Crystallization behavior of freeze-dried lactose and lactitol as detected from the loss of sorbed water

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Introduction

- Amorphous sugars and sugar alcohols may crystallize when they are stored under relative humidity (RH) and temperature conditions exceeding their critical values.
  - **Lactose**: critical RH at 25 °C **37%**
  - **Lactitol** (sugar alcohol derived from lactose): critical RH at 25 °C **12%**
- Exceeding critical RH results in increased water content of material which decreases glass transition temperature (T_g) of material to below storage temperature, thus, enabling crystallization due to increased molecular mobility.

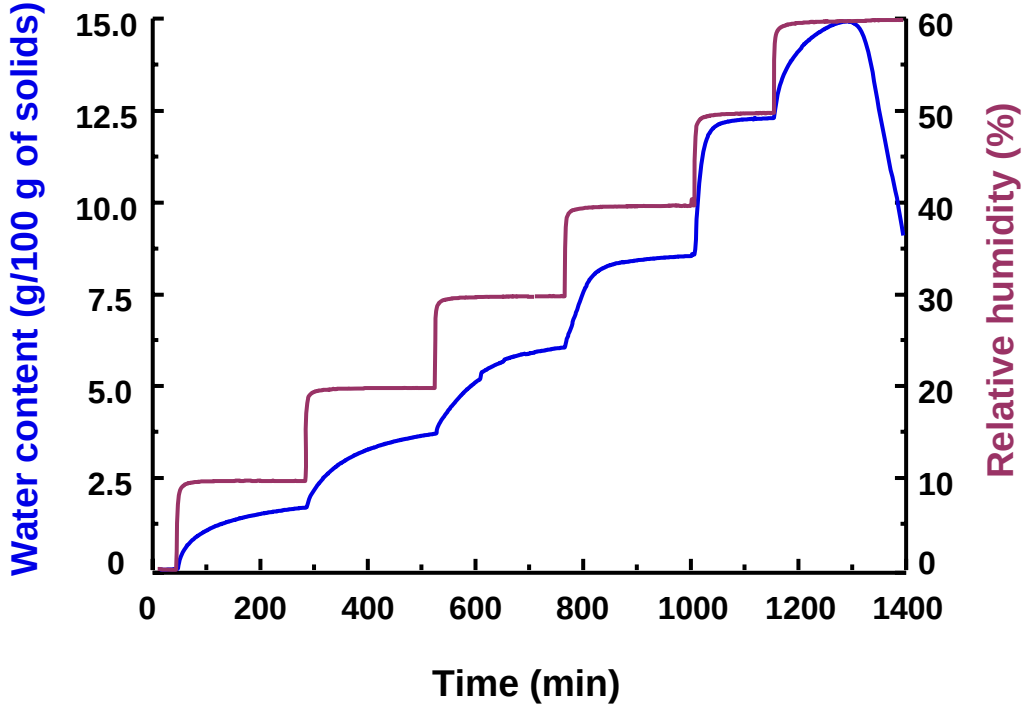
Aim of the study

- The aim of the present study was to compare water sorption and crystallization behavior of freeze-dried lactose and lactitol.
  - Water sorption behavior was detected from weight gain using either a static or dynamic analysis in which material was subjected to various RH conditions at constant temperature.
  - Crystallization behavior was detected from the loss of sorbed water using either a static or dynamic analysis.

Materials and methods

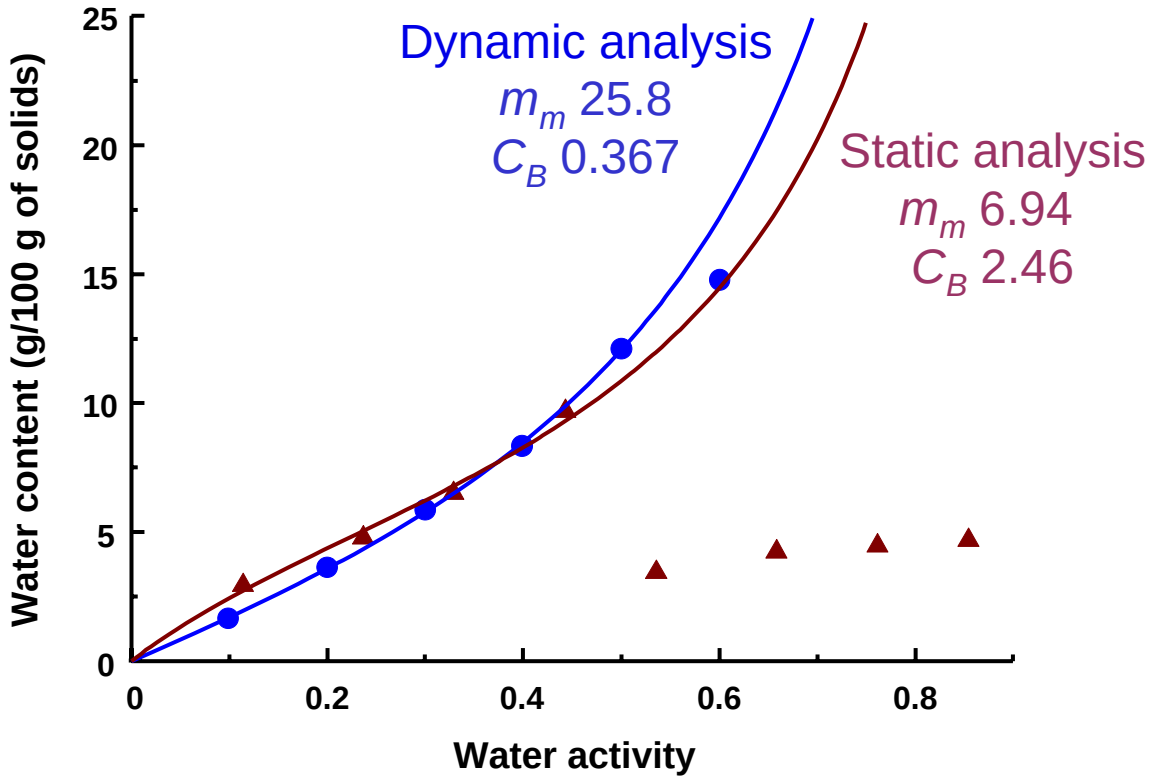
- Water solutions containing 10% (w/w) lactose or lactitol were freeze-dried and further dehydrated in vacuum desiccator over P_2O_5.
- **Static analysis**: weighing samples at intervals during storage in vacuum desiccators over saturated salt solutions giving relative vapor pressures (RVP) ranging from 11 to 85% at 25 °C.
- **Dynamic analysis**: using Dynamic Vapor Sorption (DVS) Intrinsic instrument at RH ranging from 10 to 85% at 25 °C.
- Steady-state water contents were used in calculation of the BET water sorption isotherms.

Example of DVS data

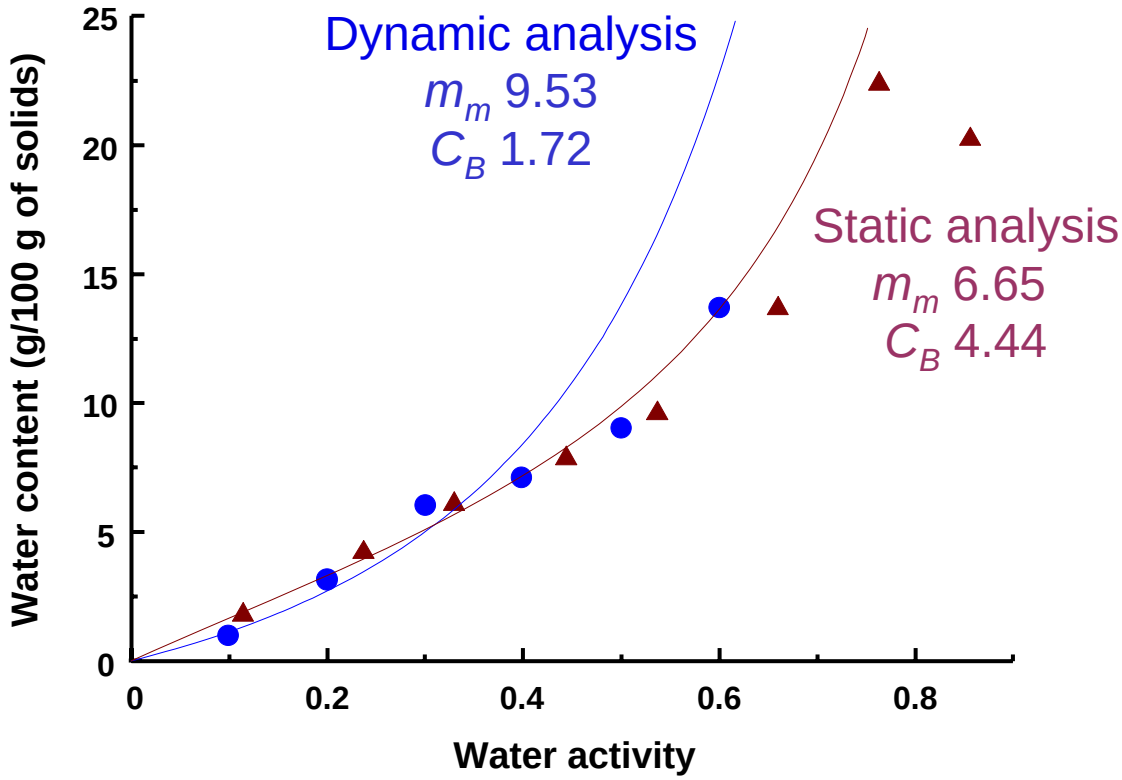


- RH ranged from 10 to 60% at 25 °C.
- Criterion used to obtain steady-state water contents was 0.002% min^{-1}. However, maximum time per step was adjusted to 4 h.

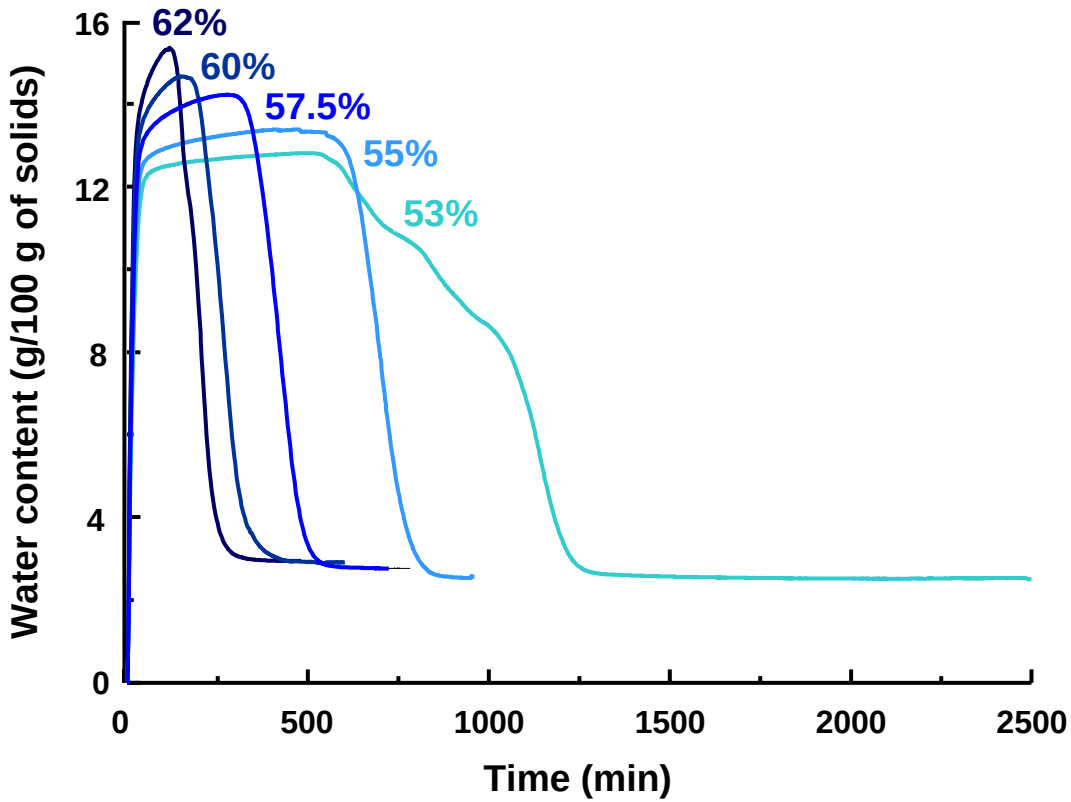
BET water sorption isotherms for freeze-dried lactose



BET water sorption isotherms for freeze-dried lactitol

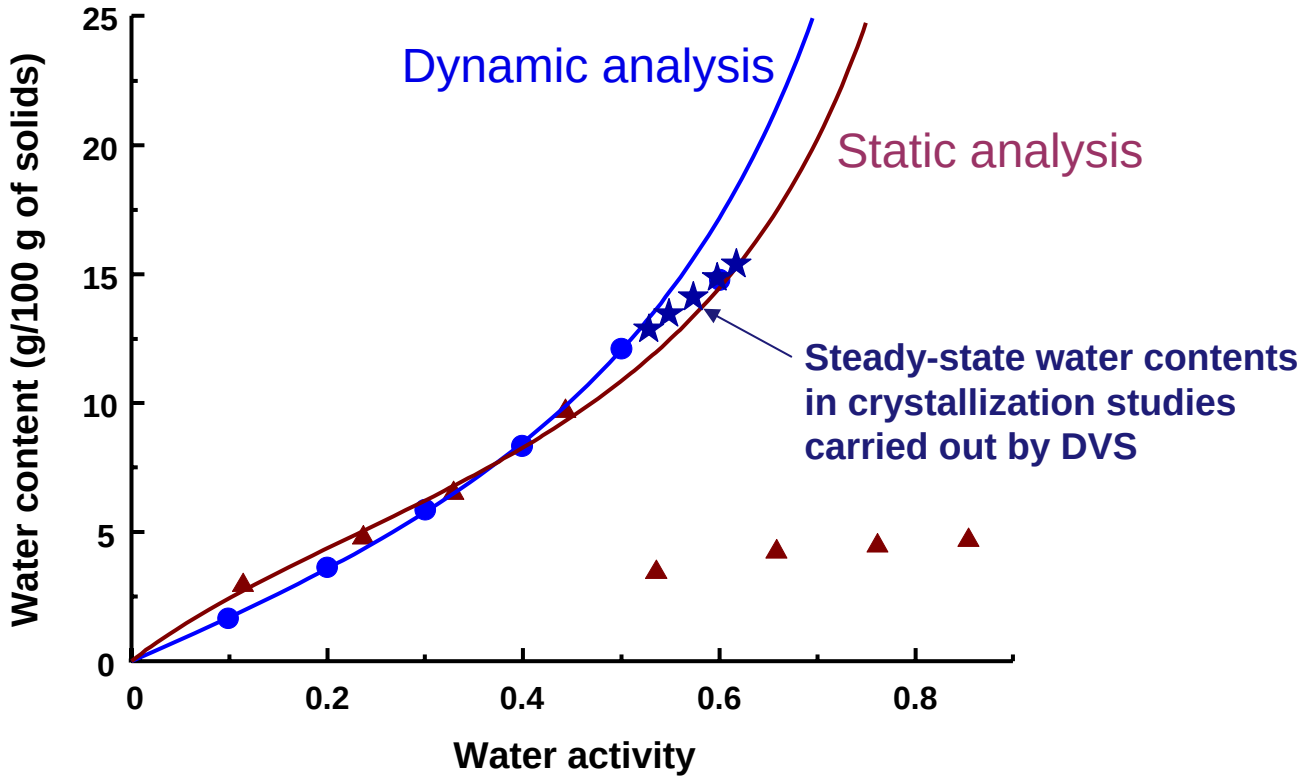


Crystallization of freeze-dried lactose in dynamic analyses

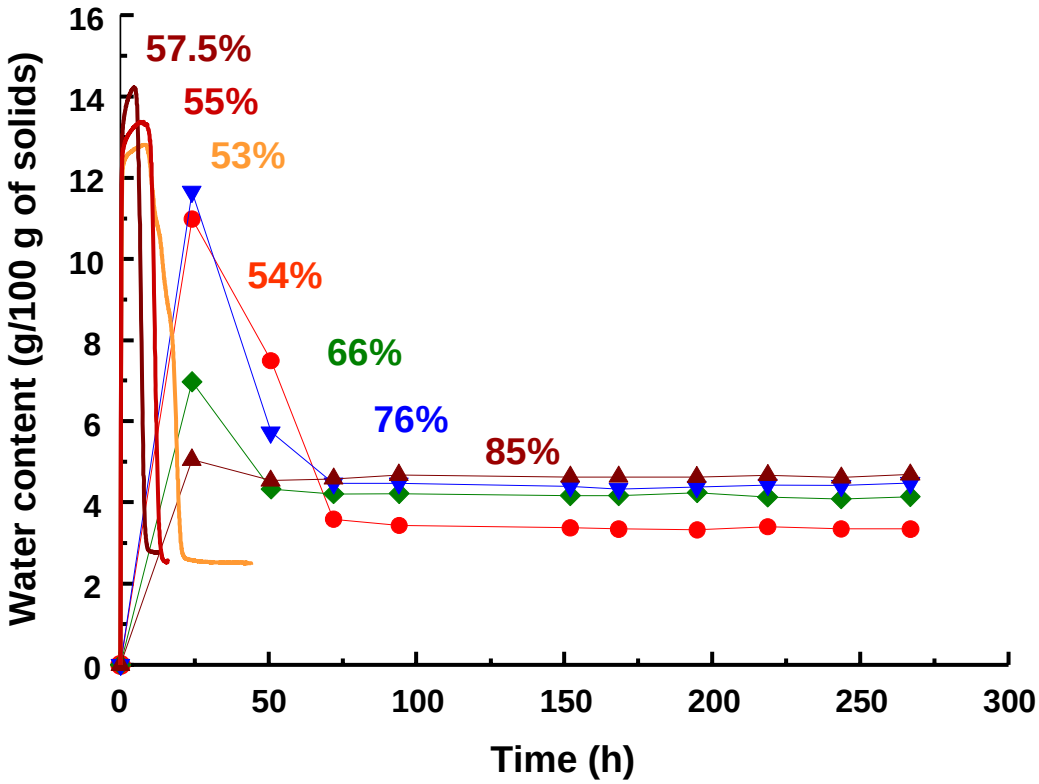


- The rate of crystallization increased with increasing RH.
- Steady-state water content was not achieved at RH of 62% => simultaneous water adsorption and water desorption due to crystallization?

Water sorption isotherms for freeze-dried lactose

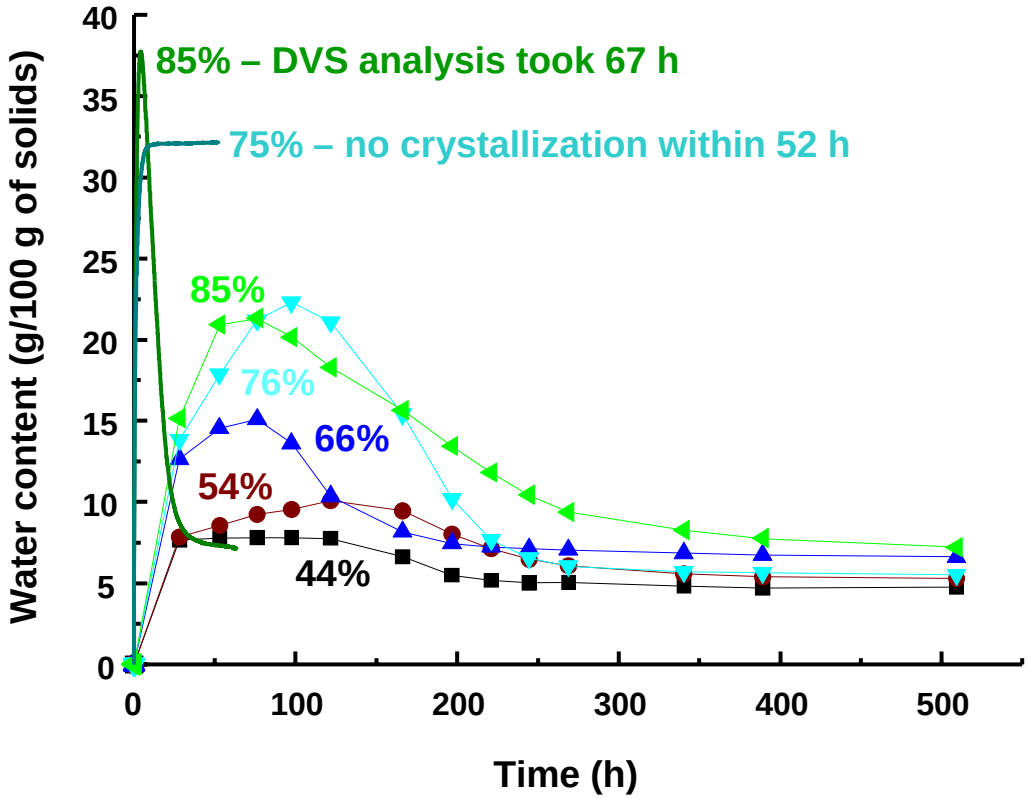


Crystallization of freeze-dried lactose



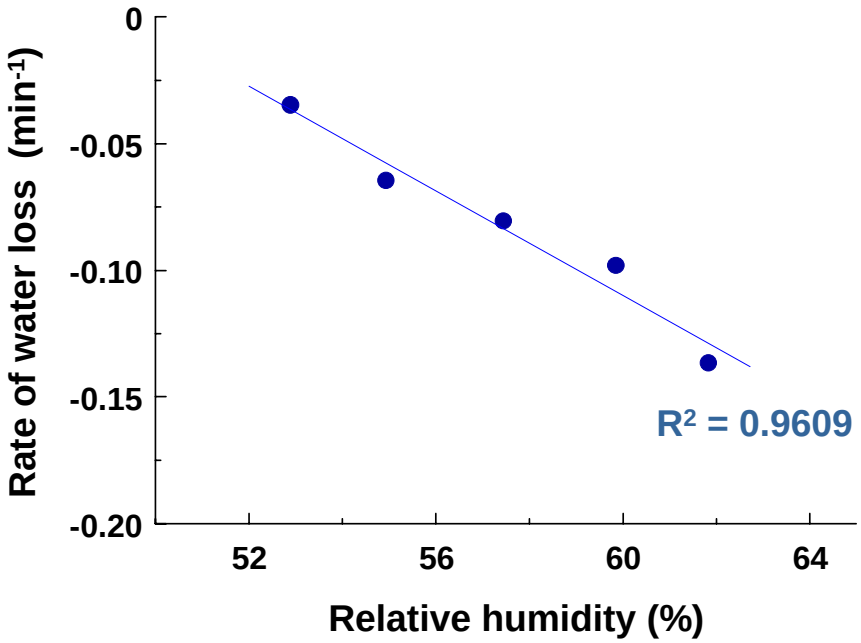
- Slower crystallization was observed in static analyses with bigger sample size (about 0.6 g) than in dynamic analyses with smaller sample size (about 10 mg).
- Simultaneous water adsorption and water desorption occurred in static analyses.

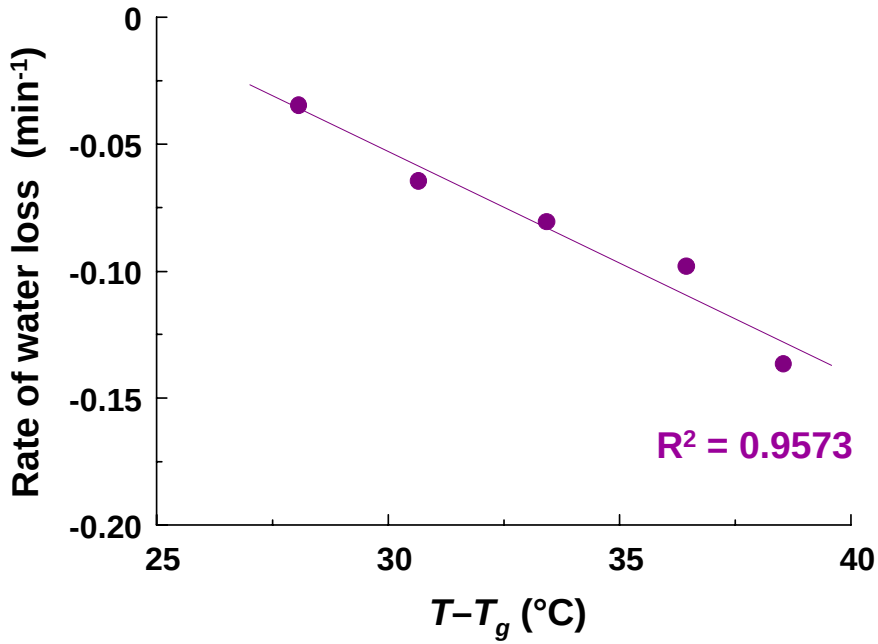
Crystallization of freeze-dried lactitol



- Simultaneous water ad- and desorption at high RVP.
- Lactitol with lower critical RH crystallized even at RH of 44% within 4 days. However, lactitol crystallizes slowly => long DVS analyses are needed.

Linear relationship between rate of water loss and RH or $T–T_g$





Rate of water loss was detected as a slope of a curve showing the biggest loss of water in time unit during DVS analysis.

Conclusions

- Both static and dynamic analysis suggested quite similar BET water sorption isotherm for freeze-dried lactose. Also, water contents of freeze-dried lactitol were quite close to each other.
- Dynamic analysis produced continuous data of crystallization. In static analysis some changes may be ignored due to weighing schedule used. Also, moving of samples may cause shocks to samples resulting in increased crystallization.
- Selecting proper conditions for crystallization is important in order to achieve steady-state water content before crystallization within a reasonable time. This is easier to do with DVS.

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