



## TA Instruments – Application Note

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### **Thermal Analysis of Starch Gelatinization**

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#### **Abstract**

Starch is a carbohydrate present in foods such as corn, wheat, and rice, which are important staples in the human diet. Although the composition of starch in these three grains has a different morphology, utilizing differential scanning calorimetry (DSC) to study each type of starch has been shown to be a powerful tool in understanding important structural characteristics of the starch that is present. DSC measures the heat flow associated with structure and changes in structure as a function of time and temperature in a controlled atmosphere. The two most frequent thermal signatures that are studied for rice, as well as other grains, are starch gelatinization and retrogradation. In this study, a Multi-Cell DSC (MC-DSC) was used to study the gelatinization thermal signature of rice starch.

#### **Introduction**

Apart from being directly consumed, rice is also a major component of many cereals, snack foods, and beverages such as sake and beer. Each different food application requires different preparation and treatment of the starch. Some food scientists are also trying to find a balance between preparing rice while conserving the nutritional benefits of the grain. One quality worth preserving is resistant starch, more commonly known as fiber. Understanding and controlling the degradation of this polysaccharide is important since a high fiber diet has been speculated to be a possible treatment for diabetes (1).

Starch is made up of two components: the linear polymer amylose and branched polymer amylopectin (1). When heated, starch becomes soluble and loses its semi-crystalline structure (2). It is during heating that the intermolecular bonds of starch are broken down in the presence of water, enabling hydrogen bonding and engaging more water into the complex (3). During gelatinization water acts as a plasticizer, influences the transition ( $T_p$ ), which in this case the minimum temperature in the gelatinization event in the thermogram (4). This study analyzed several different types of rice available at a local grocery store. In the first study, microwave ready parboiled rice is used and in the second study, the thermal signatures of three different types of traditional rice are evaluated.



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### Experimental

An MC-DSC available from TA Instruments was used to analyze the thermal properties of rice starch. In each experiment approximately 10-20 grains, or 0.300 grams, of rice were placed into the instrument in an individual sealable Hastelloy ampoule. The microwaveable rice was treated by the manufacturer and additional water is not required. In the second study an equivalent mass of water was also added to the ampoule. The instrument was set to equilibrate at 25 °C and perform a scan from 25 to 130 °C or 160 °C at 2 °C/min. Data analysis on both data sets, including background subtractions, were done using NanoAnalyze™.

### Results and Discussion

Figure 1 shows the data from the first experiment and displays the important transitions part of the complete thermogram for the 25 – 160 °C scan of the starch in microwaveable rice sample. The first endothermic peak in the thermogram is that of the gelatinization of the starch in the rice. The data indicates the gelatinization of the rice sample has a  $T_p$  of 66.8 °C, an onset temperature ( $T_o$ ) of 56.3 °C and concluding temperature ( $T_c$ ) of 79.1 °C. The enthalpy of gelatinization, 3.6 J/kg, was determined by fitting a 2<sup>nd</sup> order polynomial baseline to the data and then integrating the area under the peak and normalizing it to the mass of the sample. The second endothermic peak with a minimum at 108.8 °C corresponds to an amylase-lipid complex that has been observed at high temperatures, above 100 °C, in other rice studies (3,5).

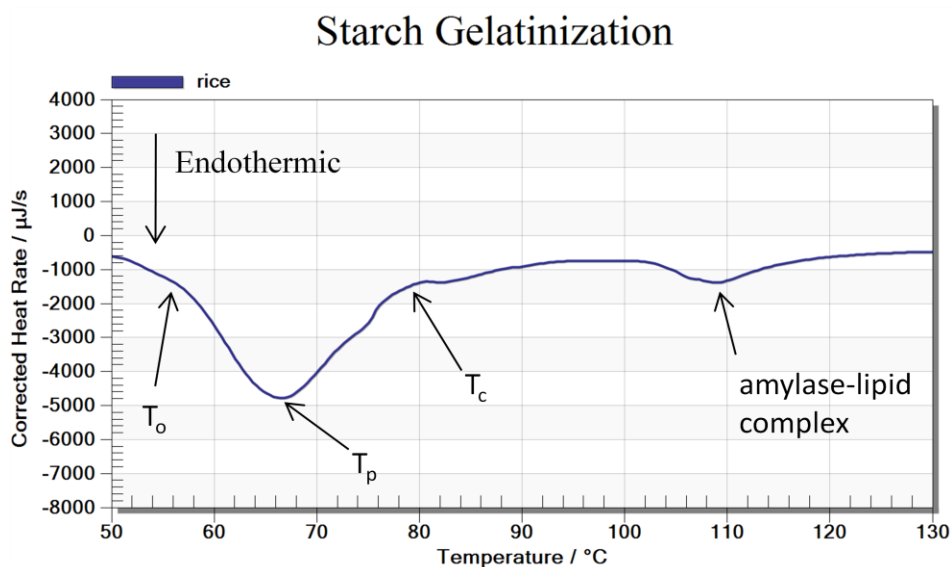
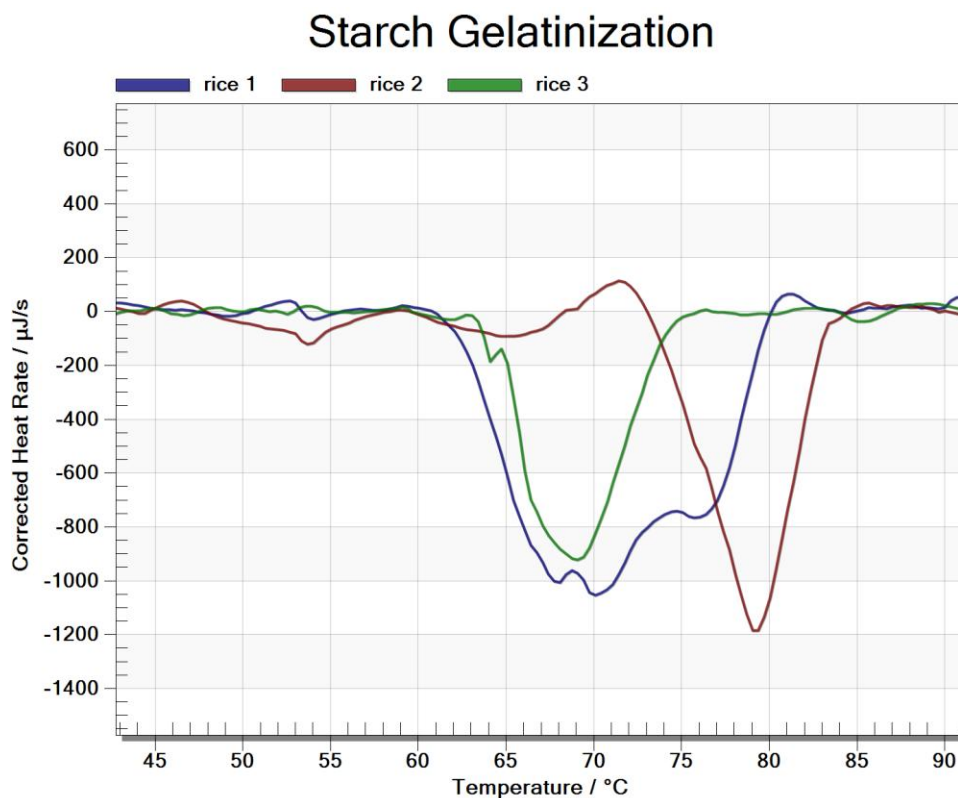


Figure 1. DSC thermogram of a parboiled rice sample.



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Figure 2 shows the data from the second multi-sample experiment and demonstrates how the instrument can be used to assay three different samples at one time. In this study, three types of rice were evaluated and upon examining the thermogram, it is shown that each rice type has a different gelatinization temperature and temperature range for the gelatinization event, indicating the different gelatinization properties of each rice type and the homogeneity of each individual sample.



**Figure 2. DSC thermogram of three different rice samples.**

Retrogradation is usually observed when the rice is cooled or stored cooled and then heated again (2). In a DSC scan of starch following cold storage, the thermal transition is usually an exothermic event. The DSC determination of retrogradation on the rice sample in this study was not performed.



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### Summary

A DSC thermogram of the starch present in rice is a powerful tool in understanding the structural characteristics of this important component of rice. The ability to predict palatability and shelf-life has been shown to be directly attributable to understanding the gelatinization process. In this study the MC-DSC from TA Instruments was successfully used to study the gelatinization of starch in rice. The data generated using the MC-DSC showed the typical endothermic gelatinization signal for rice. The thermodynamic values generated during data analysis were consistent for this type of transition seen in previous publications. There are three major advantages to collecting this type of data on the MC-DSC. First, up to three separate rice samples and one reference sample can be run simultaneously. Second, inert Hastelloy ampoules will accommodate multiple rice grains up to a maximum volume of approximately 1 cubic centimeter. Larger sample sizes allow the analysis of a more representative sample of larger batches. The sealable ampoules also allow the analysis to take place without environmental changes such as evolving gases escaping or atmosphere exchanges from outside affecting the final results. Third, the ability to run three individual samples in one single scan enables one to study many different variables simultaneously, such as water content, soaking time, grain type, and formulation steps. These advantages demonstrate that the MC-DSC could be the ideal instrument for analyzing starch gelatinization, as well as understanding other food stability formulation characteristics.

### References

1. Kemlsley, J. (2010) New fibers for foods. *Chemical and Engineering News*. **88**, 38-40.
2. Eliasson, A.C. and Gudmundsson, M. (1996) Chapter 10 Starch: physiochemical and functional aspects in *Carbohydrates in Food Chapter* ed. Eliasson, A. (Marcel Dekker, New York) pp 431-491.
3. Liu, Q. (2005) Chapter 7 Understanding starches and their role in foods in *Food Carbohydrates: Chemistry, Physical Properties and Applications* ed. Cui, S. (Taylor and Francis Group, LLC, Boca Raton, FL) pp 309-355.
4. Premavalli, K.S.; Jagannath, J.H.; Majumdar, T.K.; Bawa, A.S. (2005) Studies on phase transition in finger millet starch in relation to gelatinization. *Journal of Food Science and Technology*. **42**, 336-340.
5. Takahashi, T.; Miura, M.; Ohisa, N.; Kobayashi, S. (2005) Modifaction of gelatinization properties of rice flour by heat-treatment. *Journal of the Society of Rheology, Japan*. 2005, **33**, 81-85.