

Revealing Sorption/Desorption Properties of Micro-Crystalline Cellulose by Means of STA

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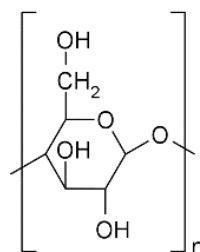
1 STA 449 F3 Nevio and modular humidity generator

Introduction

Moisture may affect the properties of a wide range of active ingredients and excipients in terms of their stability, crystallinity, bio-availability, etc. One method for determining the influence of humidity on a substance's behavior is dynamic vapor sorption (DVS), in which the mass changes in the sample are measured for different solvent vapor amounts, e.g., water vapor. [1]

Such measurements can be carried out with an STA (simultaneous thermal analyzer) connected to a modular humidity generator (figure 1). In the following, a dynamic water sorption measurement was carried out on micro-crystalline cellulose (MCC, chemical structure in

figure 2). This substance is used in tablet formulations as filler and binder. [2]



2 Chemical structure of micro-crystalline cellulose

Tab 1. Conditions of the test

Device	STA 449 F3 Nevió connected with the humidity generator
Sample	Micro-crystalline cellulose
Sample mass	41.22 mg
Sample holder	Plate made of alumina, Ø 17 mm
Temperature program	Isothermal 44°C, nitrogen atmosphere, relative humidity (RH) increased from 0 to 80%



TGA sample holder with alumina plate

Measurement Conditions

The experimental conditions are summarized in table 1.

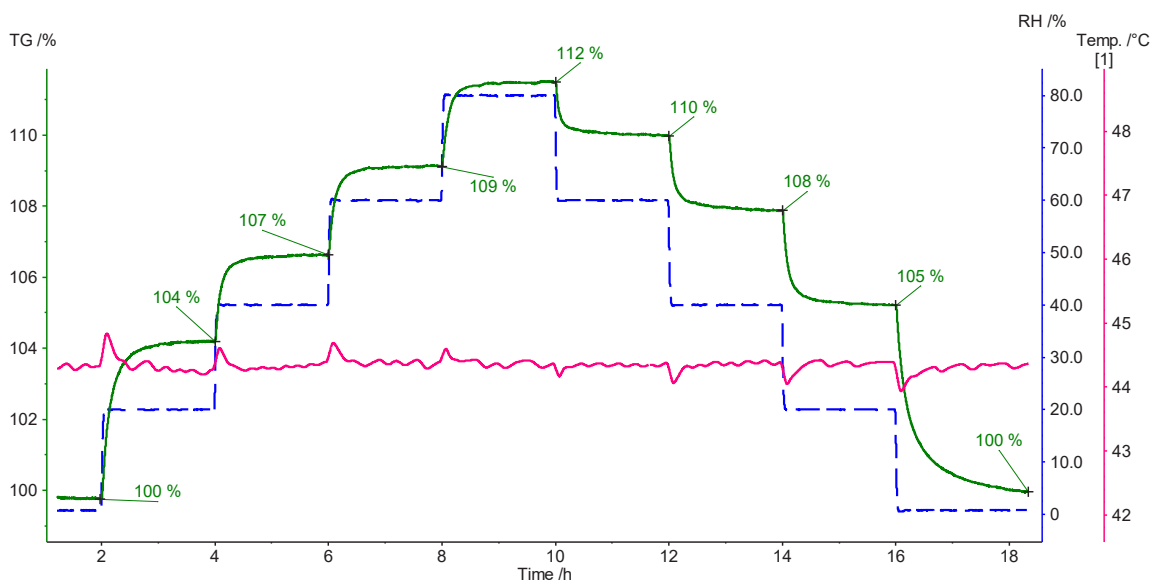
Measurement Results

Figure 3 displays the measured sample mass and temperature during the experiment.

The results demonstrate the strong hygroscopic nature of the micro-crystalline cellulose. The first increase in relative humidity from 0% to 20% (blue dashed curve) induces a mass increase of 4% (green curve). The subsequent steps

show that the higher the relative humidity, the higher the mass gain. As soon as the humidity level decreases, the absorbed and/or adsorbed water is released, resulting in a mass loss. When a completely dry atmosphere is finally reached at the end of the measurement, the amount of water absorbed and/or adsorbed will have been quantitatively released. This can be confirmed by having arrived back at the initial sample mass (100%).

Each change in relative humidity level is associated with a peak in the sample temperature curve (pink curve). This is due to the exothermal and endothermal natures of the sorption and desorption of water, respectively.



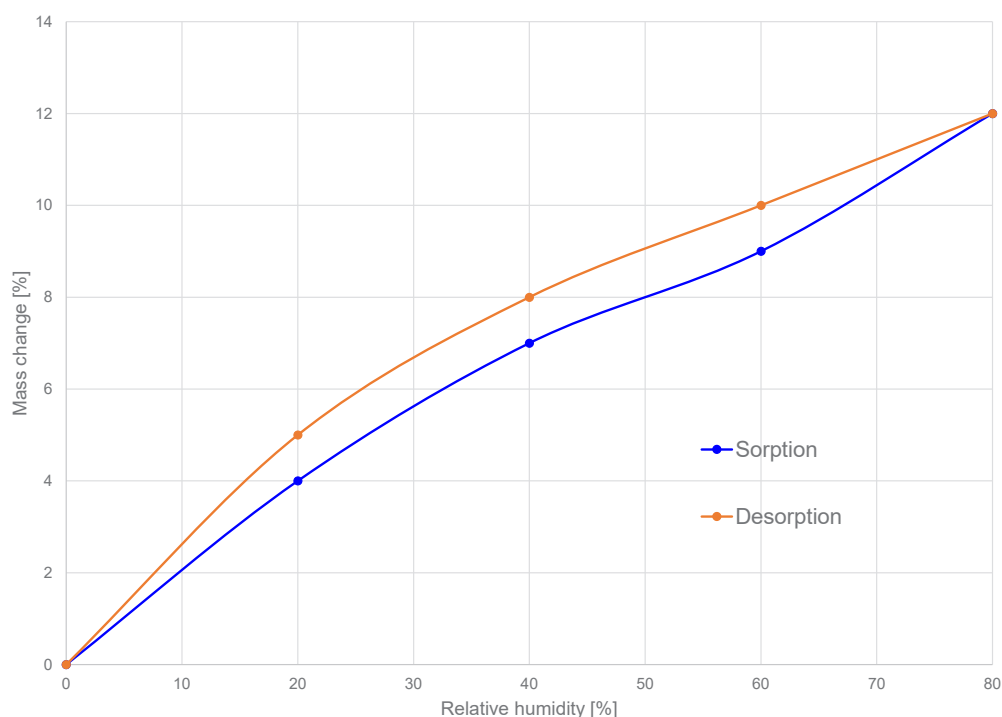
3 Mass and temperature results for micro-crystalline cellulose in an atmosphere with different humidity levels (0%, 20%, 40%, 60% and 80%)

APPLICATIONNOTE Revealing Sorption/Desorption Properties of Micro-Crystalline Cellulose by Means of STA

The mass gain and loss after reaching equilibrium are given in figure 4 for all measured relative humidity levels between 0% and 80%. The maximum mass increase amounts to 12% for a relative humidity of 80%. Micro-crystalline cellulose exhibits sorption hysteresis, i.e., the water amount in the sample is higher during desorption than during sorption (see figure 4) but, ultimately, the

starting point and ending point of the sorption/desorption cycle are identical.

This hysteresis phenomenon is typical for many porous materials. Chen et al [3] showed that the water-cellulose bonds formed during swelling of cellulose do not break upon desorption at the same chemical potential.



4 Difference in the moisture content of the sample between sorption and desorption

Conclusion

STA connected to a humidity generator allows for measurements of dynamic water sorption and desorption. The measurements on micro-crystalline cellulose highlight the hysteresis of the process: The moisture content is higher during desorption than during sorption. This phenomenon is typical for many porous material.

Literature

- [1] https://en.wikipedia.org/wiki/Dynamic_vapor_sorption
- [2] <https://www.jrspharma.com/>
- [3] Role of hydrogen bonding in hysteresis observed in sorption-induced swelling of soft nanoporous polymers, Mingyang Chen, Benoit Coasne, Robert Guyer, Dominique Derome, Jan Carmeliet, Nat Commun 9, 3507 (2018)