Characterization of the physical state of spray-dried inulin

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Introduction

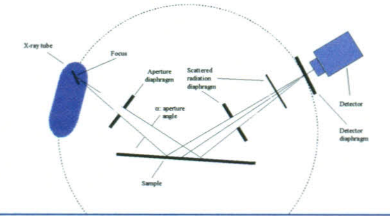
Inulin is a mixture of polysaccharides composed of a chain of fructose units with generally a terminal glucose unit. For now, commercial inulin is extracted from chicory root and is available as a spray-dried powder product, which has the advantage of low production costs compared to other drying techniques (e.g., freeze drying).

Spray-drying produces powder by atomizing a solution or slurry (named feed) and evaporating moisture from the resulting droplets by suspending them in hot gas. While moving in this hot medium, the droplets are dried into individual or agglomerate powder particles. Spray-drying trials remain very complicated and expensive under industrial conditions. For this reason, pilot scale spray-driers are interesting tools for investigating the influence of feed characteristics or drying parameters on the physical properties of the powders, which have a direct impact on the technofunctional properties (solubility or dispersibility in water, stability during storage…)

In this study, we investigated the importance of the feed temperature (40-95°C) and the inlet air temperature of the spray-drier (120-230°C) on the physical and morphological properties of spray-dried powder. The physical properties estimated were the glass transition temperature (Tg) and the crystallinity index, investigated by modulated differential scanning calorimetry (MDSC) and wide angle x-ray scattering (WAXS), respectively. In addition, environmental scanning electron microscopy (ESEM) was used for the surface and particle shape characterization of the powder.

Experimentation and results

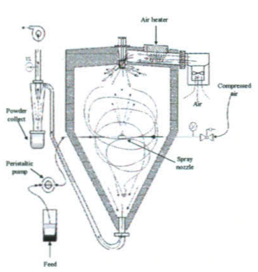
Wide Angle X-Ray Scattering (WAXS)



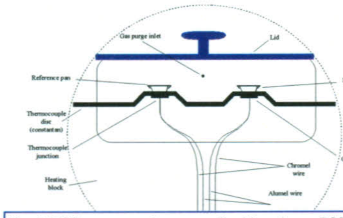
The WAXS aparatus was a PW3710 Philips Analytical X-ray B.V. with an anode device operating at 40kV and 30mA (λ = 1.54178 Å) in conjunction with a proportionnal detector in the 4<2θ<30° range.

Spray drying

Inulin (Fibruline XL obtained from Cosucra Groupe Warcoing SA) dispersions were spray dried into a mist by a two-fluid nozzle by means of compressed air. The nozzle was situated in the middle of the drying chamber spraying upwards, whereas the hot air was simultaneously introduced through an annular opening in the drying chamber ceiling. The liquid feed flow and the nozzle pressure air flow were 2 l h^{-1} and 2 bars, respectively. Two inlet air temperatures of 120 and 230°C were tested, which corresponded to an outlet air temperature of 65-67 and 120-125°C, respectively.

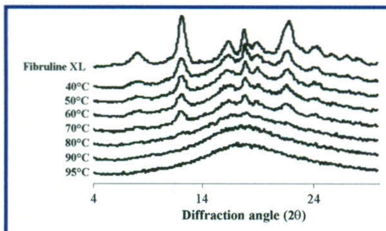


Modulated Differential Scanning Calorimetry (MDSC)



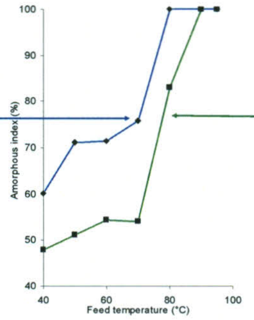
The MDSC measurements were realized by using a DSC 2920CE TA Instrument in non hermetic aluminium pans. Heating rate was of 1.5°C.min^{-1} and the DSC cell was purged with 70 cm^3.min^{-1} dry nitrogen.

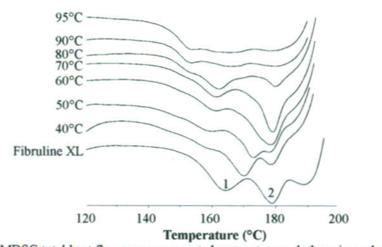
Inlet air temperature: 120°C



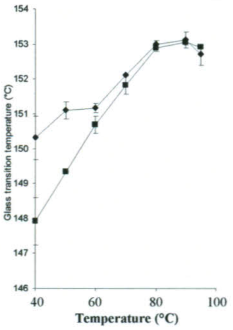
Diffractograms in the 4° < 2θ < 30° range present either a broad halo pattern, or diffraction peaks, characteristic of an amorphous or a semi-crystalline sample, respectively.

An increase in the feed temperature induced a higher solubilization of inulin and thus increased the amorphous index.

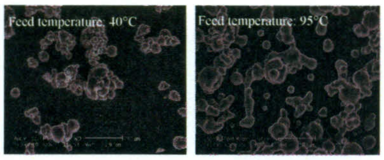




MDSC total heat flow curves presented one or two endothermic peaks labeled Tm1 (lower melting peak) and Tm2 (higher melting peak). Tm1 shifted to a higher temperature as the feed temperature increased, but Tm2 did not shift.

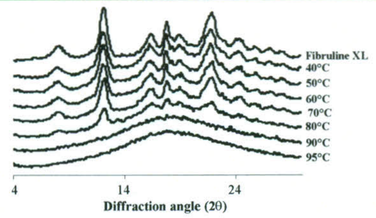


The feed temperature affected the solubility, but also the chemical composition of the soluble fraction. The average degree of polymerization of this fraction, reflecting the molecular weight of the amorphous part of the spray-dried product, increased with the feed temperature. This augmentation of the molecular weight led to a higher Tg. High inlet air temperature also induced an increase of the Tg, probably from some melted crystals.

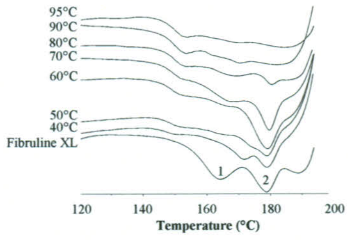


The main differences between particle morphologies were observed for the various feed temperatures, and were probably due to a difference in the melting of crystals present in the dispersion. Increasing the temperature of the feed led to a smoother powder surface, probably due to the spray drying of a solution rather than a dispersion. The drying of a dispersion containing undissolved solid particles (T feed < 80°C), led to powders with rough surfaces.

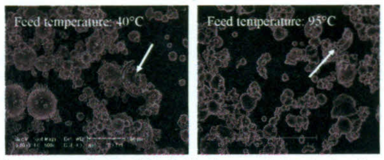
Inlet air temperature: 230°C



The amorphous content increased at an inlet air temperature of 230°C. Despite the residence time of dried inulin being relatively short in the spray-drier, we cannot totally exclude that a small proportion could have heated at a temperature above the melting point and thus have a higher amorphous content.



MDSC total heat flow curves presented one or two endothermic peaks labeled Tm1 (lower melting peak) and Tm2 (higher melting peak). Peak 1 decreased in intensity as the feed temperature increased as also observed in the increase of the amorphous index.



During the atomization of the feed in the spray drying chamber, the undissolved solid particles can fuse together, probably by the presence of solubilized inulin on their surface. The increase of the inlet air temperature induced a blow out of the particles.

Conclusions

The feed temperature and the inlet temperature were found to have significant effects on the physical and morphological properties of spray-dried inulin. An increase in the feed temperature or to a lesser extent, the inlet air temperature resulted in an increase of the amorphous content in the spray-dried products. By selecting appropriate feed or inlet air temperatures, we were able to produce spray-dried inulin with the desired physical properties. Such properties are of crucial importance to the hygroscopic properties and thus can affect the stability of the product in many domains like food technology or pharmaceuticals. As it is established that amorphous products are more hygroscopic than their crystalline counterparts, this could inevitably have an impact on their behavior during storage. For this reason, the impact of storage conditions on both stability and product quality of amorphous and semi-crystalline inulin will be evaluated and discussed in further studies.

Acknowledgments

Financial support was provided by the Walloon Region of Belgium (DGTRE) and Cosucra Groupe Warcoing SA. The authors are grateful to Mrs Lynn Doran for technical assistance.

Reference: S.N. Ronkart, C. Deroanne, M. Paquot, C. Fougnies, J.-C. Lambrechts, C.S. Blecker (2007). Characterization of the physical state of spray-dried inulin. *Food Biophysics*, 2, 83-92.