

Sublimation

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Overview

- Theory of Sublimation
- Practical aspects of Suplimation
- PAT
- Recipe & Transfer Parameters
- Hands-On: Barometric / Manometric Temperature Measurement



Theory of Sublimation



General Equation for Heat Transfer

$$\frac{dQ_{Cond}}{dQ_{Conv}} + \frac{dQ_{Rad}}{dQ_{Conv}} + \frac{dQ_{Conv}}{dQ_{Conv}} = dQ_{comp}$$

$$\frac{dQ_{comp}}{dt} = k_V \cdot A_H \cdot [T_{Siliconeoil} - T_{Product}]$$

- k_v Very simplified Heat Transfer Coefficient
- A_H Vial Bottom Area
- T_{Product} Product at the Sublimation front



Sublimation – Thermic & Pressure profile

- Frozen water evaporates
- Process requires high energy supply
- Steady conditions are recommended





Heat Flux is stationary

• Each step of energy transfer transfers same amount of energy

• Temperature difference and thermal resistance are proportional

• Temperature gradient in vial is changing during sublimation

$$\frac{1}{K_{V}} = \sum_{i=1}^{i} \left[\frac{1}{K_{i}}\right] \Leftrightarrow K_{V} = \frac{1}{\sum_{i=1}^{i} \left[\frac{1}{K_{i}}\right]}$$

Partial Heat Transfer	Coefficient [W / m² K]
Silicone Oil => Shelf Surface	40250
Shelf Surface => Vial Bottom	820
Vial Bottom => Sublimation Front	1001000

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Theory of Sublimation



Comparison of liquid and gazeous volume of same mass



General Equation for Mass and Heat balance of the vial





Sublimation at the front

- Heat intake equilibrates with heat consumption by ice sublimation
- Driving force for mass flow is pressure difference
- Driving force for heat flux is temperature difference

$$\frac{dm}{dt} = \frac{A_{P}}{R_{PS}} \cdot (P_{i} - P_{C}) = \frac{dQ_{subl}}{h_{subl}}$$





Impact of different Heat intake on Homogeneity

Vialtype:	10ml Vial
Filling:	5ml
Layer:	8mm
T _{Sh} :	0°C
T _{Rad} :	0°C
p _{ch} :	80µbar
t _{End} :	8h
m _{min} :	1,88g; 0,24g/h
m _{max} :	3,64g; 0,45g/h
Base:	3 runs aver.





Equilibrium of T_{Ice}



Limitations of Heat supply

- Heat transfer coefficient in the shelf
- Heat transfer coefficient between shelf surface and sublimation front
- Difference between T_{Ice} and T_{Silicone oil}

Limitations of Sublimation Rate

- Cooling capacity of ice condenser
- Flow resistance of Freeze Dryer
- Vapor transport in product





Course of Freeze Drying at FD-Microscope (Bookcover, Oetjen/Haseley)

Theory of Sublimation

Sublimation progress

• 90s

• 180s

• 270s

• 360s





At the end of sublimation all ice is removed

<u>Questions?</u>



Practical Aspects of Sublimation

Sublimation phase – Hints for a first run (I)

When process vacuum is reached the shelves should be heated up... ...Sublimation starts

• As shown before, the temperature at the sublimation front (T_{ice}) depends on the equilibrium of shelf temperature and chamber vacuum

 $\bullet\ T_{ice}$ must never exceed the critical temperature investigated with one of the previously described instruments

• A proper process vacuum could be the vapor pressure of $(T_{crit} - 5...10^{\circ}C)$

Sublimation phase – Hints for a first run (II)

When process vacuum is reached the shelves should be heated up... ...Sublimation starts

• A first estimation for step duration could be calculated based on sublimation progress of 0,5mm_{Laver}/hour

• Due to variations in Drying Progress over time, changes in Shelf Temperature or Process Vacuum during Sublimation should be avoided

- For a first guess, a conservative Temperature difference between $t_{\rm crit}$ and $t_{\rm shelf}$ of 15°C can be assumed



Optimization of Sublimation phase (I)

- Every raise of T_{ice} by +1°C increases process speed by 10...15%
- Frequently performed Pressure Rise Analysis enable safe monitoring of right $\mathrm{T}_{\mathrm{ice}}$
- The average T_{ice} can be also calculated based on TDLAS results



Optimization of Sublimation phase (II)

- The use of PAT Tools allows the safe control of the right step time
- The ice is safely removed, when
 - 1. the Temperature probes are above the shelf temperature (requires time margin and right positioning of the temperature sensors)
 - 2. T_{ice} has reached the equivalent vapor temperature of the chamber vacuum
 - 3. the Pirani Vacuum converges to the capacitive vacuum closer than 5%
 - 4. the indicated Lyotrack value has reduced below 0,4
 - 5. the Sublimation Rate has reduced to some percents of its maximum value



Practical Aspects Sublimation

Sublimation phase





Practical Aspects Sublimation

Sublimation phase





Temperature Measurement during Sublimation phase

Procedure	Temp. Probes	Manometric / Barometric Temperature measurement	Temperature Calculation by TDLAS
Aseptic Handling	If wireless	yes	yes
Process impact	no	Yes	no
Main Valve close time	No process impact, always open	325s	No process impact, always open
Measuring scope	Indefinable position at vial	Full load	Full load
Other Considerations	Monitoring of some Samples only, Comparability of Samples with Cycle	Detects always the most critical vials in the process	Calculates the general average of t _{ice}



PAT



PAT

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PAT @ Sublimation:

Process Feedback for Sublimation

- Ice Temperature (Frozen Product / sublimation Front)
 - Conventional Sensors (PT100 / TC / Wireless)
 - Barometric / Manometric Temperature Detection







PAT @ Sublimation:

Process Feedback for Sublimation

- Vapor Flow into Condensor
 - TDLAS
 - Analysis of Pressure Rise Measurement e.g. the frequent measurement of Sublimation / Evaporation Rate

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



PAT

PAT @ Sublimation:

Further Process Feedback for

Sublimation

- Vapor Concentration
 - Comparative Pressure Measurement
 - Cold Plasma
 - NIR Gas Analysis
 - MS Gas Analysis
 - Dew Point Detection
 - Inert Gas Flow Monitoring







PAT @ Sublimation:

Process Feedback for Sublimation

- Ice Loss of Load
 - (Micro) Balance at Product Area
 - Sample Thief with Balance
- Ice Increase at Condenser
 - Weighing Function at the Ice Condenser
 - Thickness indication of Ice Layer
- Structural change of Product (Nucleation Sensors)



Recipe & Transfer Parameters





Investigation of the sublimation process

• A freeze Drying Microscope allows a detailed analysis of the drying process and a proper assessment for the suitability of the porosity of the frozen structure.

• The Correlation of the Evaporation Rate with the Water/Ice content allows a safe prediction of the End of Primary Drying

• The profile of T_{lce} vs. Progress can be considered as transfer Parameter. As long this profile is likewise at different Lyos, the processes can be considered as comparable.



Principles for Sublimation

Procedure	Principle	Result				
BTM / MTM product parameter	Indirect calculation of T _{Ice} by Pressure Rise Analysis	Direct control of temperature at sublimation front				
Gas Flow	Indirect calculation of	Determination of end				
process	sublimation rate by	point of sublimation				
parameter	detecting vapor velocity	phase				
Gas Moisture	Direct measurement of	Determination of end				
process	partial vapor	point of sublimation				
parameter	concentration	phase				



PAT

Some Questions of mine :

- \checkmark meaning of K_v?
- ✓ Volume per kg Ice (80µbar, -44°C)?
- ✓ Increase of process speed by raise of T_{ice} +1°C?
- ✓ Difference between $T_{shelf} \& T_{ice}$?
- ✓ Difference between T_{shelf} & T_{silicone oil}?
- ✓ "Comparative Pressure measurement"?
- ✓ Indicators for end point of sublimation?



Hands-on:

Barometric / Manometric Temperature Measurement





Theory of Barometric Temperature Measurement (BTM)

Pressure profile is driving force of lyophilization

Pressure at sublimation front,







Theory of Barometric Temperature Measurement (BTM)





Theory of Barometric / Manometric Temperature Measurement (BTM / MTM)

- Pressure equalizes at equilibrium point
- Further pressure rise is result of steady warming of the whole batch => Risk of product melt
- Equilibrium point and pressure rise characteristic changes with sublimation progress





Theory of Barometric Temperature Measurement (BTM)

• Procedure is sensitive against low number of remaining vials

• A change of shelf temperature for cycle tuning is not recommendable => process inhomogeneity

• The numeric fit-procedure allows the calculation of the results with MS-Excel



Theory of Barometric Temperature Measurement (BTM)

F	$P_{(t)} = P_{lce} - (P_{lce} - I)$	$P_0) \cdot e^{-t \cdot \frac{N \cdot A_p \cdot 62, 3 \cdot T'}{18 \cdot V \cdot R_p \cdot 3600}} + 0,0468$	$5 \cdot P_{lce} \cdot \left(\frac{24,7 \cdot L_{lce} \cdot (P_{lce} - P_{0})}{R_{p}} - 0,0102 \cdot L_{lce} \cdot \frac{\left(T' - \frac{6144,96}{24,01849 - \ln(R_{lce})} + \frac{1-0,0102 \cdot L_{lce}}{1-0,0102 \cdot L_{lce}}\right)}{R_{p}}\right)$	$\overline{\underline{\mathbf{P}}_{lce})}\right) \\ \left(1 - 0.811 \cdot e^{\left(-0.114 \cdot \frac{t}{L_{lce}}\right)}\right) + EX \cdot t$
	Symbol	Unit	Description	
	P _(t)	[Torr]	Chamber pressure	Measured
	P _{lce}	[Torr]	Vapor pressure at sublimation front	To be solved
	_			

P _{Ice}	[Torr]	Vapor pressure at sublimation front	To be solved
P ₀	[Torr]	Chamber pressure at start of BTM	Measured
Ν	[]	Number of vials	To be known
A _p	[cm ²]	Average surface of the vial	To be known
T'	[K]	Shelf temperature	Measured
V	[1]	Effective chamber Volume	To be known
R _p		Flow resistance of dried cake	To be solved
L _{Ice}	[cm]	Filling level, Layer thickness	To be known
EX		Linear part of pressure rise due to leak rate and warming of product	To be solved
t	[s]	Time	Measured



Hands On











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Thank you for your attention!

<u>Questions?</u>



Addendum

Table of critical temperatures

Sucrose	amorphous	307,9µbar	-32°C
Mannitol	crystalline	3.685,0µbar	-6°C
Lactose	amorphous	466,9µbar	-28°C
NaCl	crystalline	850,2µbar	-22°C
CaCl ₂	crystalline	23,8µbar	-54°C



Addendum

Preparation to use the Excel-Solver

- Activate Macros / Deactivate "protection"
- Register Solver in MS-Excel
- Install Solver as VBA-Reference



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8	Vchami	ber	66	6,60E-2n	n ³	Tlce															
9	Filling	height	8 mm	0,80	m	T _{shelf}						Lö	schen								
10												_ Opti	ionen								
11						Rp	2,87E+1cm		1					(I/s							
12						EX	1,3	Makros in:	Alle offenen Arb	peitsmappen		•		bar/s	5,7E-5Torr/s						-
13							-	Beschreibung	g					mbar							-
14	Filling		3,00	3			_														-
15	Solid	Contont	31 50	3 155 24	0		_														-
17	Solid	Content	01,0g	0, TUL-2h	y							Abb	brechen		~			5		8	
18	Press	ure Rise	2.0E-1mbar	2.0E+2ub	ar			-		-	-	_									
19	Δt		24,0s				lce	Temperature	e Fit												
20	ER		4,9E-2µbar																		
21																					
22																					
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24																					-
14	< > H	Cockpi	t / Input / Diagra	am / Examp	le 01 /	Example 0	2 / Example 03	Example (04 / Example	e 05 / Exa	mple 06	Examp	•					_		•	
Bereit												0									
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Aicrosoft Visual Basic for Applications	- BTM-Calculato	Berlin.xlsm - [Modul2 (Code)]		
📴 Datei Bearbeiten Ansicht Ein	fügen Forma <u>t</u>	Debuggen Augsführen Extras Add-Ins Fenster ?	Frage hier eingeben	×
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Projekt - VBAProject	×	(Allgemein)		
Tabelle 1 (Cockpit)	-	Sub Clean_Tab Sheets ("II Ontionen		
Tabelle2 (Input)		Range ("A2 Eigenschaften von VRADroject		
Tabelle3 (Example 01)		Selection		
Tabelle4 (Example 02)	_	Range ("C3 Digitale Signatur		
Tabelle5 (Example 03)		Selection.ClearContents		
Tabelle6 (Example 04)		End Sub		
Tabelle7 (Example 05)	E	Sub Iterate()		
Tabelle8 (Example 06)				
Tabelle9 (Example 07)		SolverOk SetCell:="\$F\$6", MaxMinVal:=2, ValueOf:="0", ByChange:="\$F\$2:\$F\$4"		
		Solversolve		
Modul2	-	End Sub		
Eigenschaften - Tabelle1	×			
Tabelle1 Worksheet	•			
Alphabetisch Nach Kategorien				
(Name) Tabelle 1				
DisplayPageBreaks False				
DisplayRightToLeft False				
EnableAutoFilter False				
EnableCalculation True				
EnableFormatConditionsCalcula True				
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Then close all windows, except Excel table

...Run "solver" once manually...

..and careful listen to the further explanations of the instructor