Hydrate formation of L-lysine HCL analyzed by dynamic water vapor sorption analysis

– Application Note 20-06

Scope

The Application Note shows the applicability of DVS-analysis for the investigation of hydrate formation using the example of L-lysine HCl. Phase transition from anhydrate to dihydrate was determined using the multisampling moisture sorption analyzer SPSx-1µ.

Introduction

Hydrates, in general, are substances which have formed bonds with water molecules. Water binding can take place via electrostatic dipole-dipole interactions, via hydrogen bonds or coordinatively in complexes. In inorganic, crystalline hydrates water molecules are bound in a specific stoichiometric ratio.

Hydrates are usually built during crystallization from aqueous solutions. By drying and removing the crystallization water, the anhydrous crystal can be formed [1].

The respective hydrate state influences the properties of the material, e.g. the dissolution rate, the compressibility or the bioavailability of a product. Furthermore, the high hygroscopicity of the anhydrate influences the storability and handling of the product [2-5].

Therefore, identification and control of hydrate forms is important in the field of quality control, product development and material characterization.

Dynamic water vapor sorption (DVS) analysis is an effective method not only for determining the present hydrate species but also for determining the kinetics of hydrate formation as a function of temperature and relative humidity.

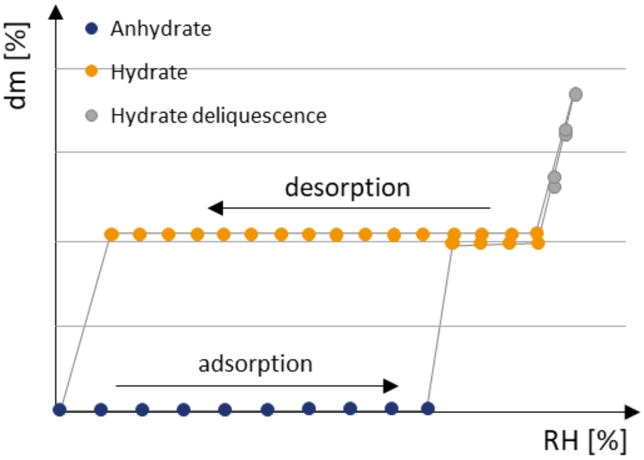

Fig. 1: Schematic drawing: Adsorption-desorption cycle with transition from anhydrate to hydrate crystals.

DVS method for hydrate identification

Different hydrate species and corresponding anhydrates vary in their stability depending on temperature and relative humidity [6]. In general, the anhydrous form is more hygroscopic. With exceeding a certain humidity level, a steep moisture increase is usually observed as shown by the schematic draw in Fig. 1. The following plateau as well as the pronounced hysteresis loop indicate the formation of a hydrate [7].

Influence of measurement settings

DVS measurements are well suited to investigate the conditions for hydrate formation and stability. However, both initiation and kinetics of hydrate formation can vary strongly depending on the substance. Therefore, the measurement settings are a decisive factor for reliable hydrate identification by means of DVS analysis.

In particular, time for moisture equilibration and resolution of humidity steps are crucial, which is demonstrated by the example of L-lysine HCL in Fig. 2.

Results show that hydrate formation could not be assessed by the settings used in Fig. 2A due to an insufficient resolution of the humidity steps. Results could be improved by a higher RH resolution in (B) which at least indicates the formation of a hydrate. An additional extension of the maximum equilibrium time in (C) finally allows the precise characterization of the RH-dependent formation and stability range of the L-lysine HCL dihydrate.

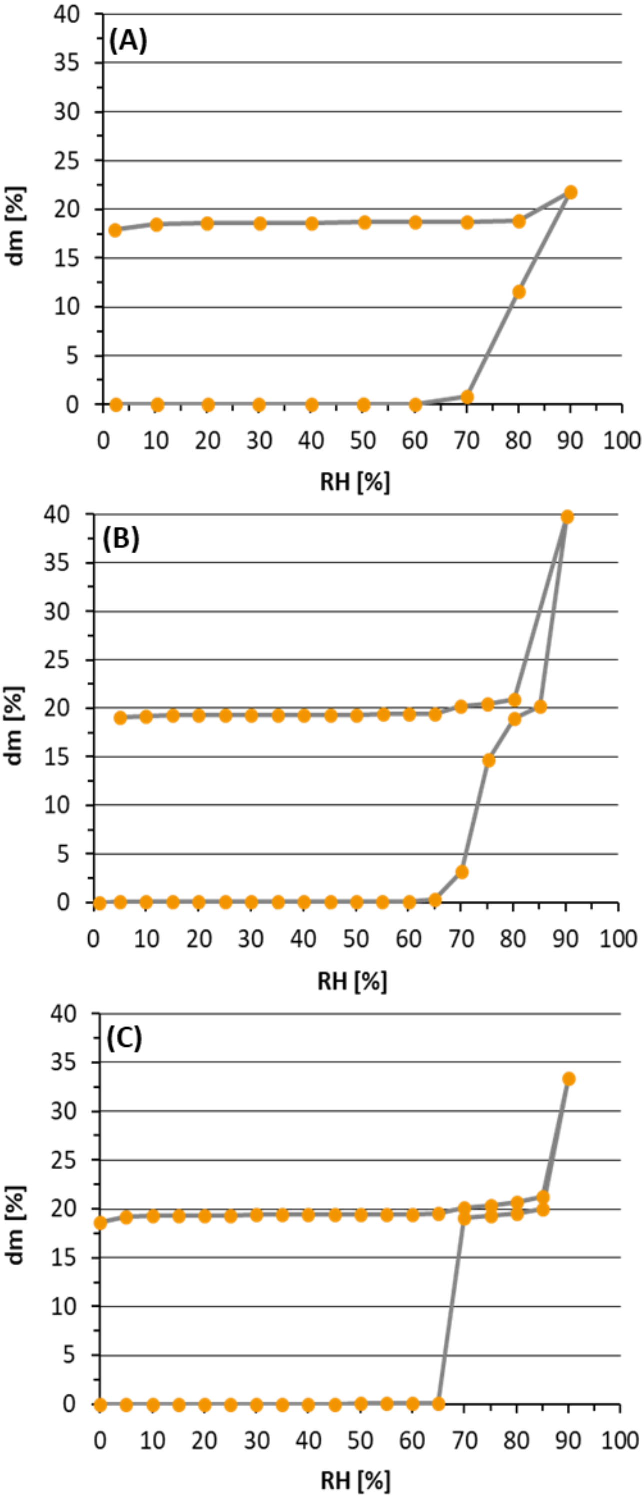

Fig. 2: Influence of the measurement settings on anhydrate-hydrate transition and identification L-lysine HCl dihydrate

Conclusion

DVS analysis was used to investigate the formation and stability of L-lysine HCl dihydrate. Measurement settings such as maximum equilibrium time and resolution of humidity steps, proved to be essential criteria for a precise determination of the boundary conditions for the anhydrate-hydrate transition.

Based on these results it was shown that DVS analyses are well suited to characterize hydrate formation and stability as a function of relative humidity.

References

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