On the understanding of the physical changes of inulin powder as a function of water activity

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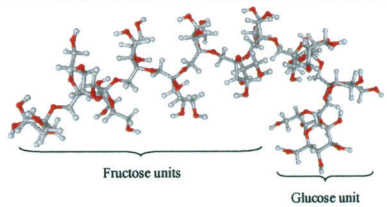
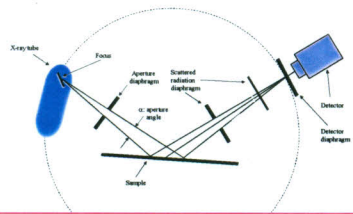
Introduction

Inulin is an oligosaccharide belonging to the family of fructans, industrially extracted from chicory root. This oligofructose is used as a nondigestible dietary fiber for its bifidogenic properties, but also for techno-functional properties in many foodstuff preparations, e.g. texturing, fat replacing… With its use in constant increase, a strong knowledge of inulin is necessary for technological and formulating purposes. For this reason, a fundamental study correlating the physical properties in a solid state and storage conditions has been done. Sorption isotherms have been established for predicting water sorption properties of the inulin powder. The aim of this work was to correlate physical properties of inulin to the water content in order to understand the change of behaviour of inulin during storage. For this purpose, crystallinity, glass transition and powder morphology have been determined by Wide Angle X ray Scattering (WAXS), Modulated Differential Scanning Calorimetry (MDSC) and Environmental Scanning Electron Microscopy (ESEM) respectively.

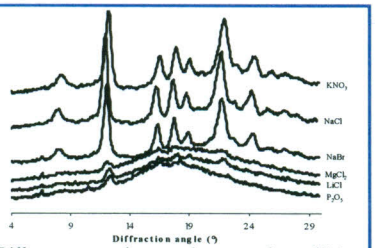


Experimentation and results

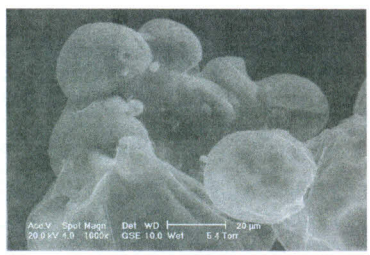
Powder X-ray diffraction measurement

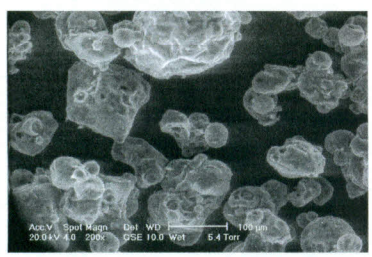


The WAXS apparatus was a PW3710 Philips Analytical X-ray B.V. with an anode device operating at 40kV and 30mA (λ = 1.54178 Å) in conjunction with a proportionnal detector in the 4<2θ<30° range.

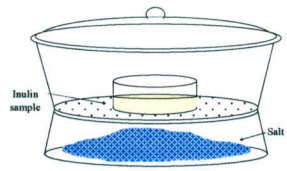


Diffractograms show an increase of crystallinity during storage at a_w>0.56

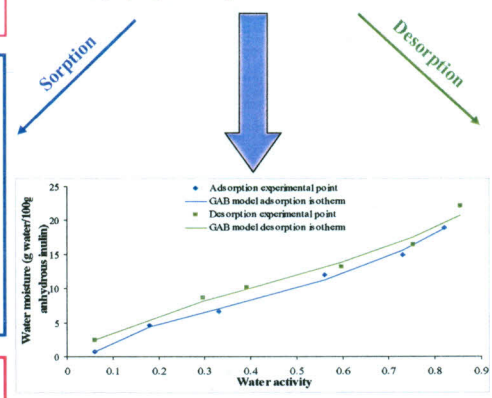




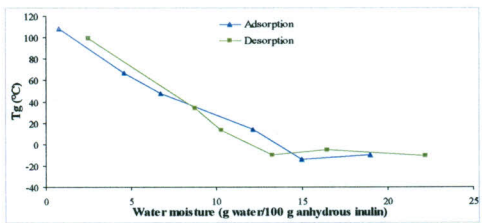
ESEM of inulin in a powdered form show amorphous spherical shapes with an average size of about 50-100µm.



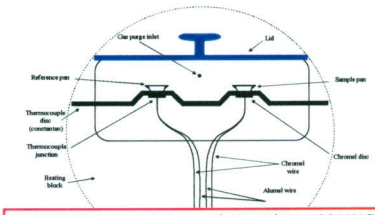
Inulin was initially stored over P_2O_5 or KNO_3. This step was done to obtain a dehydrated or humidified product. The samples were left to equilibrium for three weeks, then they were conditioned at controlled a_w. The salts used covered a large a_w range. This storage lasted at least five weeks at 20°C.



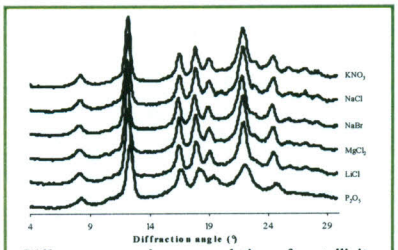
Moisture adsorption and desorption isotherm were fitted using GAB model over a large a_w range.



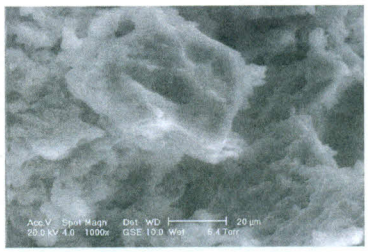
MDSC measurement

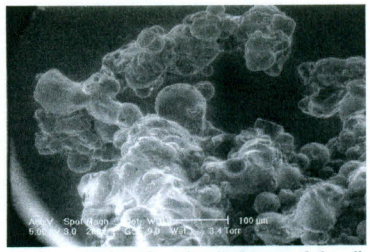


The MDSC measurements were realized by using a DSC 2920CE TA Instrument in hermetic aluminium pans. Heating rate was of 1.5°C.min^{-1} and the DSC cell was purged with 70 cm^3.min^{-1} dry nitrogen.



Diffractograms show no evolution of crystallinity during the storage at different relative humidities.





At a_w > 0.56 for adsorption isotherm and for all desorption isotherm, ESEM show a continuous mass of inulin with some crystal development between particles.

Conclusions

- ✓ Relative humidity storages of inulin influenced the evolution of several physical parameters.
  - → Confirmation of plasticizing effect of water content on inulin (decreasing glass transition temperature when water content increases).
  - → Change of crystallinity depending on the adsorption or the desorption isotherm.
- ✓ The increase of the water moisture (a_w > 0.56) decreased the Tg under the storage temperature (20°C), leading to a development of crystallinity between the amorphous particles, resulting in a caking of the powder.

Acknowledgments

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Reference: S. Ronkart, C. Blecker, C. Fougnies, J.-C. Van Herck, J. Wouters and M. Paquot. (2006). Determination of physical changes of inulin related to sorption isotherms: An X-ray diffraction, modulated differential scanning calorimetry and environmental scanning electron microscopy study. Carbohydrate Polymers, 63, 210-217.

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