COST MC Chair:
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STSM SCIENTIFIC REPORT

Action: COST FP1006
Date of the visit: 8th December – 21st December 2014
COST STSM Reference Number: COST-STSM-FP1006-21617
STSM Research Theme: Using STA for chemical analysis of bio-friendly wood preservatives and coatings
STSM Applicant: Dr Magdalena Broda, Poznan University of Life Sciences, Faculty of Wood Technology, Institute of Chemical Wood Technology, Poznan, Poland
Host: Dr Lone Ross Gobbaken, Norwegian Forest and Landscape Institute, Section Wood Technology, Aas, Norway

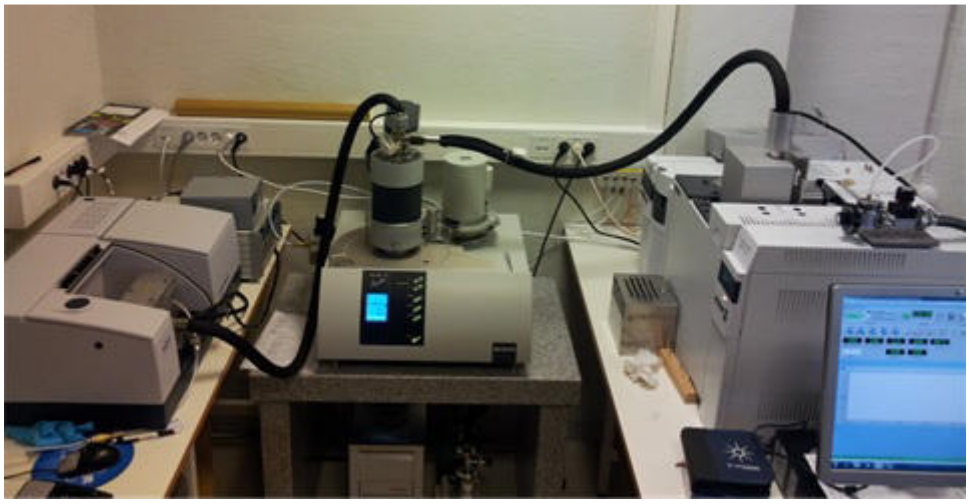
STSM Scientific Report Contents:

1. Purpose of the STSM

Within the framework of my research some new bio-friendly wood preservatives and coatings were prepared. The main objective of my project are preparations based on natural oils, silanes and inorganic ingredients. The most successful formulations are based on silanes (aminosilane, vinylsilane and methylsilane) containing carbonates and phosphates. Due to so far results there is a need to explain their mechanisms of hydrolysis and reactivity with wood.

The purpose of my STSM to Norwegian Forest and Landscape Institute was to use the STA/FTIR/GC-MS device (simultaneous thermal analysis/Fourier transform infrared spectroscopy/gas chromatography - mass spectrometry) for

complete characteristics of wood samples impregnated with silanes and inorganic compounds. The STA apparatus at NFLI offers several basic advantages: thermal analysis of temperature-depended mass changes (thermal gravimetric analysis) and caloric effects by means of the SC (differential scanning calorimetry) in a single measurement. Moreover, the STA is combined with FT-IR which allows full chemical analysis of the tested material. Such a combination of analytical equipment in one item gives the opportunity for complete analysis of new wood preservatives and coating and should give the answer for the question about the reactivity of the formulation's ingredients with wood and with each other.



STA device – FT-IR (left side), TG-DSC (Simultaneous Thermogravimeter – Differential Scanning Calorimeter - in the middle) and GC-MS (right side).

2. Description of the work carried out during the STSM

Materials:

1. Working solutions containing silanes, potassium carbonate, potassium silicate and the mixtures of silanes and inorganic compounds were prepared, evaporated and powdered.

2. Pine wood samples were subjected to two-step impregnation: primary vacuum treatment with potassium carbonate or potassium silicate and secondary

superficial treatment with silane. Additionally, wood samples without impregnation and impregnated with silane and with potassium carbonate/potassium silicate were prepared as reference. After impregnation wood samples were dried and powdered.

Method:

STA set up program for analysis of pine wood samples was prepared according to scientific literature and the experience of Janka Dibdiakova.

Parameters for samples measurement:

Instrument: NETZSCH STA 449F1 STA449F1A-0175-M
Filename: 2a_Pine_A.ngb-ds1
Date/Time: 15.12.2014 22:07:06 (UTC+1)
End Date/Time: 15.12.2014 23:13:55 (UTC+1)
Operator: Olm
Mode: DSC-TG
Measurement Type: sample with correction
Correction: Empty_20141215.ngb-bs1
Temp.Calib.: temp_N2_Al2O3_5K_min_20141010.ngb-ts1
Sensitivity: sens_N2_Al2O3_5K_min_20141010.ngb-es1
Crucible: DSC/TG pan Al2O3
DSC DSC Range: 5000 µV
TG TG Range: 5000 mg
Purge 1 MFC Range: 254 ml/min
Purge 2 MFC Range: 250 ml/min
Protective MFC Range: 250 ml/min
Sample identity: 3
Sample name: 2a_Pine_A
Sample Mass: 9.03 mg
Crucible: DSC/TG pan Al2O3
Crucible Mass: 149.39 mg
Reference crucible: DSC/TG pan Al2O3
Reference name: -----
Reference Mass: 0 mg
Reference Crucible Mass: 145.79 mg
Sample Number: 3
Sample Hole: No
Sample determination modeM**:** anual
Residuum measurement: Not possible
Furnace: Std SiC S
Sample carrier: DSC/TG Octo S
Measurement End: Normal end
Furnace TC: S
Sample TC: S
Purge 1 MFC: OXYGEN **Flow range:** 254.0 ml/min **predefined**
Purge 2 MFC: NITROGEN **Flow range:** 250.0 ml/min **predefined**
Protective MFC: NITROGEN **Flow range:** 250.0 ml/min **predefined**
Start criteria
Heating/equilibrium threshold: 7.5 K
Preheating rate: 20.00 K/min
Maximum equilibrium time after preheating: 00:30 hh:mm

Precooling rate: 50.00 K/min
Maximum equilibrium time after precooling: 05:00 hh:mm
FTIR settings
FTIR switch activity: Active in ASC run without vacuum
OPUS path same as Proteus: Yes
OPUS method: C:\OPUS_7.0.122\XPM\TGA_A587_Internal.XPM
Delay: 1 min
List of temperature steps:
Num Mode Temp. HR Acq.Rate Duration STC FT P1:O2 P2:N2 PG:N2 GC
°C K/min pts/min hh:mm
--- Start 42.0 1 0 0.0 50.0 20.0
1 Dynamic 439.0 10.000 600.00 00:40 1 1 0.0 50.0 20.0 1
2 Dynamic 710.0 10.000 600.00 00:27 1 1 50.0 0.0 20.0
--- Emergency 750.0 0 0.0 50.0 20.0

Parameters for reference sample:

Instrument: NETZSCH STA 449F1 STA449F1A-0175-M
Filename: Empty_20141215.ngb-bs1
Date/Time: 15.12.2014 17:38:36 (UTC+1)
End Date/Time: 15.12.2014 18:45:27 (UTC+1)
Operator: Olm
Mode: DSC-TG
Measurement Type: Correction
Temp.Calib.: temp_N2_Al2O3_5K_min_20141010.ngb-ts1
Sensitivity: sens_N2_Al2O3_5K_min_20141010.ngb-es1
Crucible: DSC/TG pan Al2O3
DSC DSC Range: 5000 µV
TG TG Range: 5000 mg
Purge 1 MFC Range: 254 ml/min
Purge 2 MFC Range: 250 ml/min
Protective MFC Range: 250 ml/min
Sample identity: 1
Sample name: Empty_20141215
Sample Mass: 0 mg
Crucible: DSC/TG pan Al2O3
Crucible Mass: 158.26 mg
Reference crucible: DSC/TG pan Al2O3
Reference name: -----
Reference Mass: 0 mg
Reference Crucible Mass: 145.79 mg
Sample Number: 1
Sample Hole: No
Sample determination modeM**:** anual
Residuum measurement: Not possible
Furnace: Std SiC S
Sample carrier: DSC/TG Octo S
Measurement End: Normal end
Furnace TC: S
Sample TC: S
Purge 1 MFC: OXYGEN **Flow range:** 254.0 ml/min **predefined**
Purge 2 MFC: NITROGEN **Flow range:** 250.0 ml/min **predefined**
Protective MFC: NITROGEN **Flow range:** 250.0 ml/min **predefined**
Start criteria
Heating/equilibrium threshold: 7.5 K
Preheating rate: 20.00 K/min
Maximum equilibrium time after preheating: 00:30 hh:mm
Precooling rate: 50.00 K/min
Maximum equilibrium time after precooling: 05:00 hh:mm

FTIR settings
FTIR switch activity: Active in ASC run without vacuum
OPUS path same as Proteus: Yes
OPUS method: C:\OPUS_7.0.122\XPM\TGA_A587_Internal.XPM
Delay: 1 min
List of temperature steps:
Num Mode Temp. HR Acq.Rate Duration STC FT P1:O2 P2:N2 PG:N2 GC
°C K/min pts/min hh:mm
--- Start 42.0 1 0 0.0 50.0 20.0
1 Dynamic 439.0 10.000 600.00 00:40 1 1 0.0 50.0 20.0 1
2 Dynamic 710.0 10.000 600.00 00:27 1 1 50.0 0.0 20.0
--- Emergency 750.0 0 0.0 50.0 20.0

Because of the problems with GC-MS sample loader only few wood samples were measured and, unfortunately, the obtained data were not satisfying. Therefore, after the GC-MS repair, further analyses were planned in the middle of January 2015.

Other activities during STSM:

1. Cleaning GC-MS sample loader with use of iso-propanol.
2. Reading literature about Simultaneous Thermogravimetry – Differential Scanning Calorimetry.
3. Giving a presentation of myself and my research as well as the research mainstreams of Institute of Chemical Wood Technology, Poznan University of Life Sciences for NFLI scientists.

3. Description of the main results obtained

Only few ST-DSC and FT-IR spectra were obtained. Due to GC-MS breakdown the obtained results are not satisfying and they are not suitable for publication. And, as it was mentioned before, further analyses will be done in the middle of January 2015.

4. Future collaboration with host institution (if applicable)

The STSM allowed us to exchange literature, knowledge and research experiences. I believe that the base for personal and institutional cooperation has been established during this scientific mission. We have opened several possible

topics for the future with joint experiments, projects, articles and other STSMs. In the next months I would like to go again to NFLI to finish my STA analyses, jointly interpret obtained data and prepare a publication.

5. Foreseen publications/articles to result from the STSM (if applicable)

The results obtained during this mission and in January 2015 will be presented in the Thessaloniki meeting within COST Action FP1006 and will be the issue for 1-2 articles in scientific journals as agreed with Dr. Janka Dibdiakova from NFLI.

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

The confirmation is sent as a separate file.

7. Other comments (if any)

I would like to express my sincere thanks to Dr Lone Ross Gobbaken and Dr Janka Dibdiakova for the care and support during my STSM. Research and accommodation conditions at NFLI in Aas were excellent. The research team at laboratories was highly helpful (many thanks for all of them), so I felt comfortable in this new location. STSM was a fantastic experience for me and a unique opportunity to perform the necessary analysis for my research as well as to establish cooperation with outstanding researchers.

I would also like to pass on my gratitude to the MC of COST FP1006 and to the COST Administrative Secretariat for granting the funding to allow me to carry out my first Scientific Mission.