ESRF	**Experiment title: The gelatinization of starch in limited water and aqueous sugar solutions**	**Experiment number**: 26-02-896
Beamline: BM26B	**Date of experiment**: from: 25/07/2018 to: 30/07/2018	**Date of report**: 01/10/2018
Shifts: 9	**Local contact(s)**: HERMIDA MERINO Daniel	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Goderis Bart*

Notebaert Charlotte*

Van der Flaas Kristien*

Baeten Dorien*

Verbauwhede Brecht*

Report:

To investigate the effect of the starch concentration and the presence of sugars on starch gelatinization, model systems consisting of starch, a sugar and water with variable ratios were studied. Two types of starches were investigated in combination with glucose: maize and potato starch. Systems comprising maize starch and maltose were also considered. In total 80 different mixtures were investigated. A typical experiment consisted in heating the mixtures from 30°C to 130°C at 4°C/min. SAXS and WAXD patterns were collected over consecutive 15 s time frames, corresponding to 1 set of patterns every °C.

Native starch from the highest to the lowest level of structural organization consists of granules, amorphous and semi-crystalline growth rings, blocklets, amorphous and semi-crystalline lamellae and starch double helices within the crystalline lamellae as depicted in Figure 1. During gelatinization of starch in excess water, time resolved WAXD experiments typically reveal the loss of crystallinity via the disappearing crystalline reflections whereas in SAXS the scattering associated with the crystalline / amorphous lamellar stacking declines and finally disappears. Concomitantly, a strong signal appears in SAXS at the smallest angles, which we recently associated with the sequential melting of blocklets within the semicrystalline growth rings (Figure 2, with the black and white blocklets being in the semicrystalline and molten state respectively).

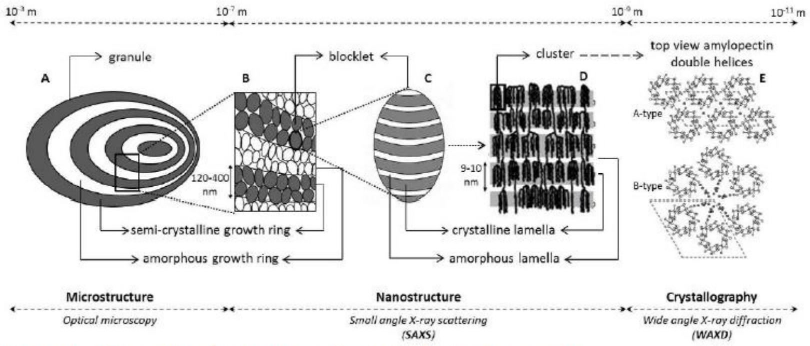

Figure 1: hierarchical structure of a typical starch granule

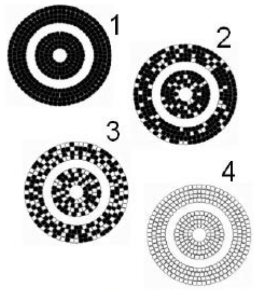

Figure 2: From 1 to 4: hypothetical starch granular breakdown during water aided gelatinization, seen at the granular and blocklet level.

In order to process the SAXS data a Matlab script was developed to interpret the patterns in terms of a superposition of semicrystalline starch stack scattering, scattering due to the presence of blocklets and a background. A typical result is shown below in Figure 3 for the SAXS patterns (multiplied by q^2) at selected temperatures for the samples composed of 25wt% maize starch in water. In Figure 4, a typical Matlab output is shown, illustrating the SAXS data fitting over the entire temperature range. Further data processing and interpretation is in progress.

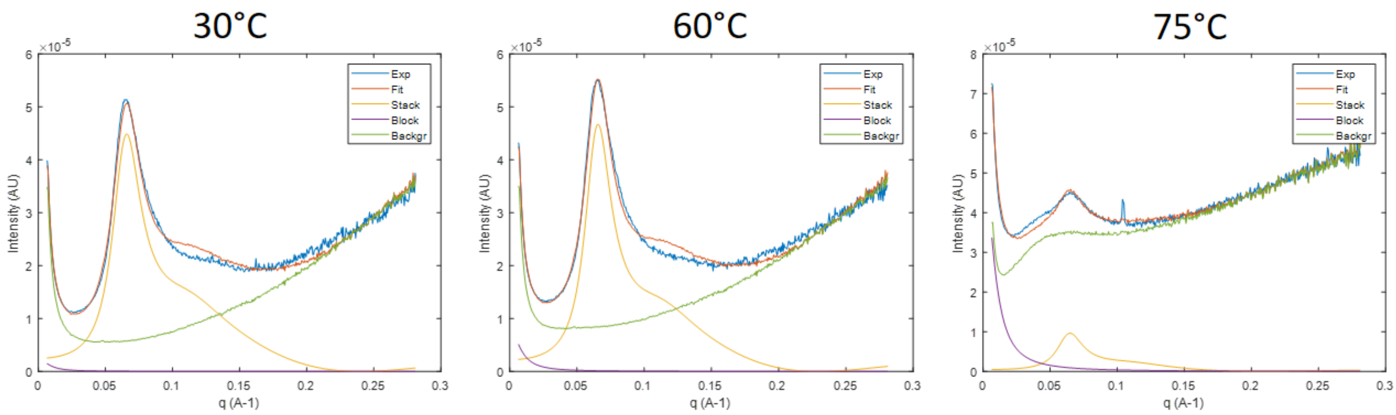

Figure 3: SAXS patterns of the blends consisting of 25 wt% maize starch in water at selected temperatures and interpreted in terms of a superposition of blocklet, stack and background scattering

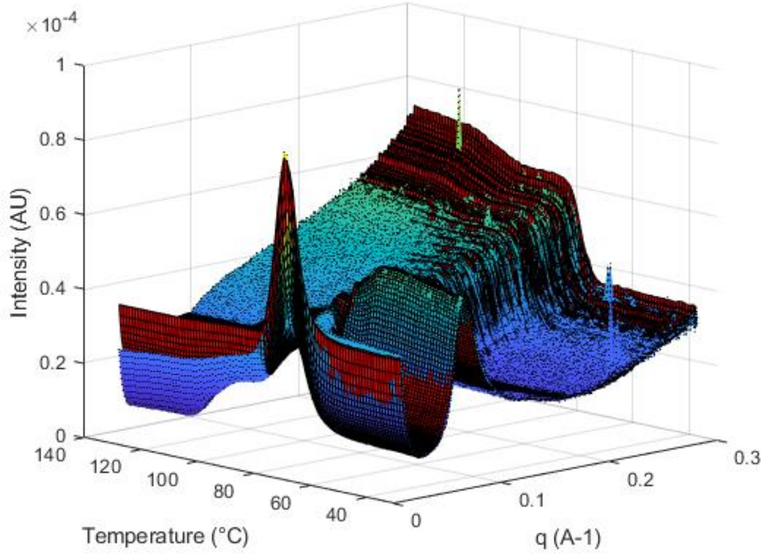

Figure 4: SAXS patterns (blue) of the blends consisting of 25 wt% maize starch in water together with theoretical patterns (fits) in red based on a model that assumes a superposition of blocklet, stack and background scattering