

Hydrothermal Changes of Starch Monitored by Combined NMR and DSC Methods

The thermal, dynamic, and structural properties of wheat starch-water systems with different levels of water content (11, 35, 40, 42, 45, and 50%, wet basis) were investigated. ¹H time domain nuclear magnetic resonance (TD-NMR) spectroscopy was used to interpret and quantify the water transfer and starch transformations in terms of water uptake, granule swelling, amylose leaching, and melting of starch polymers in relation to the different levels of water content. Complementary differential scanning calorimetry (DSC) experiments were performed to study the effects of water content on the degree of starch gelatinization. In particular, this twofold approach was applied to the first endotherm to study the mechanisms of gelatinization with a common heating range both in NMR and DSC. It was shown that the trend of the enthalpy changes in the first phase transition in starch-water (SW) mixtures was strongly correlated with the loss of solid content measured by NMR in the corresponding temperature range (55-70 °C). Based on the evolution of the relative amplitudes of T₂, structural transformations of starch were shown to occur in both crystalline and amorphous regions within SW samples, supporting the fact that the amorphous phase of starch also plays a significant role in the phase transition of granules during gelatinization. This dynamic and hydrothermal approach provided the first NMR-based interpretation of the first endotherm measured by DSC.

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