DMA Analysis of Biopolymer Film Swelling

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ABSTRACT

To be able to measure swelling and the kinetics of swelling of biopolymer films, a new measurement cell, to be used in a DMA instrument, has been developed. The cell makes it possible to add a fluid to the surroundings of a sample during a mechanical measurement. The new cell has been used to determine the swelling of starch-based films in water.

INTRODUCTION

The moisture sensitivity of biopolymer films is usually assessed through the water vapour permeability, which does not give the full picture of water sensitivity. It merely reflects the early plasticization and not the swelling. Other common methods used to measure water absorption in plastics are often designed so that the plastic sample is submerged in water for a certain amount of time and then wiped off and weighed, e.g. [ASTM D 570 – 98]. Such methods are not sufficient for biopolymer films, such as amylopectin films, which dissolve quickly when immersed in water, and they do not give information on the kinetics of moisture uptake and swelling.

The aim of the present study was to develop a method to measure kinetics of water uptake and magnitude of swelling in biopolymer films.

METHODS

A new cell to mount in a DMA instrument has been developed. In constant stress mode of the DMA instrument (Rheometric Scientific RSA-II, Piscataway, NJ, USA) the strain is measured over time, during the entire procedure from ambient conditions, to the filmstrip being completely surrounded by liquid. The magnitude of strain will increase when the fluid is added. The magnitude of strain and the change of strain over time give information of the swelling and the swelling kinetics of the filmstrip. Measurements using oscillating mode have also been performed, where it is possible to analyse the dynamics of water uptake by monitoring the change in modulus when the films are subjected to water.

DMA – CELL

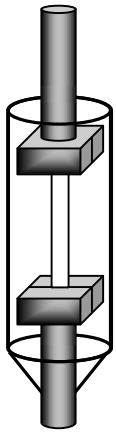

Figure 1. A filmstrip mounted in the grips, surrounded by the liquid cell.

The cell used consists of a cylindrical, transparent plastic cover, which is placed around the lower film grip, se Fig. 1. The film can easily be mounted in the grips where after the plastic cover is raised to its upright position and secured. After a desired time of measuring the fluid is added to the cell, covering the entire filmstrip. The clear plastic cover allows the operator to observe the filmstrip, controlling that it is not fractured or flexed.

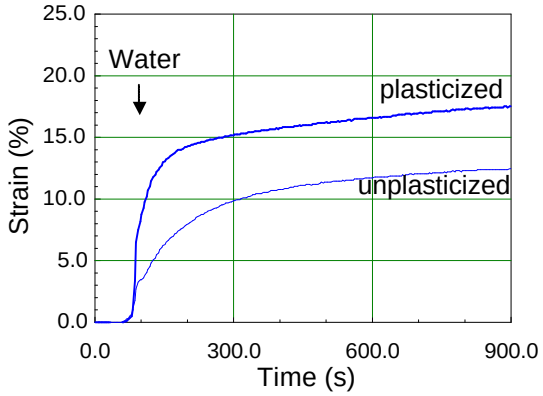

Figure 2. Films from pea starch. The upper line is from a plastiziced sample with glycerol, glycerol/starch=0.30 and the lower line is from an unplastizised sample. The DMA apparatus is used in static mode and the strain is recorded over time.

RESULTS

In Fig. 2, a filmstrip 30 mm long and 4 mm wide is used. The thickness prior to the swelling analysis was 0.1 mm. Strain was recorded over time and the swelling of two films from pea starch, one unplasticized and one plasticized with glycerol/ starch=0.30 are presented in Fig. 2. Water was added after 60s during both measurements. Fig. 2 shows that the plasticized film swells faster and to a higher magnitude than the unplasticized film.

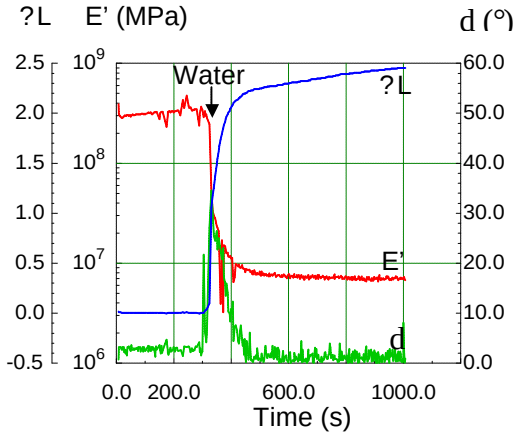

Figure 3. Film of pea starch plasticized with glycerol, glycerol/starch=0.30. DMA apparatus used in oscillating mode.

Fig. 3 also shows the swelling of a film of pea starch plasticized with glycerol, glycerol/starch=0.30. The dimensions of the film were the same as previous. The measurement was in oscillating mode and the water was added after 300 s. The drop in storage modulus, E', and the elongation, ? L, show that initial plasticization and swelling is fast but continues for 10 minutes. The pronounced peak in the phase angle, d, is similar to the behaviour at glass transition and gelation.

CONCLUSIONS

Measurements of swelling in the DMA instrument works satisfactory.

Further work in oscillating mode is needed to include inertia compensation for the water added.

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