

Introduction

Ingredients, such as sugars, can be present in an amorphous or a crystalline state. The amorphous state is often caused process-related, e.g. during spray- and freeze drying, grinding, compaction, extrusion, evaporation or baking [1].

In general, amorphous products are hygroscopic. The associated high water-binding capacity favors uncontrolled crystallization if a critical moisture content is exceeded. In consequence, product changes such as caking, reduced solubility, increase of Maillard reactions and fat oxidation as well as a rougher mouthfeel affect the physical and chemical stability, the acceptance and the quality of the product [2,3].

In this context, milk-based powders are often critical, as a certain amount of amorphous lactose often develops during manufacturing. Due to the versatile use of lactose-, milk- and whey powder this is important in both the food- and pharmaceutical sectors.

To estimate the stability of such products and formulations the determination of the amorphous fraction and their crystallization kinetics is crucial.

Principle of the method

The DVS method is based on the higher moisture sorption affinity of amorphous compared to crystalline materials. In addition, water vapor sorption induces crystallization of the amorphous parts. Both kinetics of water vapor sorption and crystallization events can be followed by DVS experiments. For quantitative determination of the amorphous content by DVS, several methods have been described in literature [1,4-6].

The basic idea is to obtain a calibration curve by measuring the sorption behavior of powder mixtures with different ratios of amorphous to crystalline.

Results

Kinetics of moisture sorption of various amorphous/crystalline lactose mixtures are shown in Fig. 1. If a critical moisture of 40 % RH is exceeded, the amorphous lactose starts to crystallize. The lower water binding ability of the crystals causes the release of water and thus a decrease in mass. In comparison, crystalline lactose shows a mass increase only above 90 % RH which can be explained by an incipient dissolution (Fig. 1).

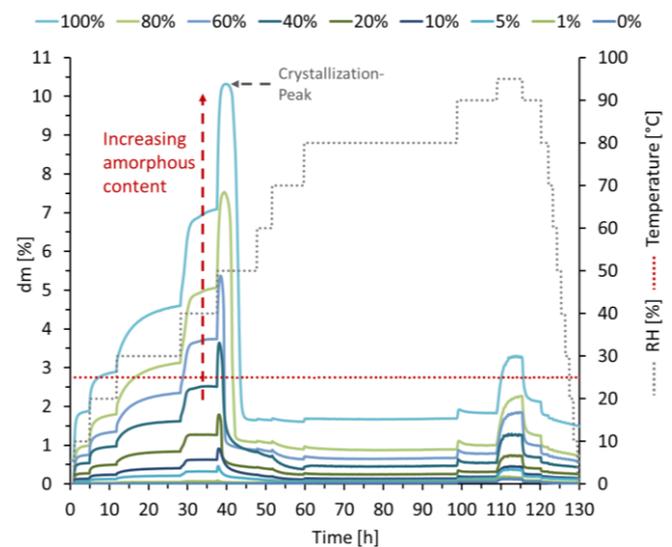


Fig. 1: Sorption kinetics of amorphous/crystalline lactose mixtures.

From curves shown in Fig. 1 a calibration curve was obtained by plotting the monomolecular moisture content X_m over the amorphous content of the mixture as proposed by Vollenbroek *et al.* [4]. A linear relationship over the entire range (0-100 %) with a correlation coefficient of $R^2 = 0.9983$ was obtained (Fig. 2).

Conclusion

Amorphous contents can be determined by dynamic water vapor sorption analysis. The use of the automated multisampling SPS device allows both a simple and highly sensitive method for a precise determination of amorphous fractions in powdered materials.

With the help of the obtained calibration, the amorphous portion in unknown lactose powders can be determined on basis of their water vapor sorption behavior.

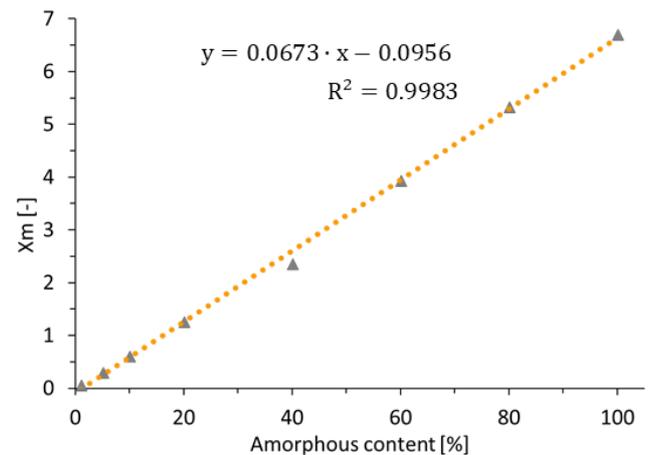


Fig. 2: Calculated moisture content of the monolayer (X_m) dependent on the amorphous lactose content in the mixture.

References

- [1] A. Saleki-Gerhardt, C. Ahlneck, G. Zograf, Assessment of disorder in crystalline solids, *Int. J. Pharm.* (1994). doi:10.1016/0378-5173(94)90219-4.
- [2] W. Danzl, G. Ziegler, Untersuchung der Kristallisation Amorpher Lactose in Milchpulver Anhand Dynamischer Wasserdampfsorption, *Chemie-Ingenieur-Technik.* (2008). doi:10.1002/cite.200700098.
- [3] W. Danzl, Milchpulver für Schokolade Quantifizierung und Auswirkung amorpher Lactose, *GIT Labor-Fachzeitschrift* 59. (2015) 20–22.
- [4] J. Vollenbroek, G.A. Hebbink, S. Ziffels, H. Steckel, Determination of low levels of amorphous content in inhalation grade lactose by moisture sorption isotherms, *Int. J. Pharm.* (2010). doi:10.1016/j.ijpharm.2010.04.035.
- [5] G. Buckton, P. Darcy, The use of gravimetric studies to assess the degree of crystallinity of predominantly crystalline powders, *Int. J. Pharm.* (1995). doi:10.1016/0378-5173(95)00083-U.
- [6] L. Mackin, R. Zanon, J.M. Park, K. Foster, H. Opalenik, M. Demonte, Quantification of low levels (<10%) of amorphous content in micronised active batches using dynamic vapour sorption and isothermal microcalorimetry, *Int. J. Pharm.* (2002).

For more detailed information: proUmid White Paper 20-01