Characterization of Gelatinization of Starches Using the Pyris 6 DSC

W.J. Sichina, International Marketing Manager

Introduction

Starch is a biopolymer comprised on amylose and amylopectin. Starches are found in corn, rice, potatoes, barley and wheat. The end use characteristics of starches, including cooking, brewing, textural and digestive properties, can be significantly affected by the physical transformation known as gelatinization. This refers to the process by which a starch/water system undergoes an order-disorder transition during heating [1]. It is hypothesized that the starch loses its crystallinity during gelatinization, and that the process is analogous to melting. Gelatinization is an important physical phenomenon, which provides information about the nature of the given starch. Critical parameters associated with the gelatinization of starches include the onset temperature, the peak temperature and the temperature range over which gelatinization occurs along with the heat of gelatinization.

Another important factor in the cooking and digestibility properties of starches is the fat/amylose complexes found in the starch.

It is desired to have an easy, convenient means of characterizing the gelatinization process and the fat/amylose interactions of starches, because of their known impact on the cooking, digestive and textural properties of food products containing starches.

DSC

Differential scanning calorimetry (DSC) is a very powerful tool for the study of starches given its high sensitivity and ease of use. For the successful characterization of starches, it is important to have a high performance DSC instrument, as the critical transitions associated with starches are very weak and can be difficult to detect.

The high performance PerkinElmer Pyris 6 DSC provides an ideal means of characterizing the properties of starches and foods as the instrument offers the following important features:

- high sensitivity for the detection of very weak transitions
- high resolution
- stable performance for reliable and reproducible measurements
- sealed, large volume DSC sample containers for the complete retention of water without rupturing or leakage
- built in gas switching accessory and flow regulator for convenience
- ease of use with Pyris Software for Windows

Pyris 6 DSC

The heat flux design of the Pyris 6 DSC coupled with its low mass furnace provides for outstanding baseline stability or reproducibility. This is important to ensure that consistent results are obtained for the gelatinization process of starches.

The characterization of the thermal properties of starch - water formulations necessitates that the mixture needs to be analyzed in a sealed sample pan, to prevent the loss of water from the formulation during heating. The Perkin Elmer DSC stainless steel sealed sample pans (PN 0497-8995 for 100) are ideal for this purpose. These pans have the additional valuable feature of holding a large volume of sample (60 µL), which is useful for starch systems as the transitions are relatively weak.

PerkinElmer™ instruments.

Experimental

Three samples of powdered rice starch were characterized for gelatinization using DSC. The following experimental conditions were utilized.

Experimental Conditions	
Instrument	Pyris 6 DSC
Sample pan	Stainless steel sealed capsules
Sample mass	10 mg starch with 25 µL of water
Reference	25 µL of water
Heating rate	5 C/min
Initial temperature	25 C
Final temperature	120 C

The DSC was calibrated for temperature and enthalpic responses using high purity indium metal.

A 25 µL quantity of water was used as the reference to counterbalance the mass of water on the sample side. This makes the DSC more sensitive to the properties of the 'solute' or starch in the starch-water sample slurry. The samples were prepared by weighing out the powdered starch directly into the pan and then adding 25 µL of water using an injectable pipette.

Results

Displayed in Figure 1 are the DSC results generated on rice starch sample E. The plot shows the normalized DSC heat flow (W/g of starch) and an endothermic event is oriented upwards.

The sample yields a well defined endothermic peak at 80.3 C which reflects the melting of the starch or the gelatinization event. The onset of the transition occurs at 73.7 C and a high temperature shoulder is obtained at 96.9 C. The total heat of transition is 13.7 J/g (based on the sample mass of the starch).

These are the critical parameters associated with the gelatinization of the starch material.

The use of the stainless steel capsules coupled with the high performance Pyris 6 DSC yields outstanding results on the relatively weak or low energy gelatinization transition.

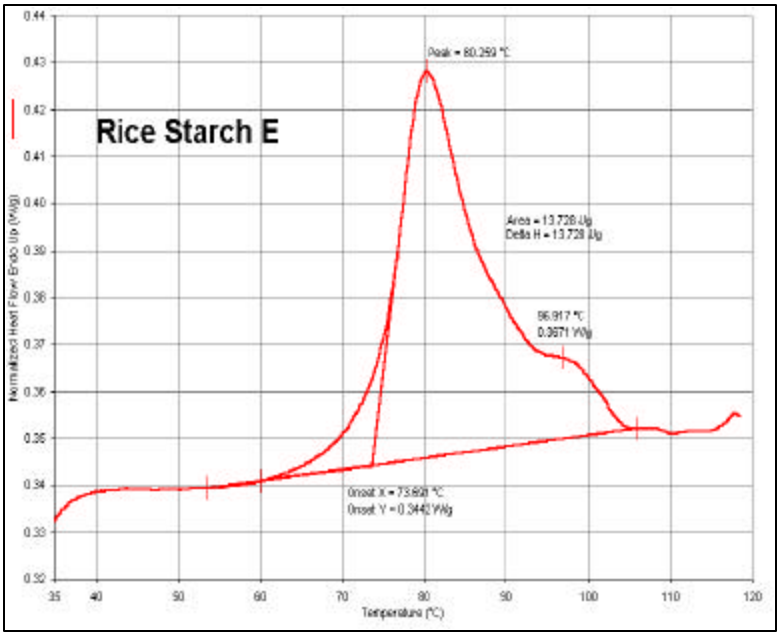

Figure 1. DSC results on the gelatinization of rice starch E

PerkinElmer™ instruments.

Displayed in Figure 2 are the DSC results obtained on rice starch sample D. These results are similar to those generated for sample E; but there are some significant differences. The peak temperature for starch D occurs at 78.1 C, which is 2 C lower than that for E. The onset temperature for rice sample D is lower than that of E (72.7 versus 73.7 C). The shapes of the main gelatinization peaks are also distinctly different for samples D and E where D has a more symmetrical shape.

The differences between the rice starch samples are more apparent in an overlay plot and this is displayed in Figure 3. It may be seen that starch sample D has a significantly more intense and narrower gelatinization transition as compared to sample E.

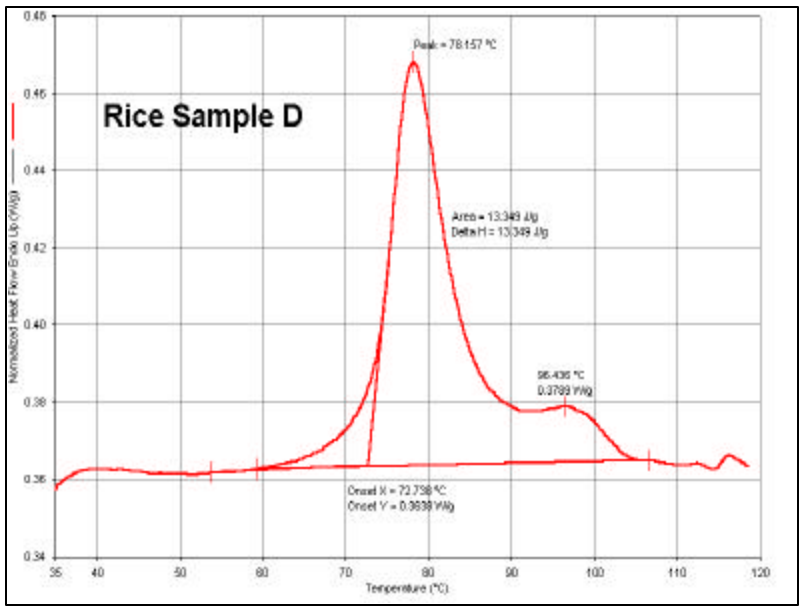

Figure 2. DSC results on gelatinization of rice starch D

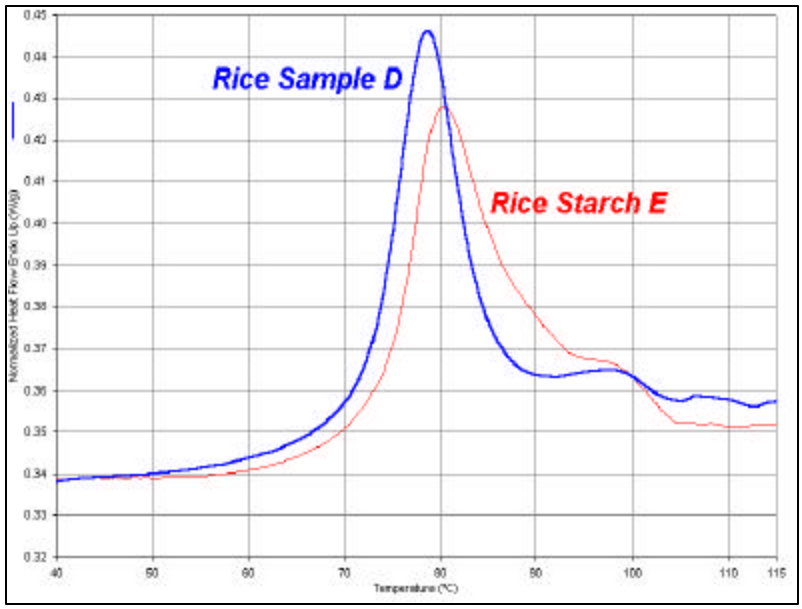

Figure 3. Overlay of DSC results on starch samples D and E

Summary

The PerkinElmer Pyris 6 DSC yields outstanding results on the gelatinization transition of starches. The high sensitivity and outstanding stability of the instrument, coupled with the use of the special large volume stainless steel sealed capsules provides excellent results on starch - water slurries. The DSC gelatinization data provides essential parameters, which are relatable to the end use properties of the starches.

Reference

[1] Hau Liu, John Lelievre and Wendy Ayoung-Chee, *Carbohydrate Research*, 210, 1991.

PerkinElmer™ instruments.