Experimental report

29/11/2018

Proposal: **1-04-136**

Council: 4/2018

Title: Penetration of waterborne coatingson model wood substrates

Research area: Materials

This proposal is a new proposal

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Samples: cellulose nanocrystals
polyurethane dispersions

Instrument	Requested days	Allocated days	From	To
D17	1	0		
SUPERADAM	2	1	22/09/2018	23/09/2018

Abstract:

Wood is a natural renewable material widely applied for construction, both interior and exterior. Because of being a material highly susceptible to degradation, coatings are often used for its protection. For the long-term performance, the wood-coating interface is of major interest as it determines e.g., the adhesion of the coatings. If the coating/wood interface fails, the coating will disintegrate within a short time and will blister, crack and peel. This failure can result in damage to the wood surface and in more costly and difficult refinishing. Coating adhesion is determined, among other factors, by its penetration through the wood interface. For the specific case of waterborne coatings, it is difficult to characterize their penetration by means of common techniques such as microscopies or tomographies as they do not typically fill the wood cell lumen. Instead, it is more likely that waterborne coatings penetrate the wood cell surfaces instead. At present, there is not a well-established methodology for these studies. We aim at proving with this experiment that Neutron Reflectometry is an optimal tool for these studies.

Wood is a natural renewable material that in the entire spectrum of building materials is still widely applied for construction, both interior and exterior. Being a multipurpose building material, wood is a major substrate for paint and other finishes produced by the coating industry. Because of being a material highly susceptible to degradation, one of the main functions of wood finishes is protection [1]. For the long-term performance, the wood-coating interface is of major interest as it determines e.g., the adhesion of the coatings. If the coating/wood interface fails, the coating will disintegrate within a short time and will blister, crack and peel. This failure can result in damage to the wood surface and in more costly and difficult refinishing. Because of wood being a highly porous material, coating adhesion is determined, among other factors, by its penetration through the wood interface [2]. However, the penetration of coatings into wood is not easy to study. Even with X-Ray tomography experiments on a variety of waterborne coatings (of characteristics like those available in the market) on wood surfaces, we could not observe filling of the inner cell lumen by the coatings. Thus, it is more plausible that penetration of waterborne coatings takes place at a smaller (possibly nano) scale and probably at the cell wall surface. However, a solid methodology to investigate this process has not yet been established. Neutron Reflectometry (NR) has already proved its potential as a tool for extracting structural information [3]. In order to study the penetration of waterborne coatings on model wood substrates, an experiment was realized on the SuperADAM reflectometer.

As model wood substrates, a layer-by-layer nanostructured films consisting of alternating layers of cellulose nanocrystals and nanoplatelets of gibbsite were deposited on Silica substrates (5 mm thickness, 5 cm diameter). Then, these nano-cellulose films were coated with macroscopic (>10µm) waterborne coatings (formed from aqueous polyurethane dispersions) and allowed them to dry at ambient conditions (RH=30%). The waterborne polyurethane dispersion was used on two different cellulose substrates. Due to time restrictions (one day to perform the experiment), only the surfaces with the cellulose already formed and the coated cellulose + silica systems were characterized by NR. The collection of data was made as follows:

- NR characterization of Cellulose 1, positive and negative angles.
- Coating of Cellulose 1
- NR characterization of Cellulose 2, positive and negative angles.
- Coating of Cellulose 2
- NR characterization of Coated Cellulose 1, negative angles.
- NR characterization of Coated Cellulose 2, negative angles.

Positive (negative) angles refer to data collected with the neutrons impinging from air (silicon). Data were reduced using the MATLAB GUI "Sared", with the help of Alexey Klechikov (local contact and co-responsible instrument scientist on SuperADAM).

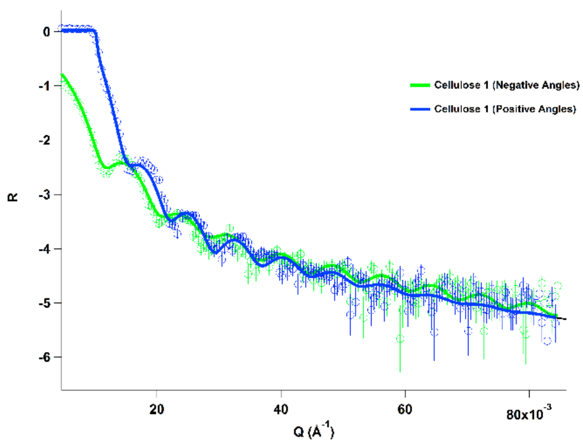

Figure 1. NR curves for Cellulose 1 (positive and negative angles). The preliminary fits (solid lines) corresponds to a 4-cellulose layer model.

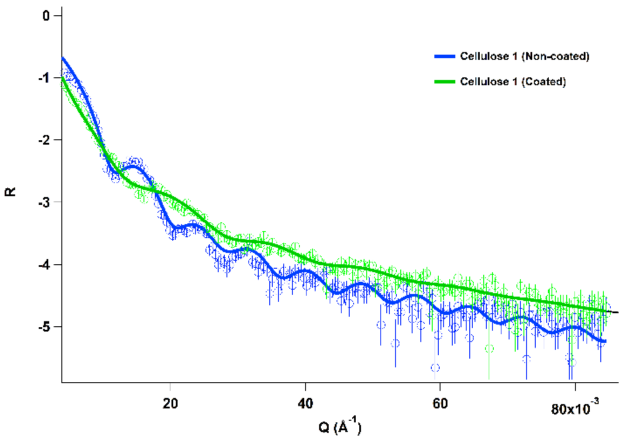

Figure 2. NR curves for non-coated and coated Cellulose 1 (negative angles).

For Cellulose 1, a model with four cellulose layers was used (Fig. 1). The SLD remains similar for both measurements (positive and negative angles) and compatible with the values obtained in the literature [3]. However, the SLD values from the fits (Fig. 5a) show that the configuration of the layers is not homogeneous, and some asymmetries could be present inside the volume. For both coated cellulose samples (Cellulose 1 and 2), only negative angles were measured due to the high thickness of the coating. Data were fitted using the previous result for Cellulose 1. However, in order to get a reasonable fit, the values for the thickness, roughness and solvent content for the previous cellulose layers were also changed (Fig. 5a).

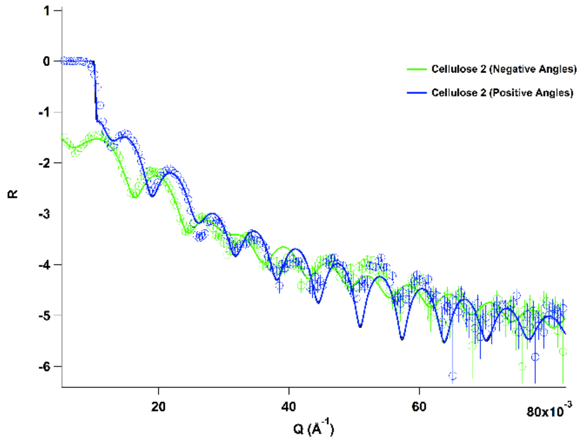

Figure 3. NR curves for Cellulose 2 (positive and negative angles). The preliminary fits (solid lines) are done with a 7-layer model.

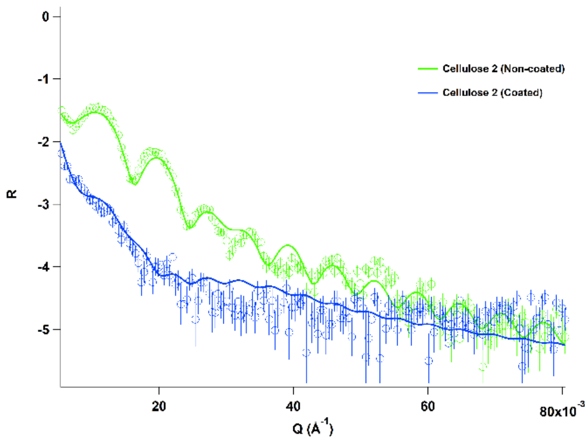

Figure 4. NR curves for non-coated and coated Cellulose 2 (negative angles).

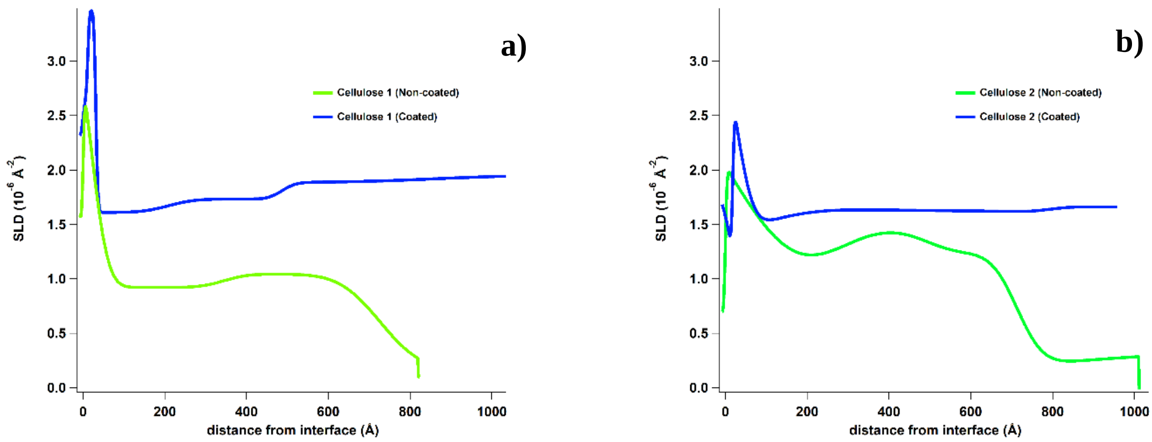

Figure 5. SLD values obtained from the fit for Cellulose 1 (a) and Cellulose 2 (b) (negative angles only).

The same procedure was followed for Cellulose 2 (Fig. 3 and Fig. 4), where the SLD values obtained (Fig. 5b) required the same fitting steps as for Cellulose 1.

The main (preliminary) conclusion from the acquired data is the increment of the average thickness and roughness from the non-coated cellulose to the coated cellulose. However, further complementary analysis will be done to improve the fits and check this tendency and its origin.

References: [1] Williams, S.R. Finishing of Wood, in Wood Handbook. 2010, General Technical Report FPL-GTR-190: Madison, WI. [2] Kamke, F.A. and J.N. Lee, Adhesive penetration in wood - a review. Wood Fiber Sci., 2007. 39: 205-220. [3] Martin C., Barker R. et al., Structural Variations in Hybrid All-Nanoparticle Gibbsite Nanoplatelet/Cellulose Nanocrystal Multilayered Films. 2017. Langmuir. 33: 7896–7907.