Desorption

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Overview

- Theory of Desorption
- Practical aspects of Desorption
- PAT

Theory of Desorption

Theory of Desorption

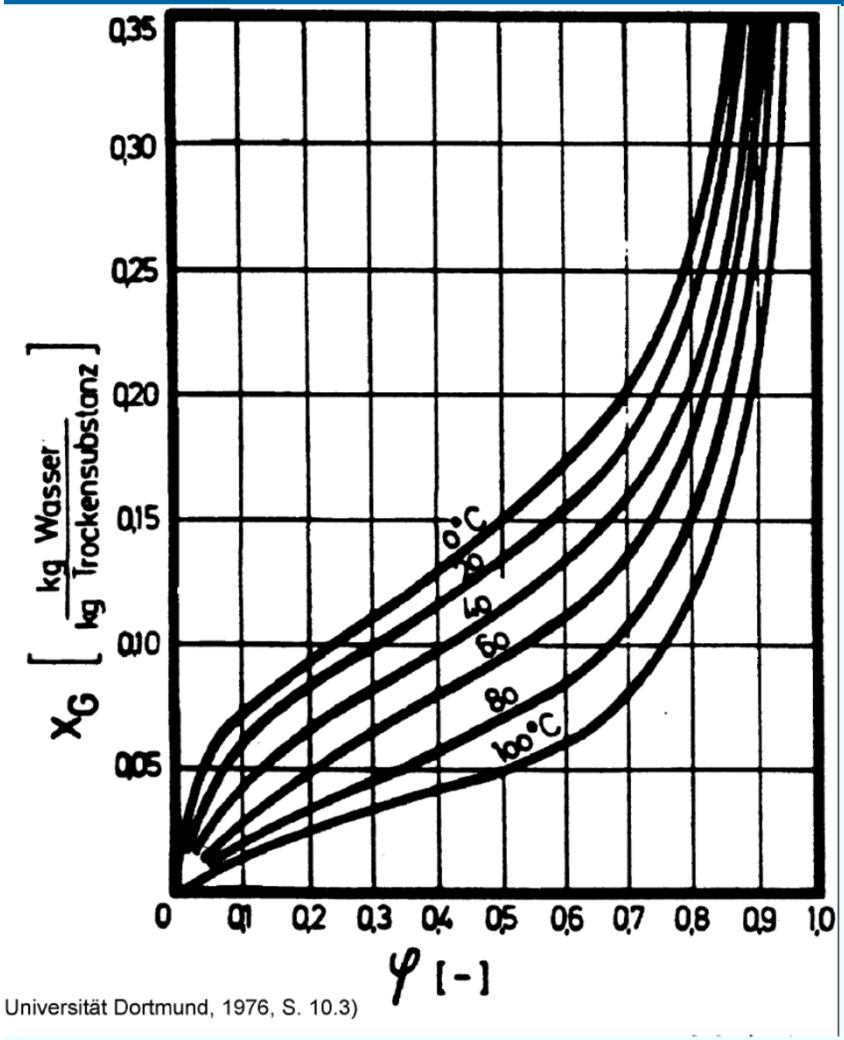
Residual moisture

• When leaded into process, the water content of the product was defined by water mass percentage to full mass

• The residual moisture is calculated by the water mass to dry mass => moisture levels above 100% are possible

• For long-term-storage the moisture content is of interest => moisture levels of 2...5% are common, sometimes a minimum moisture level is also required e.g. viruses or proteins

Theory of Desorption



Universität Dortmund, 1976, S. 10.3)

Sorption Isotherm of dried potatoe pieces

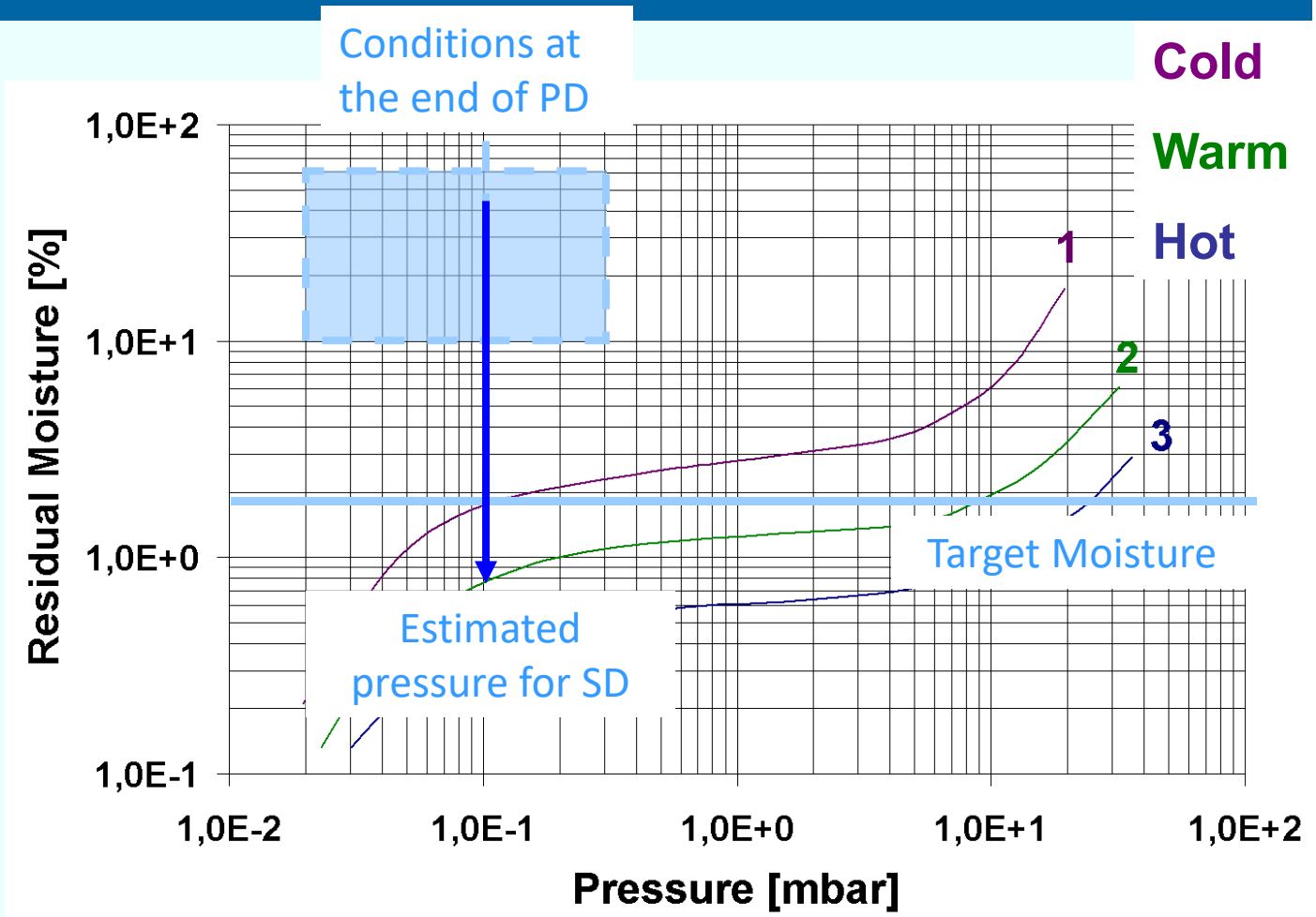
Sorption Isotherm

- Residual moisture level depends on Temperature and surrounding (partial) vapor pressure
- Moisture equilibrium is shown in sorption isotherm
- Conditions above/below isotherm lead to desorption/adsorption
- Water molecules are directly bound to dry product

Theory of Desorption

Sorption Isotherm

- Residual moisture level depends on Temperature and surrounding (partial) vapor pressure
- Convergion against the sorption isotherm



Adsorption / Desorption

• Residual moisture (detected by Karl Fischer-Method) consists of desorbable and non-desorbable water

• The integration of the desorption rate versus infinity allows – at least – theoretically the prediction of the residual moisture

• Overdried products would adsorb water at same conditions (“remoistering”) e.g. freeze dried coffee

Theory of Desorption

Arrhenius is everywhere

W_B	molar Bonding Energy (fractions…several eV)
K	Sorption Constant
R	general Gas Constant
T	absolute Temperature

- K < 0 => Desorption
- K > 0 => Adsorption

$$\text{Sorption Rate} = K \cdot e^{\frac{-W_B}{R \cdot T}}$$

Theory of Desorption

The Oetjen Idea

$$dW_{(t)} = \int_{t}^{\infty} \left(K \cdot e^{\frac{-W_B}{R \cdot T}} dt \right)$$

- Prediction of Residual Moisture is possible when Equilibrium moisture of pressure and temperature is known

Theory of Desorption

Desorption

- The Desorption Rate is linear depending on the Residual Moisture

- An empiric model (RM vs. DR) can be defined during cycle development for more effective cycle control with reduced development effort

$$DR \sim \dot{RM}_{KF(t)}$$

Practical Aspects of Desorption

Desorption Phase

- Process Temperature profile equilibrates during desorption phase
- Shelf Temperature at Desorption should be near the max. storage temperature, not above
- Process vacuum is of minor interest, higher pressure level allows good moisture equilization, but inhibits desorption

Optimization of Desorption Phase

- A general hint to estimate duration can not be given
- The use of PAT Tools allows the safe control of the right step time
- Frequently accomplished Desorption Rate Measurements enable safe detection of end point (historical Freeze Dryers can use the right calculated pressure rise)

$$DR = \frac{\Delta p / [\mu bar]}{\Delta t / [s]} \cdot \frac{V_c / [m^3]}{\vartheta_S / [K]} \cdot \frac{1}{m_{Tr} / [kg]} \cdot 0{,}7803 = \left[\frac{1}{h}\right]$$

Further Optimization of desorption phase

- As shown before the desorption rate is the first derivation of the desorbable Water
- Desorption time depends on concentration and crystal structure during freezing, impact of layer height is marginal
- Remark that the pharmaceutical standard for moisture measurement detects all Water content, including non-desorbable Water

PAT

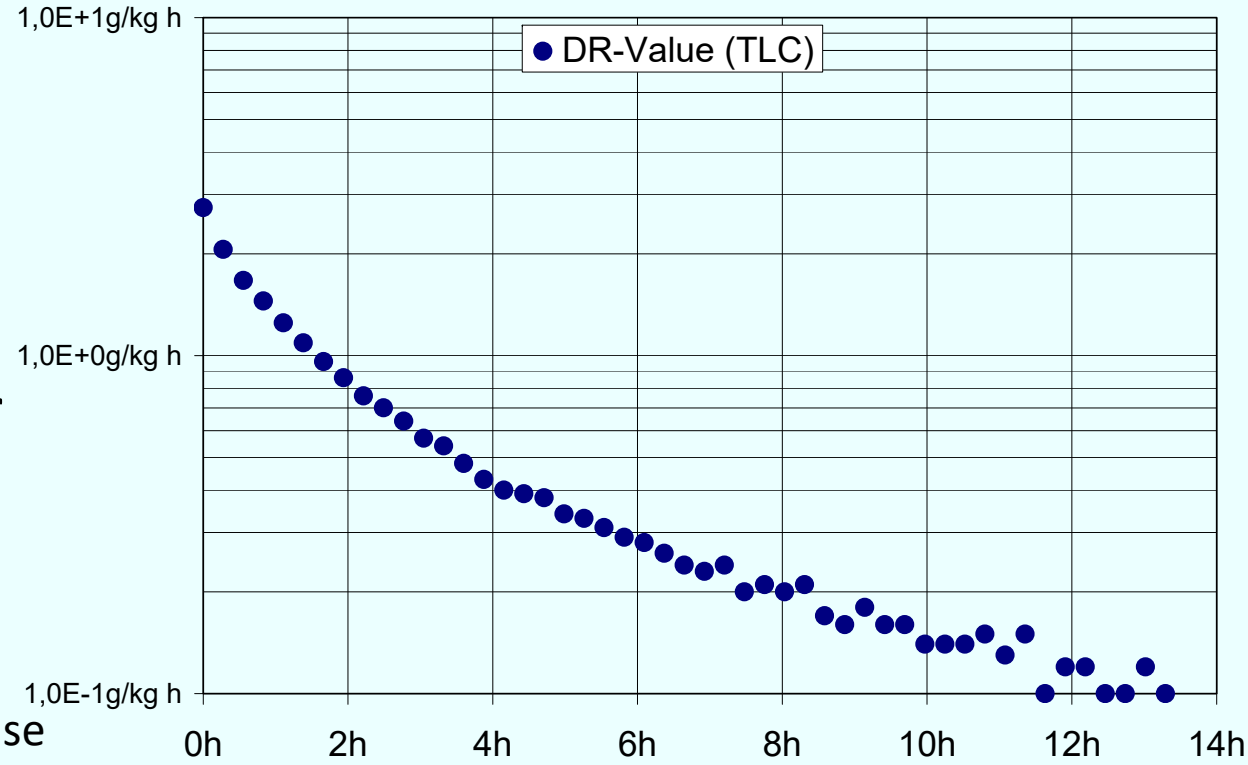
PAT @ Desorption:

Process Control for Desorption

- Shelf Temperature
- Chamber Pressure

Process Feedback for Desorption

- Desorption Rate
  - TDLAS
  - Analysis of Pressure Rise Measurement



PAT @ Desorption:

Further Process Feedback for Desorption

- Vapor Concentration
  - Cold Plasma
  - NIR Gas Analysis
  - Dew Point Detection (minor accuracy)
  - Inert Gas Flow Monitoring (minor accuracy)
  - Comparative Pressure Measurement (minor accuracy)

R&D Equipment

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Some Questions of mine:

✓ Relation between residual Moisture and Desorption Rate?

✓ What is most important, when achiving a minimum and maximum moisture level

✓ PAT Tools for desorption?

Thank you for your attention!

Now its correctly dried

Questions?