

**Project name:** "Research of lignin"

## Beamtime Report

**dd.mm.yyyy - dd.mm.yyyy** *(Date of the report to be added)*

### General information

Name of the rapporteur	Name of the rapporteur's organisation
Egle Urbonaite	"Lutora"
Type of research (nanotechnology/health care/chemistry etc.)	Name of the research facility
Environment and Energy	Max IV Laboratory (synchrotron rings)
Date of the measurement, duration	Location of the event
2013 09 11	Lund, Sweden
Facility personnel participating in the measurement	
<i>Anders Engdahl, Per Uvdal, Andreas Lassesson.</i>	

### Description of the project

Research description (short summary as written in the application)
<p>From 1930 till the end of 1990 wood hydrolysis industry in the entire Soviet Union held the established niche in the manufacturing sector. After the Soviet Union's collapse huge unprocessed waste landfills remained in the Eastern Europe posing serious environmental problems due to self-ignition, soil and groundwater contamination. These, richly lignin-saturated waste streams can be seen as a unique resource for production of bio-based chemical substances and products. Polyphenolic lignin nature and its low toxicity, along with other positive qualities (thermal plasticity, resistance to UV rays etc.) make it an ideal substitute for the polymers made from oil.</p> <p>Tomas Elenbergas' individual company "Lutora" for already 16 years provides the European market with engineering services, now in order to contribute to the development of Resource Efficient Europe policy initiatives is expanding its operations into high-value products from bio-waste. The main goal of this new activity is full recovery of hydrolyzed lignin depositions, processing them into raw materials for a variety of organic substances and products. The intensive cooperation with Lithuanian (Vilnius Biotech University), Latvian (Wood University), and Canadian R &amp; D partners, hydrolyzed lignin was processed into higher value-added products: "pure" lignin, which may be suitable for bio-plastics production and bio-based acid catalyst that could replace petroleum-based extracted catalyst widely used in the food, textile, chemical and polymer industries. However, because of limited opportunities of local chemistry laboratories we have problems with our new product evaluation and analysis.</p> <p>Science Link project is a unique opportunity by using radiation source (synchrotron) to qualitatively explore the products obtained (by setting the potential areas of use for this bio-waste) as well as a great opportunity to find co-points.</p>

**Summary of activities (experiments performed, beamtime used, preliminary overview of results, next steps and other relevant information)**

Seven different samples were measured with FT-IR at the D7 beamline on the MAX III ring at MAX-lab. The samples were powder of lignin. Pellets of KBr with the powder imbedded (1 mg in 200 mg KBr) was pressed at MAX-lab and used for measurements.

Three different possibilities existed to study the sample.

1. Microscope (Globar)
2. Sample compartment.
3. Microscope (SR).

In the sample compartment measurements the whole area of the pellet are illuminated and analysed. In the two other methods a small subarea is analyzed in each run. If the sample is homogenous they all are expected to give the same result.

When synchrotron radiation (SR) is used instead of the global source the signal is higher, in fact the aperture has to be smaller not to overload the detector. A smaller area of the sample is studied in each run, meaning that with an inhomogeneous sample there is a possibility to get much better spatial resolution with the synchrotron than with the global. Also the SR can penetrate somewhat thicker samples than the global.

The fact that it was powder samples also means that spatial resolution is not important on these samples. If thin slices (10 micron thick) of the samples were studied instead one could make chemical maps and see how different substances are distributed in the sample. It is also questionable if the synchrotron really improves things in this case. A good sample compartment measurement would give a very nice signal to noise ratio. Synchrotron would help if the signal with the global is very low, if spatial resolution is needed and if one would like to make a spectral map of the sample.

The bands were identified by MAX-lab researchers by studying literature, compare peak positions and from that make an assignment table (see below). For the future MAX-lab recommends that ab initio calculations on lignin are performed (hard because the exact structure has to be known) and compare the results to the experimental data thereby getting an assignment of the individual bands. However, this was beyond the scope of this investigation.

For the future one should also measure on known, clean substances to be used as references. The calculations could then also be made on these substances as a check on how good the calculations work.

For a full list of samples, see the last figure in the annex.

Techniques:

1. Global 50x50. Run in the microscope with a 50x50 micron aperture, meaning looking at just one small part of the KBr pellet in each run. The presented spectrum is a mean of several runs. Light source global.
2. Sample compartment. Run in the spectrometer using the whole KBr pellet (13 mm diameter). Light source global.
3. SR 30x30. Run in the microscope with a 30x30 micron aperture, meaning looking at just one small part of the KBr pellet in each run. The presented spectrum is a mean of several runs. Light source Synchrotron Radiation.

#### Assignment Lignin sample 1

##### Peaks in Sample 1

3386 cm<sup>-1</sup>, O-H stretch (hydrogen bonded). Possible origin: hydroxyl groups in phenolic and aliphatic structures,

2933 cm<sup>-1</sup>, C-H stretch. Possible origin: aromatic methoxyl groups and methyl and methylene groups of side chains.

2852 cm<sup>-1</sup>, C-H stretch. Possible origin: aromatic methoxyl groups and methyl and methylene groups of side chains.

1706 cm<sup>-1</sup>, C=O stretch (unconjugated). Possible origin: unconjugated carbonyl/carboxyl stretching

In between 1706 and 1606 can come from protein impurities or water associated with the lignin.

1606 cm<sup>-1</sup>, Aromatic skeletal vibration + C=O stretching.

1512 cm<sup>-1</sup>, Aromatic skeletal vibration.

1462 cm<sup>-1</sup>, C-H deformation (methyl and methylene)

1426 cm<sup>-1</sup>, C-H in plane deformation with aromatic ring stretching

1368 cm<sup>-1</sup>, ?

1316 cm<sup>-1</sup>, C-O of syringyl ring

1270 cm<sup>-1</sup>, C-O of guaiacyl ring

1217 cm<sup>-1</sup>, C-O of guaiacyl ring

1153 cm<sup>-1</sup>, Guaiacyl C-H and Syringyl C-H

1115 cm<sup>-1</sup>, ?

1059 cm<sup>-1</sup>, ?

1033 cm<sup>-1</sup>, C-O of primary alcohol, guaiacyl C-H

857 cm<sup>-1</sup>, C-H out of plane

798 cm<sup>-1</sup>, ?

#### **How would you describe cooperation and assistance from industrial liaison officers and national contact points while preparing and carrying out the research at large scale facilities?**

Cooperation and assistance in this project, we appreciate the positive. This will contribute significantly to the further development of our research.

#### **Other personal remarks**

## Annexes

### Annexes

(list of annexes; meeting minutes, graphical illustrations, tables and other supplementary data)





















