X-ray and FTIR μ-CTs for morphological and chemical characterization of eco-sustainable insulating foams

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Abstract

Here it is reported a multidisciplinary approach based on tomography and infrared techniques applied to the characterization of tannin porous rigid foams, potentially usable as new insulating materials in green building technology. With conventional x-ray tomography it was possible to preliminary evaluate the homogeneity of the samples at low resolution, while then, thanks to the synchrotron source, it was possible to obtain more detailed information at a micro-scale level. At the same time chemical characterization was done through Fourier Transform infrared (FTIR) imaging. Conventionally, FTIR imaging is limited to a planar projection, not considering the 3D structure of the material. To avoid this limitation, a FTIR 3D-tomography setup was built and the foams characterized by a chemical point of view. The idea is to directly correlate these data with the 3D-structural information obtained with the x-ray computed tomography exploiting the synchrotron radiation as source, allowing a complete characterization of the material morphology and chemistry at the microscale.

Keywords: tannin foam, mesoscale, FTIR, computed tomography, tomoIR.

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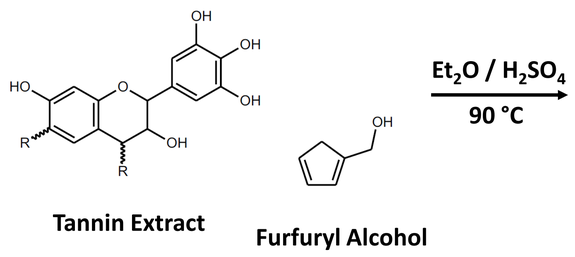
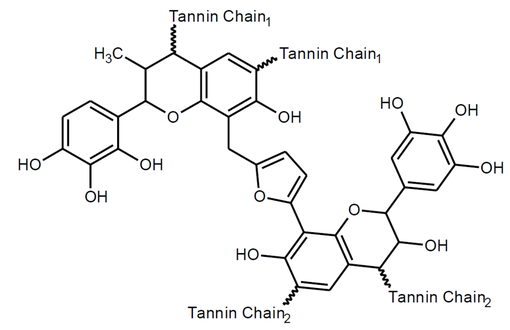
1. Glinz, J., Senck, S., Kastner, J. & Tondi, G. Determination of pore size distribution in Tannin- and Lignin-based foams using X- ray microcomputed tomography. in 8th Conference on Industrial Computed Tomography, Wels, Austria (iCT 2018) 2–8 (2018).

Tannin Porous Rigid Foams

Tannin foam polymerization



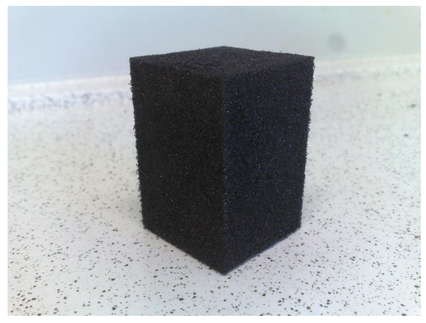
Tannin Extract **Furfuryl Alcohol**

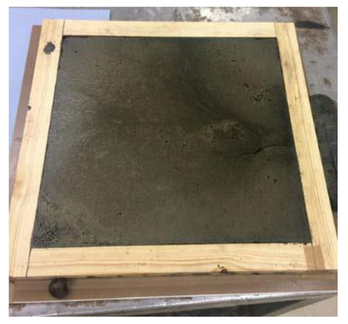


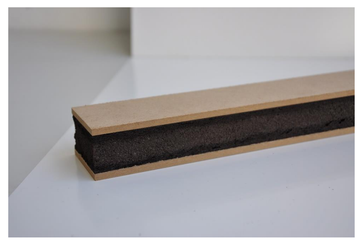
Tannin-furanic polymer

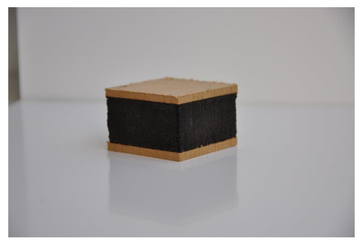
Acid catalyzed polymerization between the tannin extract and furfuryl alcohol leads to the tannin-furanic polymer. The blowing agent (diethyl ether) as well as the temperature are necessary for the growing of the foam, ending up in a porous and rigid material. (*) Here it is reported one of the possible product chemical structures which are still under investigation.

- **100% natural structure (tannin and furfuryl alcohol)**
- **Not soluble in water, solvents and acids**
- **Fire resistant: self-extinguishing**
- **Thermosetting polymer**







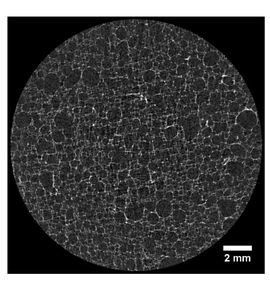


- **Density >30 kg/m³ (from 30 up to 300 kg/m³)**
- **Compression resistance (from 0.01 up to 0.5 N/mm²)**
- **Insulating material (30 mW/m.K < thermal conductivity < 50 mW/m.K)**

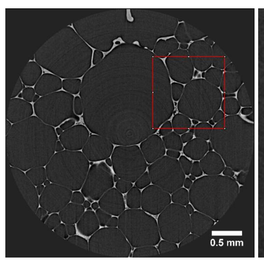
Characterization Techniques

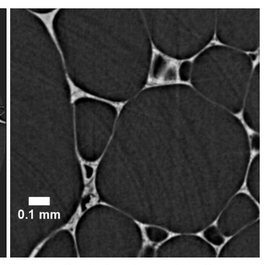
X-ray Micro-Computed Tomography (μ-CT)

Preliminary screening for homogeneity assessment: Cone-Beam μ-CT slice
(local area, low resolution Isotropic voxel 9 μm)

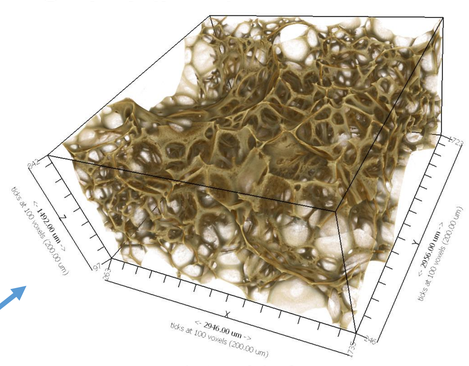


Morphological characterization: Synchrotron Radiation (SR) μ-CT slice
(Propagation Based phase-contrast CT in local area with white beam at high resolution, isotropic voxel 2 μm)

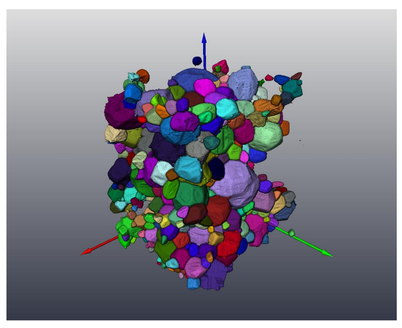




Detail of the SR μ-CT slice

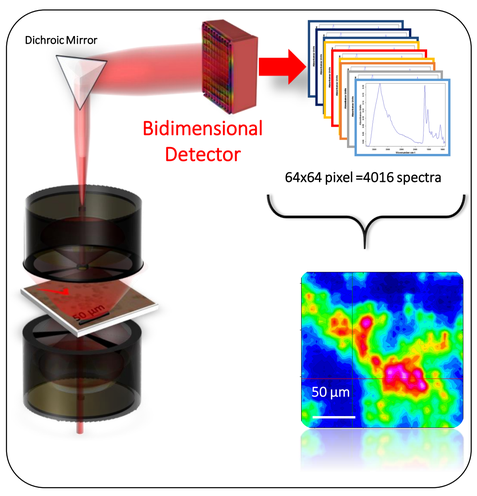


Rendering of the foam



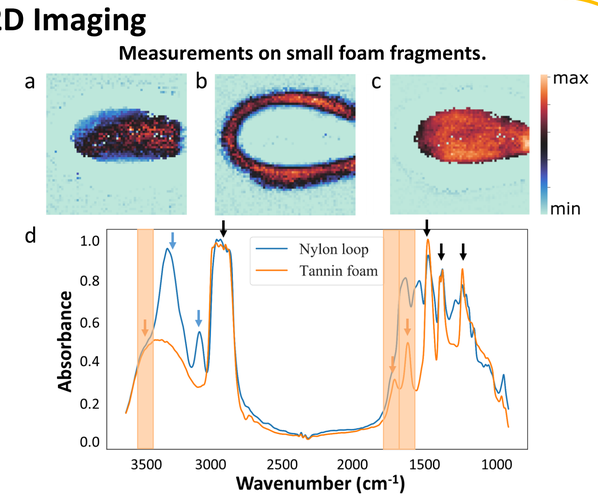
Cell segmentation and analysis

FTIR 2D Imaging

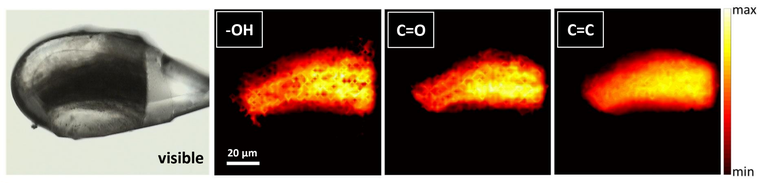


FTIR IMAGING

Measurements on small foam fragments.



a) **Tannin foam contribution: ν (-OH)**
b) **Nylon loop contribution: Amide-B**
c) **oil contribution: δ(C-C)**



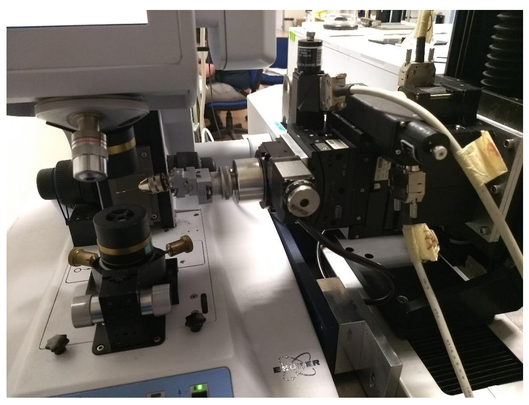
Data pre-processing is a mandatory step to separate the contributions of the foam from those of the nylon loop and the oil used to fix the sample. The data treatment is divided in two steps:

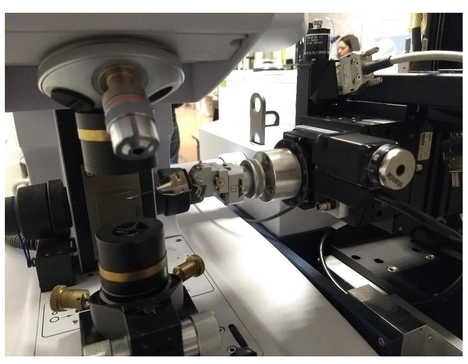
1. **Estimation of the nylon contribution to the spectrum (spectral range 3040-3120 cm^{-1});**
2. **Subtraction of the nylon contribution from the foam spectra.**

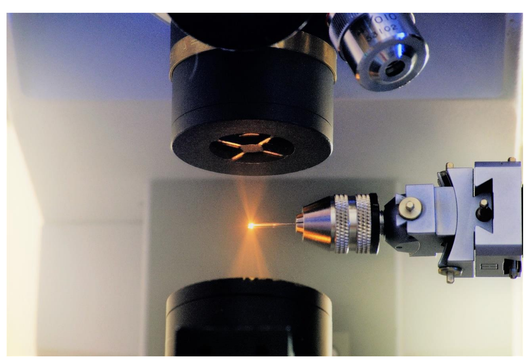
FTIR 3D Tomography

Setup

- **5 degrees of freedom;**
- **Nylon loop (50 μm loop hole and 20 μm nylon wire diameter) as sample holder;**
- **mineral oil (NVH Oil);**
- **72 projections (each 5°);**
- **Automatized rotation and measurements**

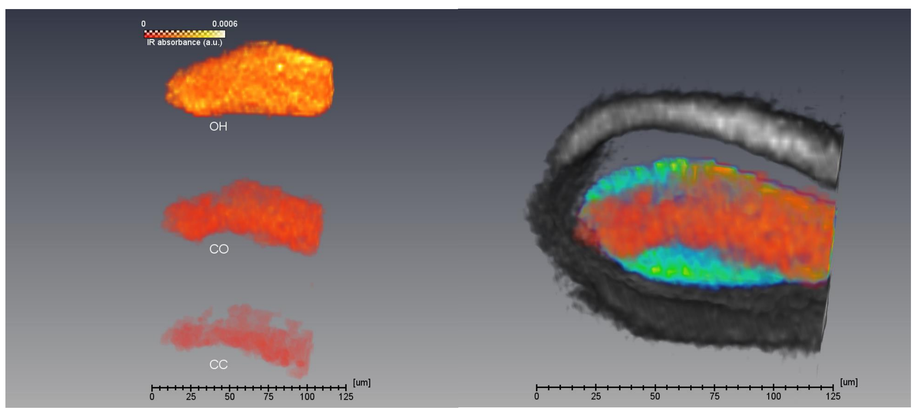






Preliminary Results

- **Each stack of projections, for each band, is divided by the corresponding molar extinction coefficient**
- **FBP reconstruction**
- **Pre-processing and segmentation using Avizo® 9.3**



Conclusions

- **Software and hardware implementations for fast and reliable data acquisition**
- **3D-reconstructions of the desired chemical moieties**

Future Perspective

- **Implementation of the IR-setup with objective at lower magnification to increase the field of view**
- **Development of new mathematical models to take in account the real physical processes**

Development of a robust technique to obtain 3D chemical mapping at the microscale level integrated with the morphology from X-ray μ-CT

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