Experimental report

09/08/2019

Proposal: **CRG-2538**

Council: 4/2018

Title: Polysaccharide dynamics in rehydrated freeze dried chia seed mucilage

Research area:

This proposal is a new proposal

Main proposer: **Judith PETERS**

Experimental team: Fanni JURANYI
Judith PETERS

Local contacts: Judith PETERS

Samples: chia seed mucilage in cryogel form

Instrument	Requested days	Allocated days	From	To
IN13	10	10	08/10/2018	19/10/2018

Abstract:

Polysaccharide dynamics in rehydrated freeze dried chia seed mucilage

Abstract: Mucilage is a natural polysaccharide hydrogel, which is vital for plants and relevant for industry (mainly food and pharmacy) due to its water binding property. Here we would like to complete our study on freezedried chia seed mucilage, which was rehydrated with D2O. The large q range of IN13 will provide details of the localised polymer diffusion. Additionally, the slightly better energy resolution in comparison to MARS (PSI) is expected to verify the compelte mobility of the system. For this we ask 10 days on IN13 to perform QENS and FWS measurements.

Setup: Elastic position is at 3.5 mueV, reactor 51MW, for Empty, EC and Van 35MW
λ_i=2.23 Å (16.45 meV); CaF_2(422) mono&ana, displex (no HT)
sample: chia_2017_14_100RH_D2O h=0.7218, m_dry=0.1625g, m_D2O=0.1173 (T=93.15%**)**
sample holder: cylindrical Alu can, d_i=22mm, fill h=~45mm
Vanadium: V can and 8.66 g foil spiral, slightly narrower (can: d_o=20.5mm, d_i≈20.0mm, estimated weight eqivalent = 4.78g, estimated T =84.8%, measured one: 85.7%)

Elastic scan has been performed in the temperature range between 20 and 300K during heating. Empty instrument and empty can runs could be added and used as background. Alu Bp range was masked. For Vanadium only QENS spectrum was measured. For the normalization the values at the elastic position (at 3.5μeV) were extracted. Spectra of back and forth scan was shifted by 1.73μeV. For almost all detectors the ratio of the elastic intensity between the two scan directions was deviating less than 15% from unity.

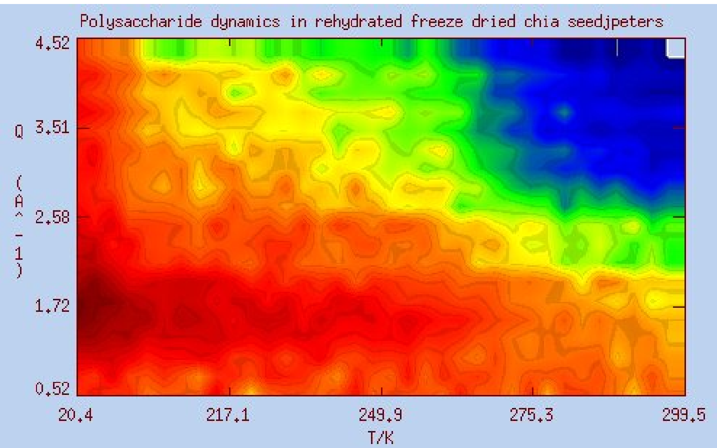

Fig. 1: FWS of chia mucilage hydrated with D2O. Intensity is normalized to Vanadium.

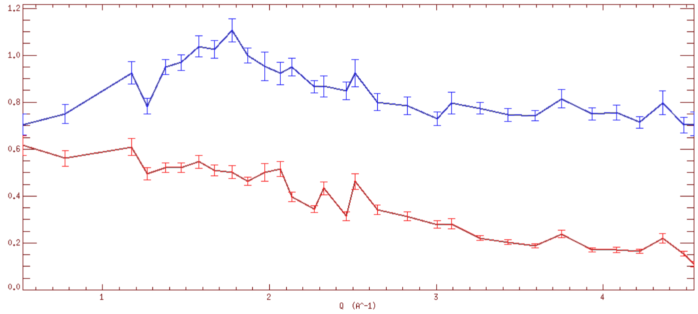

Fig. 2: Elastic intensity at 20K (blue) and at 300K(red).

At low temperature the intensity is higher in the range of q=1.3-2.2$Å^{-1}$ due to the ice Bragg peaks [Fig. 1.]. Melting is observed as a stepwise intensity decrease between ≈260-275K, not only in the Bragg peak range, but in the whole q range. This is in agreement with our older data from MARS at PSI, where we have observed that the polymer motions started simultaneously with the water melting (the sample in that case was higher hydrated). Additionally, a q dependent intensity decrease is visible, which is caused by the Debye-Waller factor.

Unfortunately it is not possible to extract a reliable MSD due to the extended q range of the coherent scattering [Fig. 2.]. The spectrum is similar to the one of pure D_2O measured at IN13 (see. Fig. SI 3 in [1]).The same sample measured at FOCUS, PSI, with λ_i=6Å shows clear ice Bragg peaks and a broad bump below.

QENS has been performed using 0.33 µeV/min scan rate of the monochromator. Measurement at 300K between ±90µeV, with additional data collection between ±50µeV took 3.7 days, whereas at 20K between ±90µeV 27h. Vanadium has been measured in a reduced energy range between -40 and 30µeV during 44h, which is almost the same time per energy points as in case of the sample. The data was reduced by LAMP.

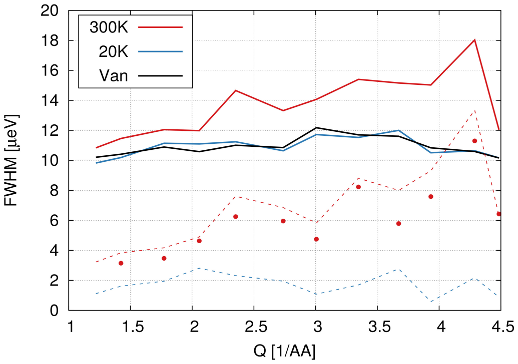

Fig. 3: Single Gaussian fit:width of the elastic line, without (solid line) and with (dashed line, dots) using Van as resolution. For dots the E range was limited to ±10µeV. Statistical error is not repressentative, it is much smaller than the scattering of the values.

Since the intensity contribution from the sample holder was negligible in case of the FWS setup, and the 20K data was constant apart of the elastic peak, the background was not measured with QENS. Instead, a constant was subtracted: in case of Van the intensity was averaged between -40 and -30µeV, in case of the 20K data the spectra were fitted by a Gaussian and a constant, and this q dependent constant was used as flat background. At 300K the same flat background was used as @20K, since we know that QENS spreads over the whole energy range. The spectra has been normalised to the integrated Vanadium after flat background was subtracted from both. The energy scale was corrected by shifting the channels. To avoid any artefact, it has been done in all possible manner. Data was binned with dQ=0.2$Å^{-1}$ & dE=2 µeV. Fit was performed using the PAN module of DAVE (from NIST).

Unfortunately, QENS was not observable at 300K, most likely due to the high background and limited data quality. The q dependent intensity summed over E outside the elastic line leads to the same conclusion. Therefore the main aim of the proposal to determine the EISF over an extended Q range failed unfortunately.

A second purpose was to verify the broadening of the elastic line, which was indicated during previous measurements (MARS, PSI dE≈13µeV). Alone this could be better done at a cold backscattering instrument, but as a byproduct it was possible to show this at IN13.

As first all spectra are fitted with a single Gaussian without using a resolution (Fig. 4). The fit is not very good, the asymmetrical shape results in an increased line broadening (see worst case scenario at Fig. 3.) The resolution was found to be ≈11µeV.

The 300K data shows increasing line broadening, contrary to Vanadium and the 20K data, which is even clearer, if the model fit function is convoluted with the resolution function (Fig. 3.). The fit was repeated in a restricted energy range of ±10µeV (Fig. 3, dots), where the line shape does not deviate from a Gaussian in case of the Vanadium. The broadening is still significant, i.e. we detect a slow dynamics.

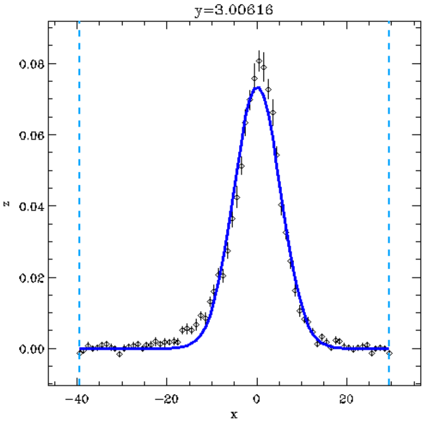

Fig. 4: Fit of the Vanadium spectrum, q=3Å-1 with a Gaussian.

In summary, it was not possible to explore the geometry of the localised polymer motion (FWHM=120µeV, known from complementary experiment), because of limited signal quality. Nevertheless, the presence of a slow motion of the whole polymer could be proven, even if the fit parameters are quite unprecise.

[1] M. Zanatta et al.: Sci. Adv. **4** (2018) eaat5895