Effect of moisture uptake on amorphous inulin properties

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Introduction

Inulin is a natural storage carbohydrate composed of a chain of fructose units with generally a terminal glucose unit, industrially extracted from chicory root and commercially available in the powdered form. In a previous study, we engineered physical properties and controlled the amorphous/crystallinity content of inulin by selecting appropriate fixed temperature and/or inert air temperature of the spray-drier.

Unlike a crystalline structure, the amorphous state has a kinetically non-equilibrium structure. Amorphous solids are commonly formed through rapid cooling of a liquid melt to a certain temperature so that the molecules in the melt do not have enough time to rearrange and are frozen in their original position. An amorphous solid is also called a glass, and is characterized by a glass transition, which refers to the phase transition when a glass is changed into a supercooled melt. The glass transition is an important parameter for understanding the mechanisms of transformation processes in foods and for controlling their shelf-life. Depending on the moisture and/or the storage temperature, the amorphous product can physically change in order to attain a more thermo-dynamical stable state. For this reason, the aim of the present paper was to determine the kinetic of the physical changes of amorphous inulin powder stored at high relative humidity. The physical parameters investigated were the glass transition temperature (Tg) and the crystallinity index, determined by Modulated Differential Scanning Calorimetry (MDSC) and Wide Angle X-ray Scattering (WAXS), respectively. Temperature-resolved WAXS was used to understand the MDSC thermograms when crystallization occurred. In addition, surface analysis was used to correlate the measured parameters to the observed macroscopic property changes of the amorphous powder.

Wide Angle X-Ray Scattering (WAXS)

The powder X-ray diffractometer used was a Panalytical X’pert Philips Analytical X-ray B.V. with a Ni-filtered CuKα radiation, generated by an anode device operating at 40kV and 30mA in conjunction with a proportional detector. The patterns were recorded with a fixed time of 0.4s per step of 0.02° in the 2θ=20-60° range.

Experimentation and results

Inulin conditioning

The powder was stored over P2O5 for 2 weeks at 20°C to obtain a dehydrated product, then conditioned over KNO3 for different times.

Modulated Differential Scanning Calorimetry (MDSC)

The MDSC measurements were realized by using a DSC 2920 TA Instruments in hermetic and non hermetic aluminium pans. Heating rate was of 1.5°C min⁻¹ and the DSC cell was purged with 70 cm³ min⁻¹ dry nitrogen.

Conclusions

The effect of moisture uptake during storage on amorphous inulin properties has been investigated. Water content, crystallinity indexes, thermal properties and glass transition temperature evolution permitted the understanding of the physical and behaviour changes of the amorphous material. The Tg - water content state diagram allowed us to point out three zones. Zone I was the plasticization effect of water on Tg with inulin in a powdered amorphous state. The defined zone II was an intermediate state between glassy amorphous and crystallized inulin, with some macroscopic and thermal property changes. In zone III, the product crystallized, caked and no glass transition was observed. An endothermic peak appeared at the initial glass transition, which was attributed to the melting of inulin crystals, as confirmed by Temperature-Resolved Wide Angle X-ray Scattering.

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