Experimental report

23/11/2022

Proposal: 9-12-663 **Council:** 4/2021

Title: On the internal structure of modified starch particles.

Research area: Soft condensed matter

This proposal is a resubmission of 9-10-1705

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Samples: Starch

Instrument	Requested days	Allocated days	From	To
D11	4	2	02/10/2021	04/10/2021

Abstract:

Starch particles have been used to stabilize O/W food emulsions. This is because starch is a naturally occurring polysaccharide that is safe to use in foods and because it is abundant, biodegradable and inexpensive. Native (non-modified) starch, however, has limitations in its applications, due to its hydrophilic surface properties, which makes it less suitable as a stabilizer. Starch particles modified by esterification with dicarboxylic acids to give octenyl succinic anhydride (OSA) starch is an approved food additive that can be used to stabilize oil in water emulsions used in foods and drinks. In this study, we plan to make use of deuterated octenyl succinic anhydride and isotopic contrasts to locate the modification. Evaluation of data will be formed using core shell models with SASView. This knowledge is essential for the understanding the interfacial behavior of the starch particles.

Exp. 9-12-663 "On the internal structure of modified starch particles"

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In this experiment we utilised SANS on D11 to investigate how the degree of OSA substitution influenced the internal structure of quinoa and rice starches.

All measurements were performed on 5 weight % starch dispersions at detector distances of 1.7 m, 10.5 m, and 38.0 m to cover a q range from 0.46 $Å^{-1}$ down to 0.00046 $Å^{-1}$. The starch dispersions were constantly rotated throughout the experiment to avoid sedimentation.

We prepared quinoa starch dispersions at several D_2O/H_2O contrasts to determine the starch SLD match point. The water compositions selected provided a wide range of contrast, with one sample very close to the contrast match point and a number on both the H-rich and D-rich sides. The contrast match point for waxy maize starch has previously been ascertained to be approximately 53% D_2O. Subsequent measurements were performed on quinoa and rice starches with no substitution followed by 0.6, 1.2, 1.8, 2.4, and 3.0 % OSA substitution in three solvent contrasts (pure D_2O, pure H_2O, and 55%D_2O-45%H_2O).

While we can clearly see variations in the results as a function of OSA substitution, particularly around the broad peak situated at ~0.065 $Å^{-1}$, a follow up experiment utilising deuterated OSA for could be significantly more informative, especially relating to the exact location (core vs shell) of the substituted molecules.

Figure 1 presents the results for the quinoa series in D_2O while Figure 2 shows the results for the rice series in D_2O.

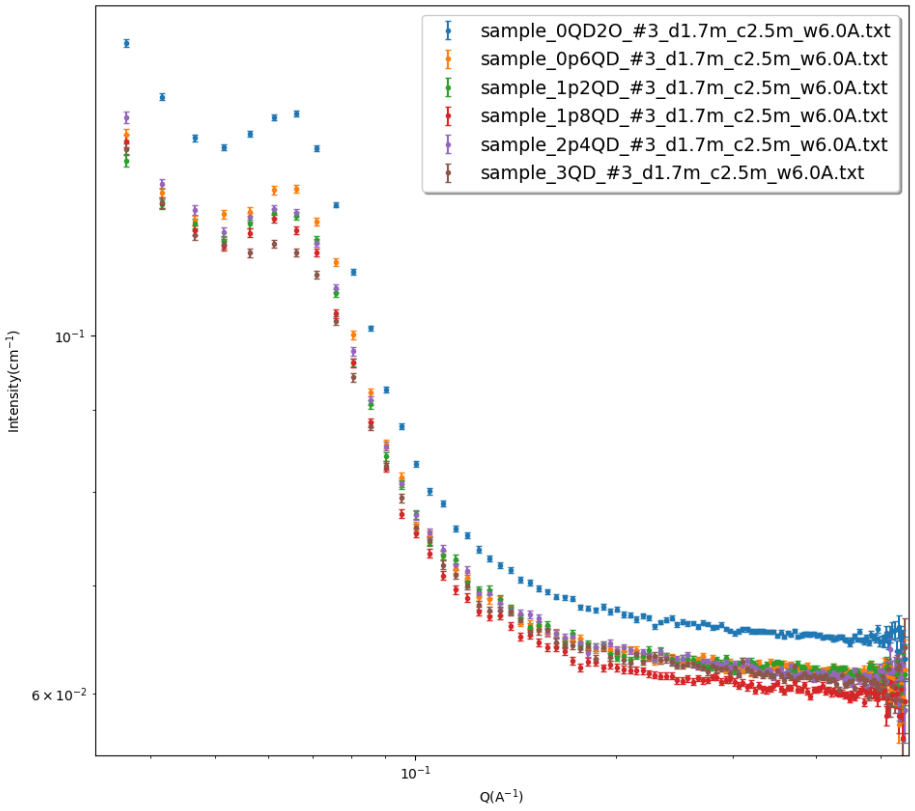

Figure 1. SANS results for 5 wt% Quinoa dispersions in D_2O as a function of OSA substitution at a detector distance of 1.7 m.

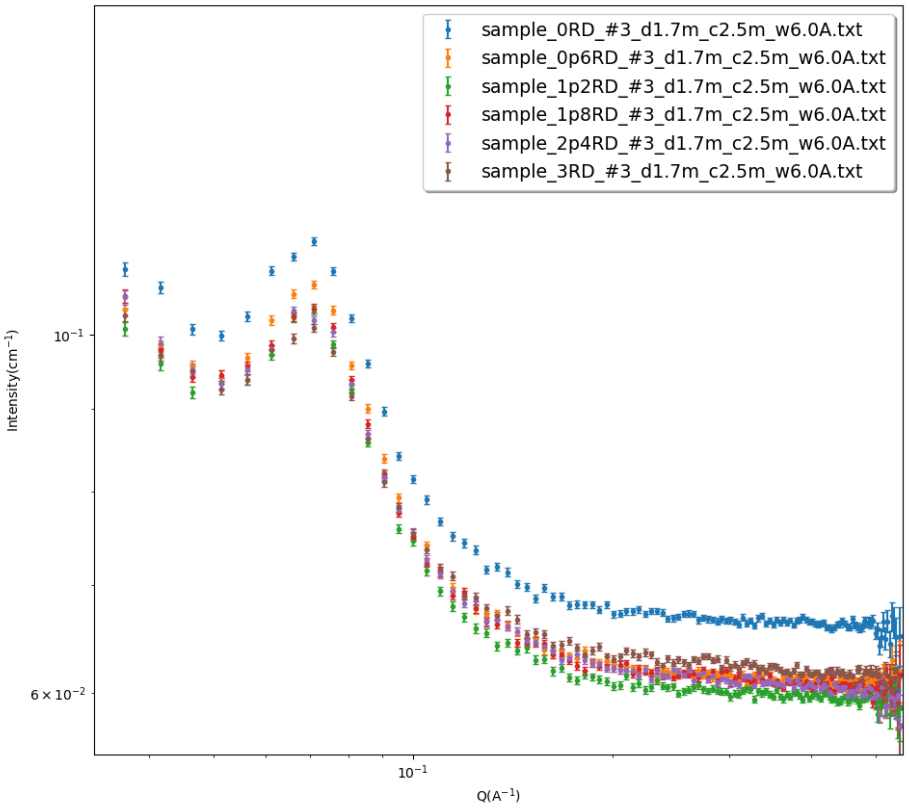

Figure 2. SANS results for 5 wt% Rice dispersions in D_2O as a function of OSA substitution at a detector distance of 1.7 m.

While measurements were performed at the three detector distances, and the raw data looked good whilst performing the measurements, after subtracting the background for the empty cell or solvent the error at the high q region for each detector distance made stitching the data complicated. Also, the location of the stich between the 1.7 m and 10.5 m distances was at a significant region of interest and therefore not ideal. If this experiment was to be repeated, greater overlap between q ranges (easier to stitch data) and better choice of detector distances (avoiding stitch around the peak at ~0.065 $Å^{-1}$) would be beneficial as well as increased count times to reduce the error bars.

The next step is to assess whether it is worthwhile removing measurements with large error and manually stitching the three detector distances together. However, this is unlikely to reveal any significant variations in scattering at lower q values than that presented in Figures 1 and 2 from detector distance of 1.7 m as there is very little variation in contrast observed as a function if OSA substitution