Dynamic vapor sorption analysis to determine amorphous sucrose fractions

– Application Note 20-02

Introduction

During the production of sucrose or its further processing in food amorphous fractions can be formed.

For example, high forces during fine grinding in the production of icing sugar or the rapid solidification during spray- or freeze drying of sucrose containing foods favors an increased amorphous content [1].

The hygroscopicity of amorphous components favors the adsorption of moisture from the environment and the transition to a thermodynamic stable crystal state. Due to the lower water binding capacity of the crystals the adsorbed moisture is released which may cause undesired product changes, such as caking and reduced flowability, a lower microbial- and chemical stability or modification of the product's appearance as color changes and loss of transparency (Fig. 1) [2-4].

To estimate the stability of such products and formulations, the knowledge about the amorphous content and the recrystallization kinetics is crucial.

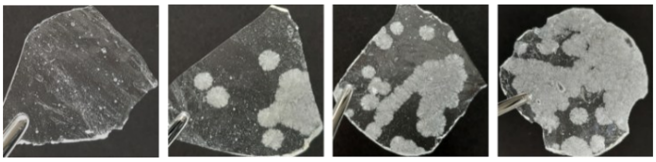

Fig. 1: Progressing crystallization of amorphous sucrose

Principle of the method

The idea behind the DVS method for determining amorphous contents is to measure the sorption kinetics of powder mixtures with a known amorphous concentration. As amorphous materials show a higher affinity for moisture adsorption than crystalline, the sorption behavior can be correlated with the amorphous content. In follow, the amorphous content of unknown sample materials can be calculated with the help of the obtained calibration curve.

Additionally, the multisampling SPS sorption test systems can be used to characterize material specific crystallization conditions and kinetics dependent on temperature and relative humidity.

Fig. 2 shows the sorption kinetics of several amorphous/crystalline sucrose mixtures and the onset of crystallization at a relative humidity of ~ 35 %.

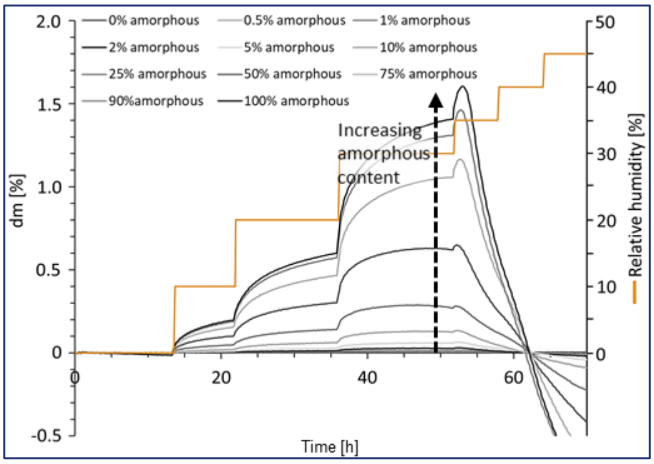

Fig. 2: Sorption kinetics of amorphous/crystalline sucrose mixtures. Mass loss at a RH ~ 35 % is attributed to the onset of crystallization.

Conclusion

Based on these sorption measurements (Fig. 2), a calibration curve with a correlation factor of $R^2 = 0.9997$ was obtained. This calibration can be used for the determination of amorphous fractions in unknown sucrose samples, e.g. for quality control or in R&D applications.

The use of the automated multisampling SPS device provides both a simple and a sensitive method for the quantitative determination of amorphous fractions as well as the kinetics and conditions of crystallization-induced phase transitions.

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