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Application News

No. T157

Thermal Analysis

Measurement of Gelatinization and Retrogradation of Starches by DSC

Carbohydrates such as rice and bread invariably contain starch. When starch is heated in the presence of water, the starch granules swell and break down, and gelatinization occurs. The "sticky" or glutinous feel of cooked rice and baked bread are due to gelatinization. As a distinctive feature of gelatinization, gelatinization behavior differs depending on the type of starch and the environment in which gelatinization occurs. Moreover, if gelatinized starch is allowed to stand, the amylose and amylopectin which exist in swollen starch granules will release water due to aggregation and rearrangement, causing the granules themselves to harden. This phenomenon is called retrogradation of starch. Because drying due to this retrogradation phenomenon is a cause of hardening of bread and rice with time, food manufacturers may take measures to retard the retrogradation rate of commercial breads and retort-type rice products.

In this article, the differences in the gelatinization characteristics of various types of starch using water and salt water and differences in the retrogradation rate of bread made with wheat starch with time were evaluated by measurement by differential scanning calorimetry (DSC).

A. Kawaguchi

Measurement of Starch Gelatinization

The gelatinization temperature of various types of starch (wheat, corn, sweet potato) using water and salt water was measured. Samples were prepared by introducing 5 mg of starch and 10 µl of water or 10 µl of water containing 10 % added table salt into sealed cells, and DSC measurements were carried out by raising the temperature to 100 °C at 10 °C/min. Fig. 1 to Fig. 3 show the results for the respective starches.

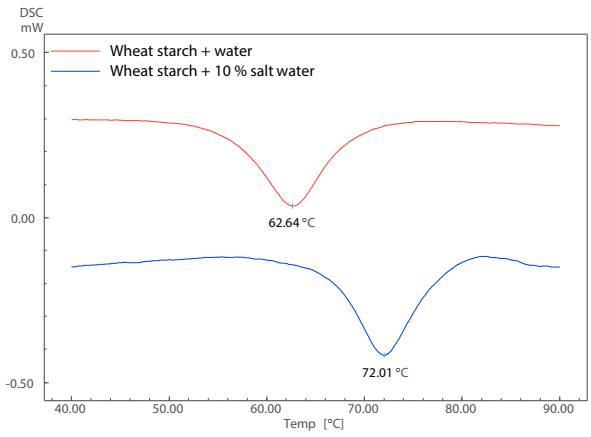

Fig. 1 DSC Measurement Results for Gelatinization of Wheat Starch

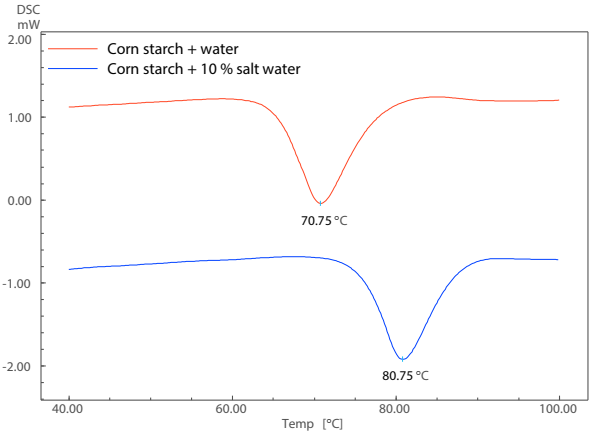

Fig. 2 DSC Measurement Results for Gelatinization of Corn Starch

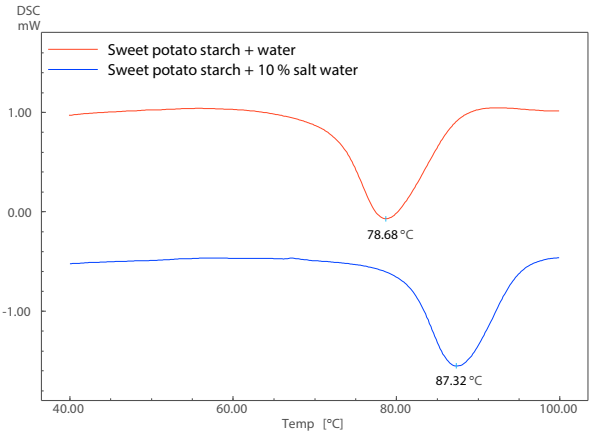

Fig. 3 DSC Measurement Results for Gelatinization of Sweet Potato Starch

The endothermic peaks of the DSC curves in Fig. 1 to Fig. 3 indicate gelatinization of the starch. Gelatinization of the wheat starch in Fig. 1 occurs at approximately 62.6 °C with the sample containing water, but at approximately 72.0 °C with the sample containing water with 10 % added salt, showing that the gelatinization temperature increases when salt is added. The same tendency was also observed with the corn starch in Fig. 2 and the sweet potato starch in Fig. 3. The order of the gelatinization temperature was wheat starch < corn starch < sweet potato starch regardless of whether salt was added or not.

As this experiment demonstrates, the gelatinization temperature of starches changes due to the effects of the type of starch and the presence of additives such as salt and sugar. The gelatinization temperature is also closely related to the heating conditions of food products that contain starch. From these measurements, it can be understood that study of those heating conditions is possible by DSC measurement.

Measurement of Starch Retrogradation in Bread

Next, starch retrogradation in bread made with wheat starch was measured. Here, samples were prepared by introducing 10 mg of bread and 10 µl of water into a sealed cell. DSC measurement was then carried out while heating the samples from 0 °C to 100 °C at 10 °C/min, and the relationship between retrogradation and the elapsed time (days) after the bread was baked was investigated. Fig. 4 to Fig. 7 show the measurement data for the bread immediately after baking and after 1 day, 5 days, and 9 days, respectively.

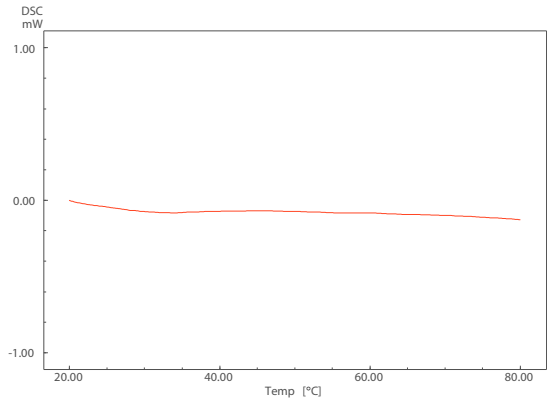

Fig. 4 DSC Measurement Results for Bread (Immediately After Baking)

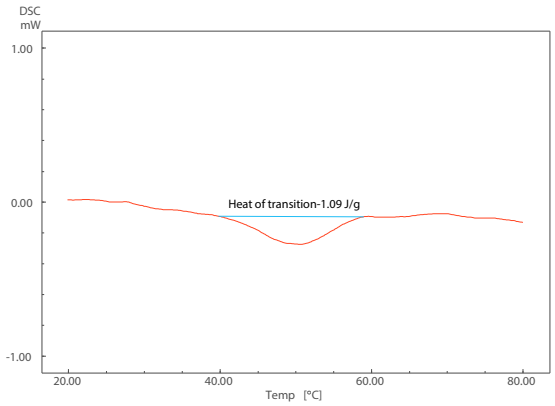

Fig. 5 DSC Measurement Results for Bread (After 1 Day)

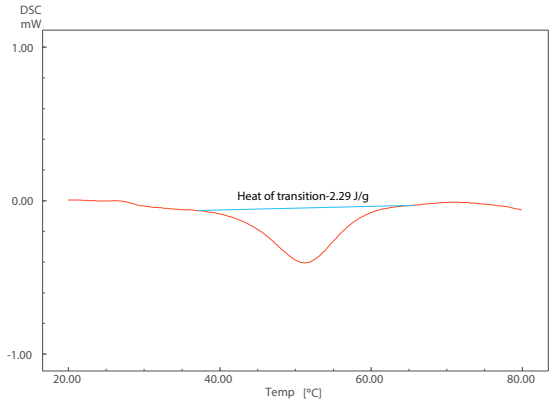

Fig. 6 DSC Measurement Results for Bread (After 5 Days)

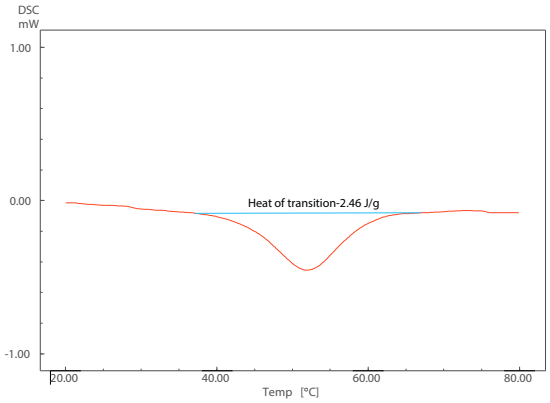

Fig. 7 DSC Measurement Results for Bread (After 9 Days)

Although no peak was observed in the data measured immediately after the bread was baked (Fig. 4), an endothermic peak due to the gelatinization appeared after 1 day (Fig. 5), and the peak became larger with time at 5 days (Fig. 6) and 9 days (Fig. 7). Since the size of this peak indicates the degree of progress of retrogradation, it can be understood that retrogradation progresses with time.

Fig. 8 shows the result of plotting the relationship between the elapsed time (number of days) immediately after baking and the size of the endothermic peak due to the gelatinization transition (heat of transition).

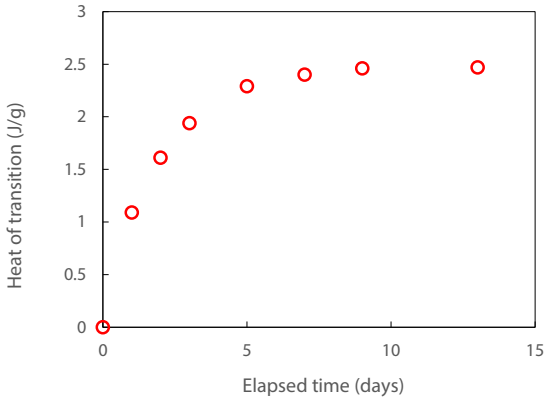

Fig. 8 Relationship of Elapsed Time and Size of Endothermic Peak (Heat of Transition) for Bread

Conclusion

Although the heat of transition increased with elapsed time immediately after baking, no change occurred in the heat of transition from the 9th day. This result shows that retrogradation progressed from immediately after baking but stopped around the 7th day. The speed of retrogradation varies depending on the type of starch and its sugar content, the presence or absence of oils and fats, and other factors. As shown here, it is possible to evaluate the time-dependent change in the hardness of foods that contain starch, such as various types of bread and rice products, by retrogradation measurements by DSC.

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