Experimental report

13/02/2019

Proposal: **INTER-378** **Council:** 4/2018

Title: Investigating the pore accessibility in wood using SANS

Research area:

This proposal is a new proposal

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Samples: Wood (mainly cellulose, hemicelluloses, lignin) and polyethylene glycol in D2O

Instrument	Requested days	Allocated days	From	To
D11	1	1	02/04/2018	03/04/2018

Abstract:

Investigating the pore accessibility in wood using SANS

1. Background

Wood is an abundant and renewable material with various applications, which would benefit from a better understanding of its nanoscale structure. Small-angle neutron scattering (SANS) is an excellent tool for studying the nanoscale structure of wood [1] and other cellulosic materials [2,3], especially due to the possibilities of contrast variation. In this experiment, we collected SANS data from wood samples impregnated with polyethylene glycol (PEG) in order to investigate the penetration of this water-soluble polymer to pores of different sizes in the wood cell wall. As PEG is often used to preserve historical woods and to prevent shrinkage of wood during drying, the results are expected to be important for both applications and a more detailed understanding of small-angle scattering data from wood samples.

2. Experimental

Never-dried samples from birch, pine and spruce were collected from Eastern Finland and stored refrigerated in 30% ethanol solution [1]. Approximately 1 mm (R) x 10 mm (T) x 10-15 mm (L) pieces were cut with a razor blade and immersed in D_2O. The PEG treatment was done by gradually increasing the concentration of PEG in D_2O from 10% (w/v) to 60% (w/v) during a time of several weeks and the samples were allowed to equilibrate at the final concentration for 6 months. PEGs of three different molecular weights, BioUltra 300, BioUltra 1,000 and BioUltra 4,000, were all obtained from Sigma-Aldrich.

The PEG-impregnated wood sections were placed in quartz cells with a light path of 2 mm and SANS was measured at D11 using sample-to-detector distances of 1.4 m, 8 m and 39 m and a neutron wavelength of 6 Å. For the purpose of background subtraction, solutions of 60% PEG in D_2O were measured in a similar way. The wood samples impregnated with PEG4000 were additionally measured after immersing them into fresh D_2O and the measurement at the 1.4-m distance was repeated after 2.5-3 h to control if any PEG was transferred to the solution during this time.

3. Results

An example of a two-dimensional SANS pattern from pine wood impregnated with PEG1000, obtained at the shortest sample-to-detector distance (1.4 m) is shown in Fig. 1a. The two equatorial peaks in the pattern correspond to the regular aggregation of cellulose microfibrils with a centre-to-centre distance of 3-4 nm. The integrated equatorial SANS intensity from the corresponding sample after subtracting the background from PEG solution is presented in Fig. 1b. A peak corresponding to a real-space distance of about 3.5 nm, similar to the one marked in Fig. 1b, was found in all of the PEG-treated woods. An equatorial peak at slightly smaller q-values is observed in never-dried woods in 100% D_2O, which indicates that PEG affected the aggregation of cellulose microfibrils. A minor shift of the peak to lower q was also observed when the wood samples with PEG4000 were placed in pure D_2O solution.

For further analysis of the data, the isotropic intensity contribution from the 2D patterns will be subtracted and model fitting will be done following the procedures of reference [1]. The results from this analysis and a paper featuring also Raman microscopy results will be published during the year 2019.

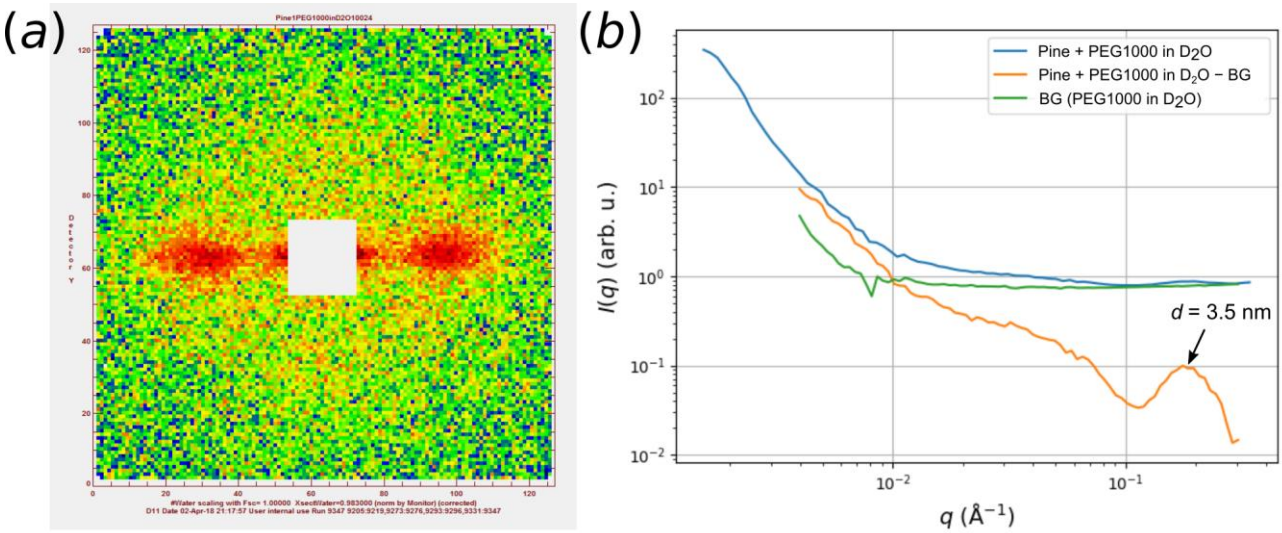

Figure 1. (*a*) Two-dimensional SANS patterns of a pine sample impregnated with PEG1000 (60% PEG in D_2O). (*b*) Equatorial intensity from the same sample before and after subtracting the solution background from PEG1000 in D_2O ("BG").

References

[1] P. A. Penttilä *et al.* (2019), Small-angle scattering model for efficient characterization of wood nanostructure and moisture behavior, *J. Appl. Crystallogr.*, accepted

[2] P. A. Penttilä *et al.* (2018), Biomimetic composites of deuterated bacterial cellulose and hemicelluloses studied with small-angle neutron scattering, *Eur. Polym. J.*, 104, 177-183

[3] P. A. Penttilä *et al.* (2018), Multimethod approach to understand the assembly of cellulose fibrils in the biosynthesis of bacterial cellulose, *Cellulose*, 25, 2771-2783