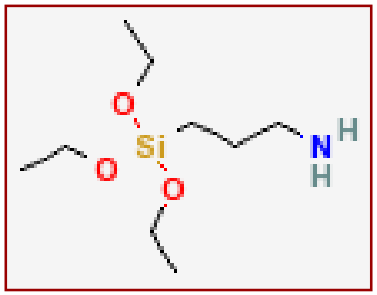
Where X = inorganic hydrolyzable group
and Y = organofunctional group

Chemical Background

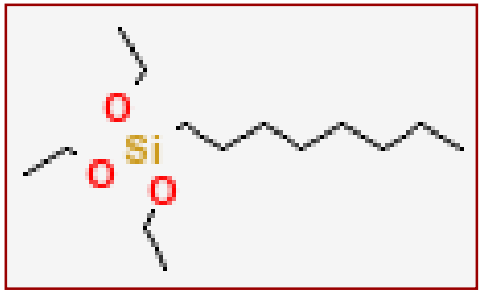
Silane compounds used during experimentation include:



Tetraethylorthosilicate (TEOS)



ɣ-aminopropyltriethoxysilane



Octyltriethoxysilane

Chemical Background

Sol-gel process

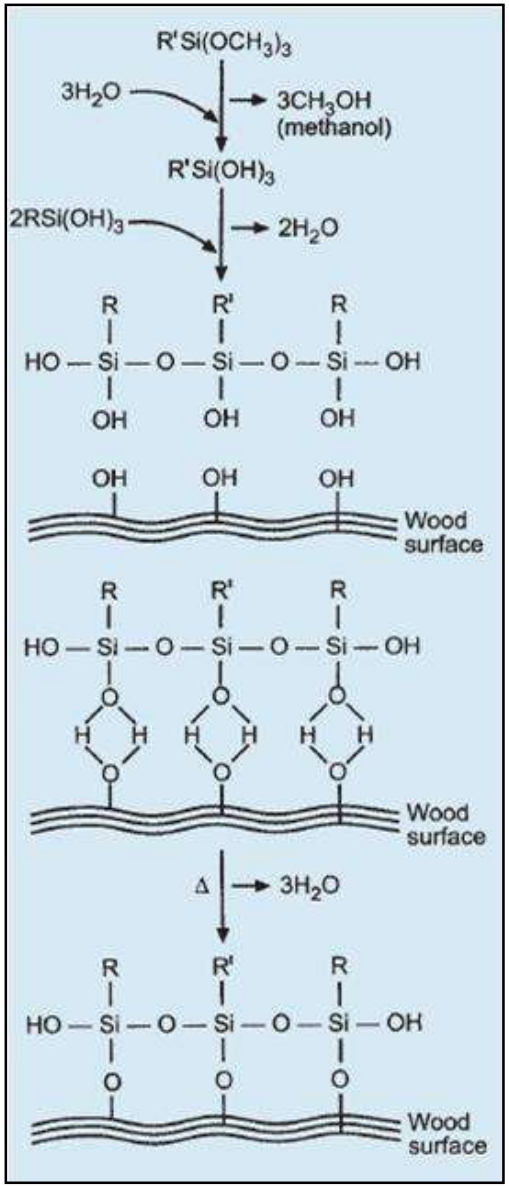
1) Hydrolysis

   • alkoxy groups are hydrolyzed to form silanols and alcohol is released

2) Condensation

   • silanol + silanol: Si-O-Si network formed

   • silanol + surface (wood): covalent bonding of silane to the wood



Questions

Thank you for your attention!