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Igor Bodnár
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Introduction

Biopolymer films, like films made out of proteins, show interesting behaviour for applications in food and pharma. They can be insoluble, semi-permeable, and are food-grade. In order to get a better insight into the micro-structural organization, and its consequences on the mechanical and permeability properties, different series of films have been prepared, where the following parameters have been varied: pH, amount of plasticizer, type of plasitcizer, and enzymatic crosslinking.

Materials and Methods

Films were prepared by casting whey protein aggregate solutions in combination with plasticizer. The whey protein aggregates were created by heating solutions of 8% whey protein isolate for 30 minutes at 80 °C. After casting the films were left to dry over several days at ambient conditions.
From the films samples of 1 cm were punched out. The samples were measured using the ID-26 beamline (DUBBLE) and the SAXS-equipment, with the detector at 8 m or 5½ m. Measuring times varied from 180 seconds to 10 minutes. The data were corrected for the background, the instrument characteristics and subsequently radially averaged.

Preliminary Results

First results show that the level of glycerol induces microstructural changes on short length scales (high q-values).

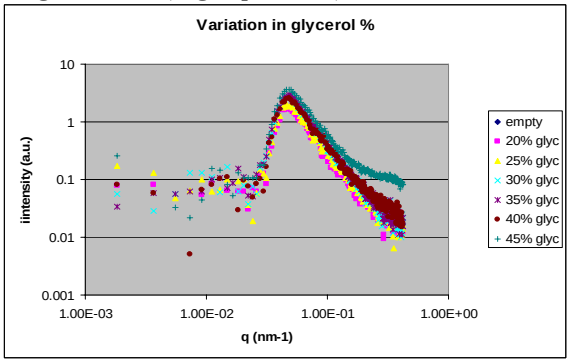

Figure 1. Influence of increasing amount of glycerol only becomes noticeable at high q-values, indicating short length scales.

Furthermore it becomes apparent that certain additives, like starch and methylcellulose (MC), have different effects on the organization of the films. Whereas the addition of starch leads the a different scattering pattern, the addition of MC does not have a significant effect withi nthe q-range probed here.

Furthermore, experiments with extra crosslinking using transglutaminase show that, although microstructural changes take place, these changes are not picked up by SAXS. It is likely to assume that the crosslinking thus only changes the organization on a length scale smaller than measured by SAXS.

The most interesting results are obtained for the series of films with varying glycerol concentration, that were soaked in water before the measurements. In these cases a ring pattern is observed, indicating a dominant preferent lengthscale in the system. It seems that the position of this ring is dependent on the amount of plasticizer used. This could give indications to help understand the permeability of the films in dry and in wet conditions.

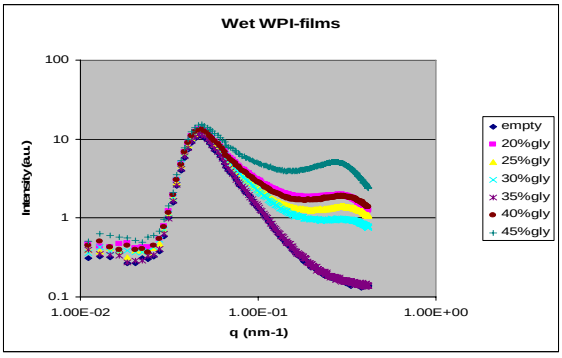

Figure 2. When wet films are measured, a distinct scattering peak becomes visible in the high q-range. The coinciding length scale could be correlated to a dominant length scale, originating from phase separation or charateristic mesh size of the network.

Conclusions

SAXS can be a powerful tool in understanding microstructural changes in protein films. Combination with mechanical measurements leads to the understanding of the origin of different behaviour: whether this is on a molecular level (not directly detectable with SAXS) or mesoscopic level (giving rise to different scattering profiles using SAXS).

Specifically the appearance of a scattering ring in wet films opens up possibilities to investigate the structural changes in protein films under environmental interesting conditions. It is proposed to continue these investigations, where the scattering experiments can be performed at different levels of swelling of the films. Very recently it has been shown in our lab that the swelling behaviour is very strongly dependent on the pH of the aqueous solution in which the film is being soaked.