INDUSTRIAL PILOT PROJECT

Development of encapsulation technology enabled with synchrotron X-ray scattering

THE INDUSTRIAL CHALLENGE

Spexímo AB develops oil-in-water emulsions by utilizing Pickering properties of quinoa starch. With starch granules as a barrier between oil-water phase, such Pickering emulsions have great possibilities to encapsulate active ingredients. The encapsulation is improved by creating a gelatinized starch barrier under heat treatment (Figure 1). However, no detailed studies of the structural properties of quinoa starch and the effect of differently modified starch entities have been performed in the past. This would help understanding Spexímo encapsulations in detail.

WHY USING A LARGE SCALE FACILITY

By using the high photon flux from a synchrotron beamline, it is possible to map a broad range of samples (different in composition, treatment, formulation) in short time. The two scattering techniques used also enables analysis of structural changes *in situ* and provide quantitative information not possible to obtain with other methods.

HOW THE WORK WAS DONE

In the project both native and modified (technically and chemically) quinoa starches were studied in different formulations. The same samples were sequentially analysed by two scattering techniques, Small-Angle X-ray Scattering (SAXS) and Wide-Angle X-ray Scattering (WAXS). The structure of starch in dispersion, emulsion, and encapsulations was studied, including the effect of storage. The experiments were performed at the SWING beamline at SOLEIL synchrotron in Paris using 1Å wavelength and 2 sample-detector distances (WAXS:521mm, SAXS:6235 mm) covering the continuous q-range 0.001 to 1.900 $Å^{-1}$. Quinoa starch dispersions, emulsions and encapsulation formulations were prepared and treated at different temperatures at simulated process conditions and *in situ* (samples mounted in capillaries at the beamline in Figure 2).

Complementary information was obtained using differential scanning calorimetry, light scattering and microscopy.

THE RESULTS AND EXPECTED IMPACT

The synchrotron experiment provided novel information on the molecular organization of quinoa starch as a function of both composition and treatment. The lamellar structure, studied by SAXS, was shown to be similar in dispersions and emulsions for differently modified starches. The degree of crystallinity, studied with WAXS, decreased with thermal treatment for all starches, with slight difference in melting rate between starches. Starch retained its properties when stored in wet and dry formulations. The obtained structural results will be very useful for Spexímo in further product development. This project and the collaboration with MAX IV laboratory resulted in increased competence of the industrial partner using synchrotron SAXS/WAXS techniques and analysis of data towards future utilization of the large-scale photon infrastructures.

"The X-ray scattering methods shed new light on the properties of Spexímo encapsulations"*/Nina Filenko, Project Manager at Spexímo*

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