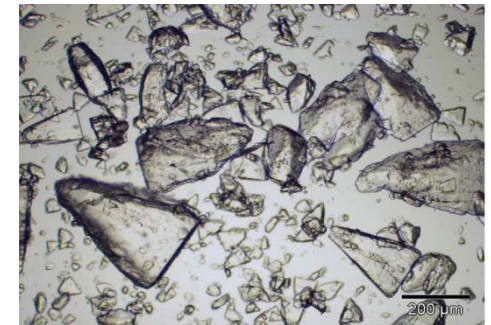
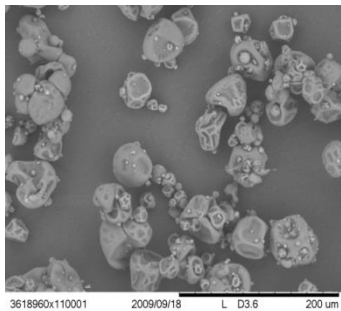
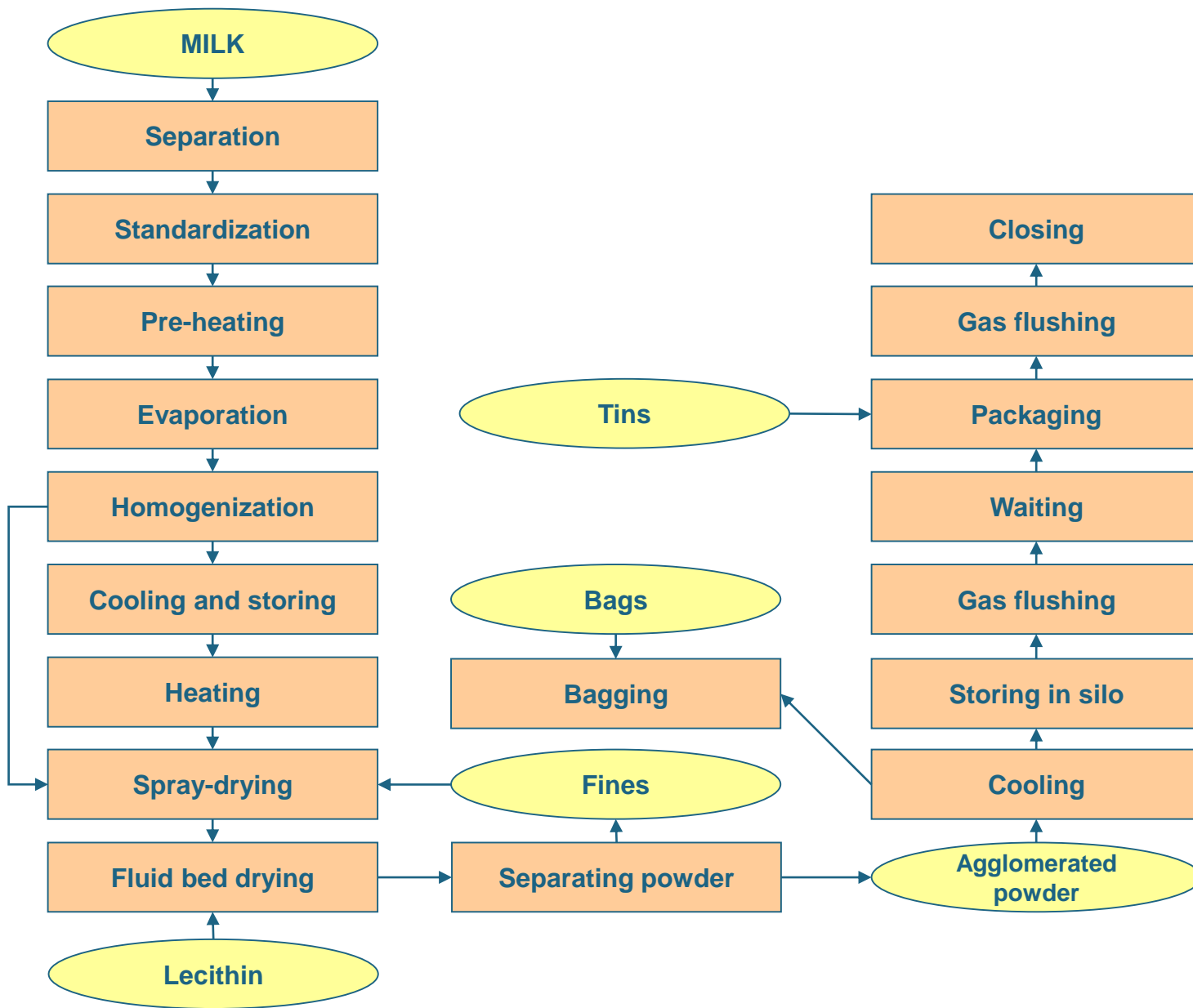


WHOLE MILK POWDER MANUFACTURE: OPPORTUNITIES AND CHALLENGES



thom.huppertz@nizo.com



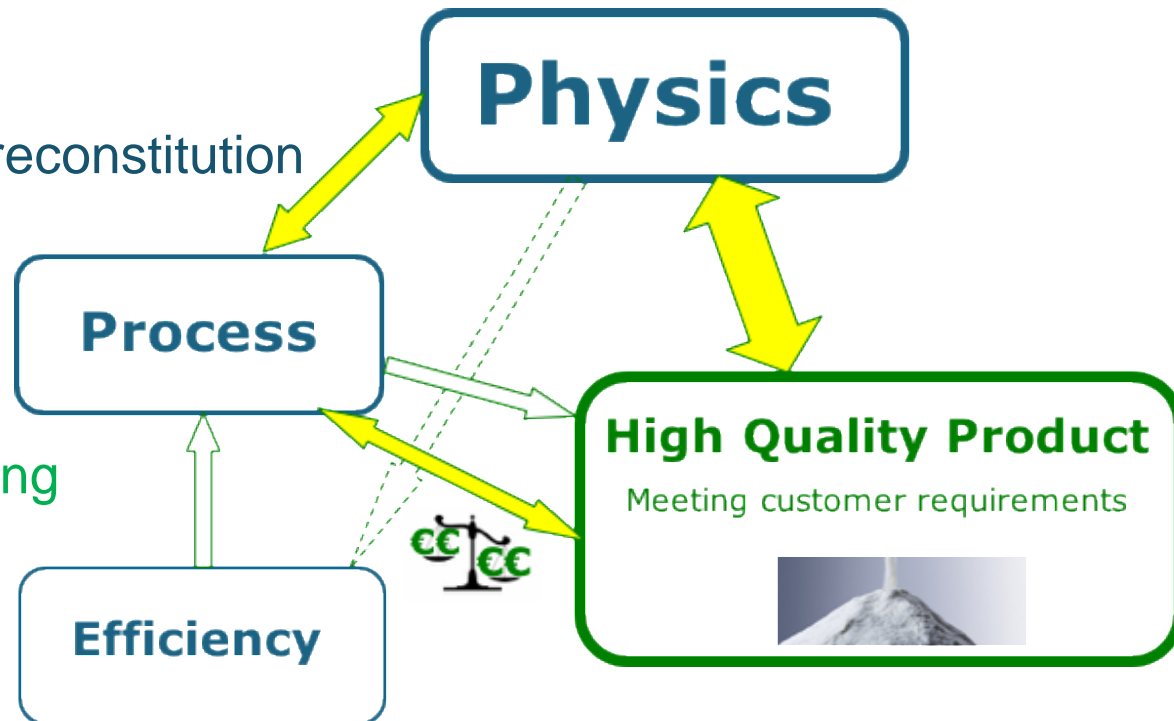
The ideal powder

For the user:

- Delivers all required nutrition
- Excellent physical appearance of powder
- Easy to handle
- Easy to dissolve
- No physical defects after reconstitution
- No off-flavor or off-taste

For the manufacturer:

- High capacity and yield
- No issues during processing
- Long shelf-life



What is needed to be on top of the game?

Know everything about **Product Characteristics**

- **Physics**, phase diagrams, sorption, T_g
- Sensitive ingredients
- Morphology in relation to bulk properties
 - Density, flow, reconstitution, etc.

Know everything about **Drying Technology**

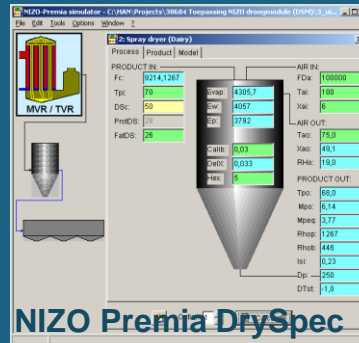
- Unit Operations, process steering & control, Modeling processes
- Heat / Mass balances, Mollier Diagram
- Various drying techniques
- Air de-humidification

And being able to **combine and apply** this knowledge

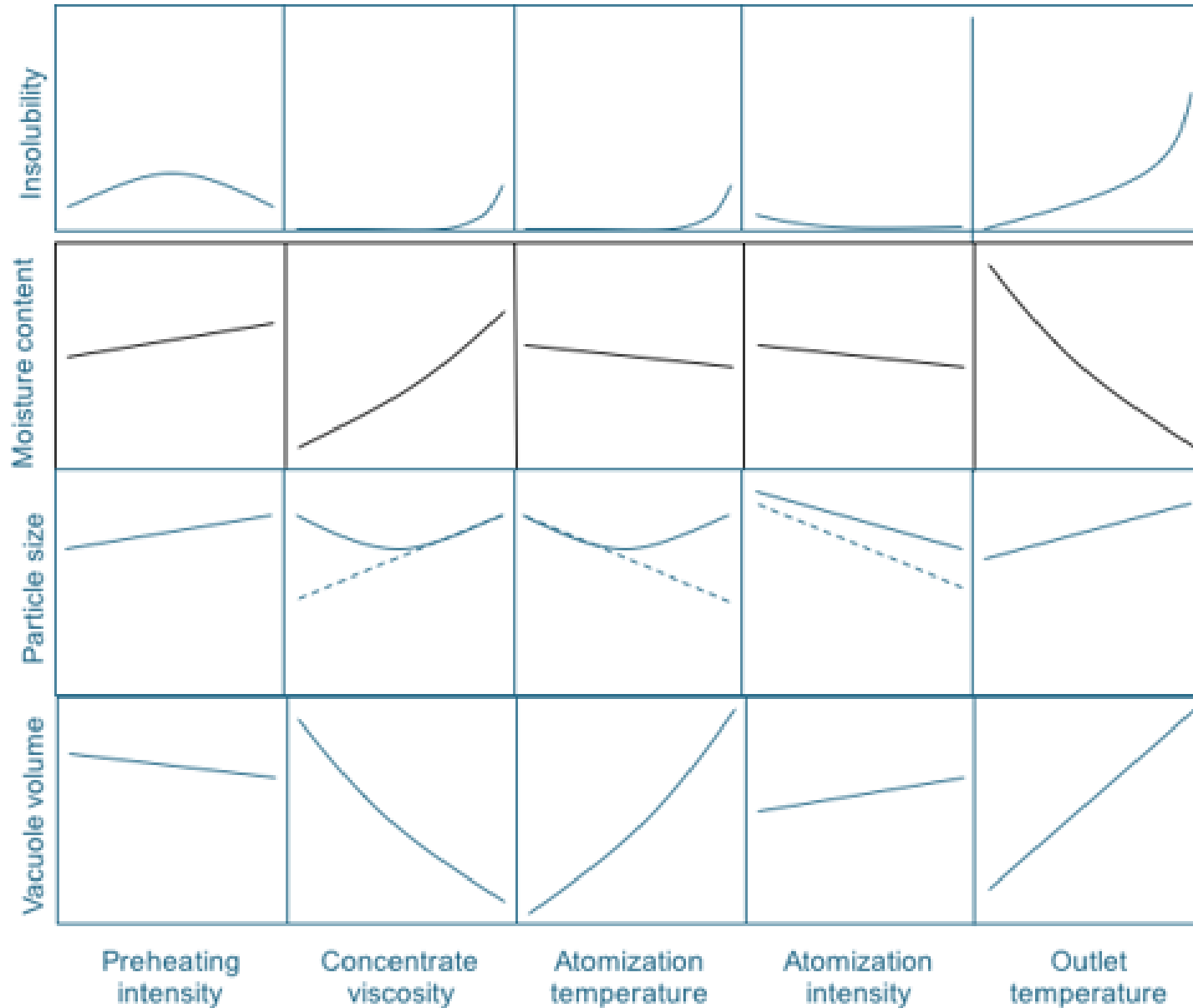
Product characteristics

Characterize your powder:

- Bulk density (free/tapped/true)
- Particle size distribution
- Specific surface area
- Particle density
- Stickiness (T_{sticky})
- Modulated DSC (T_{glass})
- Water sorption isotherm
- Dispersability
- Wettability
- Flowability
- Microscopy
- Process simulation
- Computational Fluid Dynamics



Controlling product properties



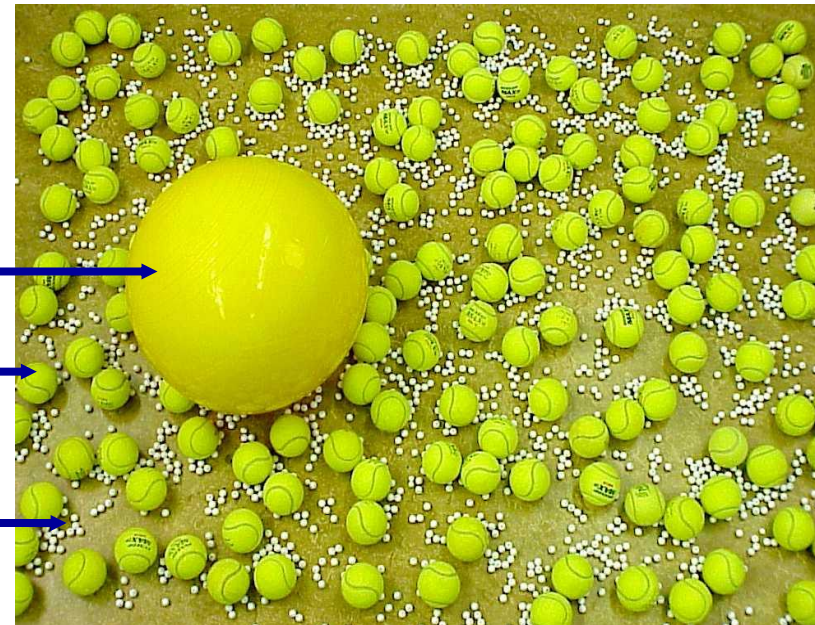
Particles in milk

	Average composition %	Emulsion type oil/water	Colloidal solution/suspension	True solution
Moisture	87,0			
Fat	4,0	X		
Proteins	3,5		X	
Lactose	4,7			X
Ash	0,8			X

fat globule

casein micelle

Whey proteins



Whey protein denaturation

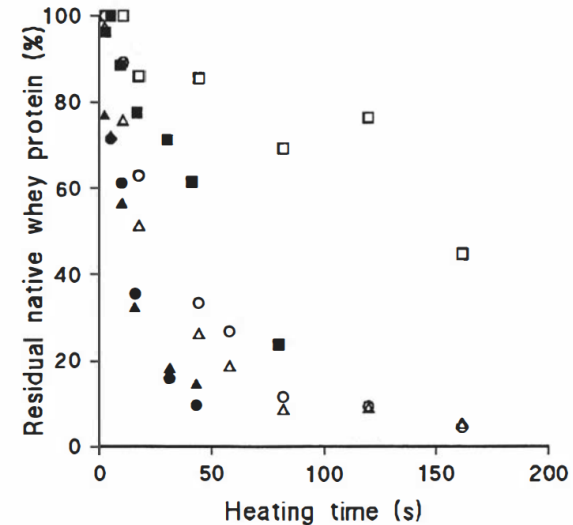
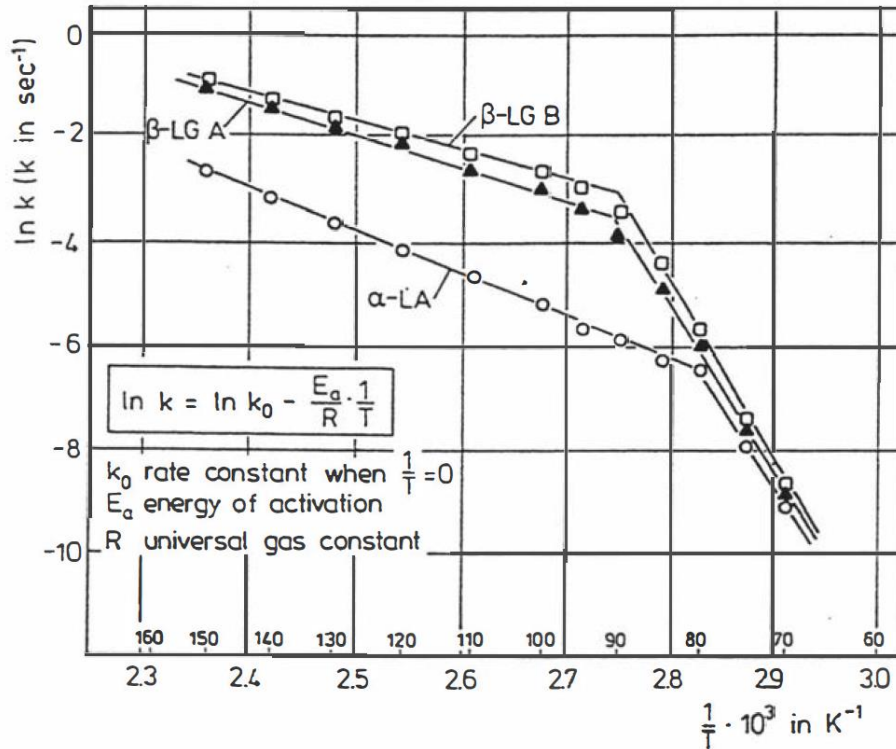
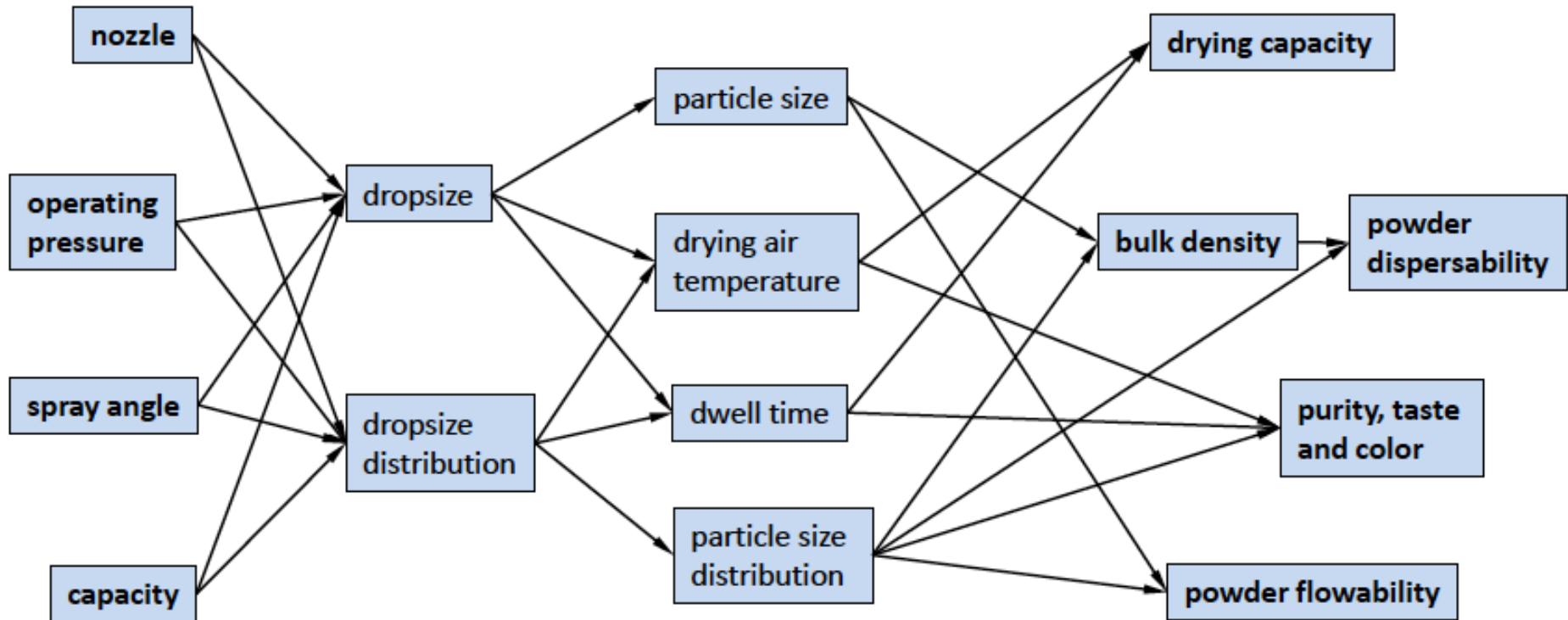


Figure 4.5 Loss of individual whey proteins from native-PAGE gels of ultracentrifugal supernatants obtained from skim milk heated at 100°C, β -lg A (O), β -lg B (Δ), α -la(\square), and 120°C, β -lg A (\bullet), β -lg B (\blacktriangle), α -la(\blacksquare).

- Whey proteins differ in susceptibility to thermal denaturation
- b-Lactoglobulin generally is the main determinant of whey protein denaturation and aggregation because of:
 - Highest concentration
 - Free—SH group which governs aggregation behavior

Atomization is key

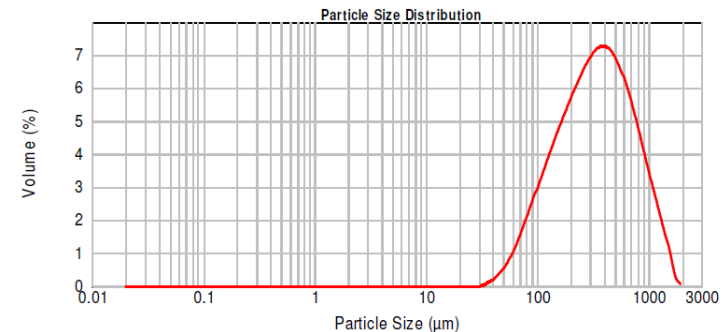


Droplet size distribution effects on the operational spray drying process

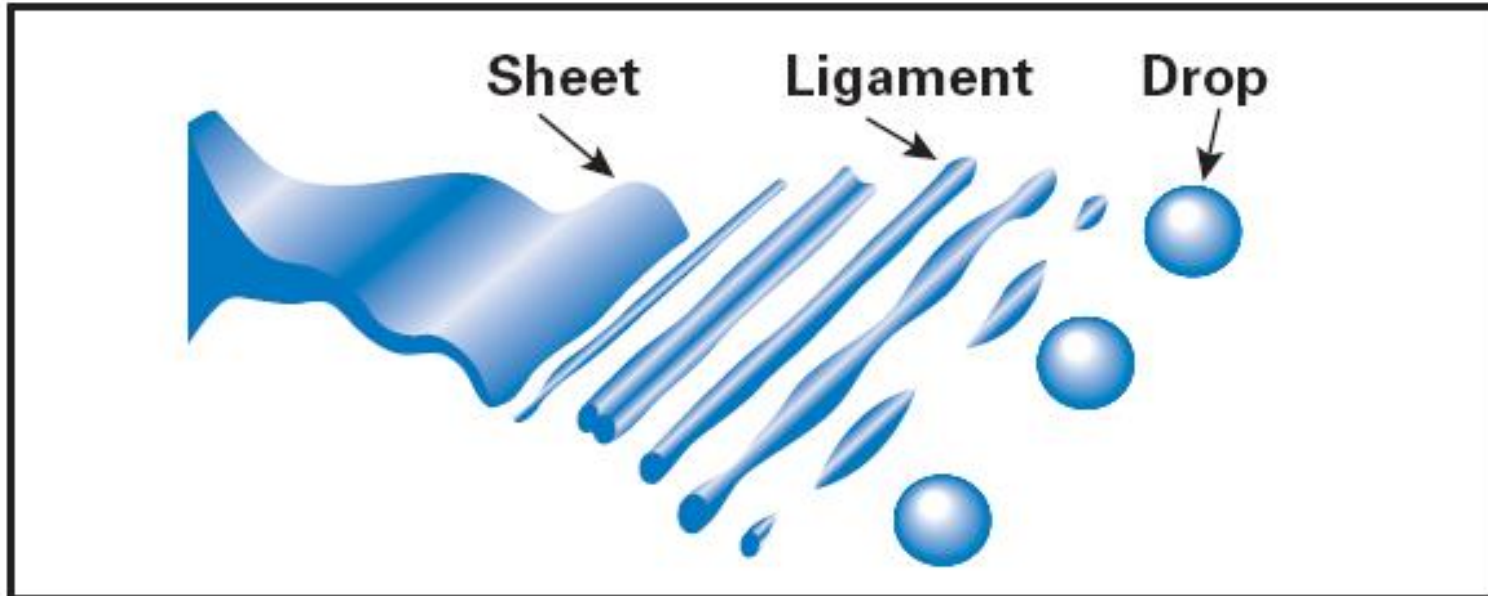
- Surface area is proportional to evaporation rate:
 - Large particles will dry slow
 - Small particles will dry fast
- Insufficient dried droplets can cause:
 - Fouling of the drying chamber
 - Blocking of the rotary valve
 - Lumping in internal static bed
 - Blocking of cyclones
- Very small particles can cause:
 - Overloading of the cyclones, plugging of bag houses
 - Powder emissions/product losses

→ Ideally, very small and very large droplets should not be present in a spray:

- Maximize D_{10}
- Minimize D_{90}



Atomisation



Specific surface area (a) in m^2/m^3 :

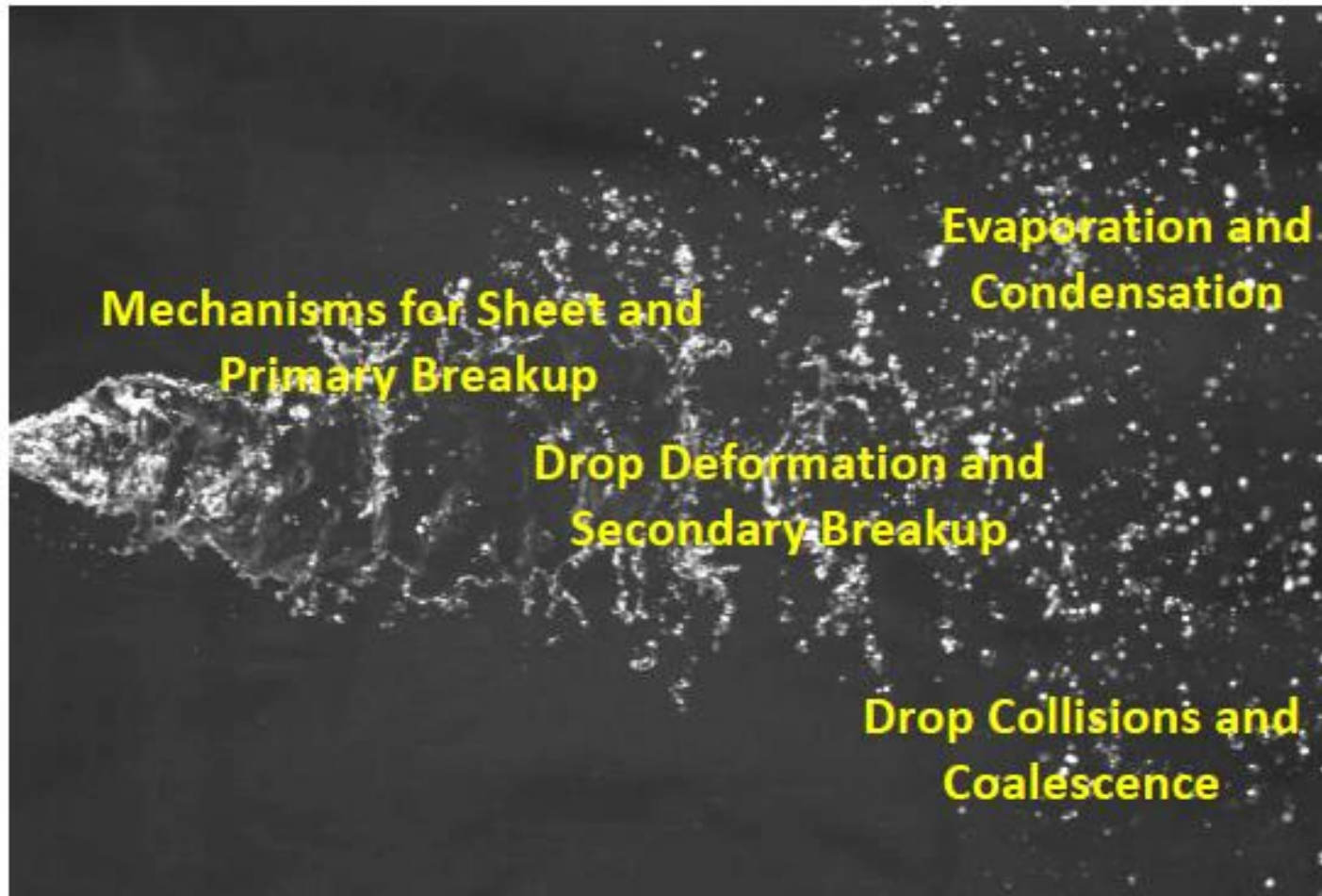
$$a = \frac{A_p}{V_p} = \frac{\pi d_p^2}{\frac{\pi}{6} d_p^3} \quad a = \frac{6}{d_p}$$

d_p μm	a m^2/m^3
1000	6.000
100	60.000
10	600.000

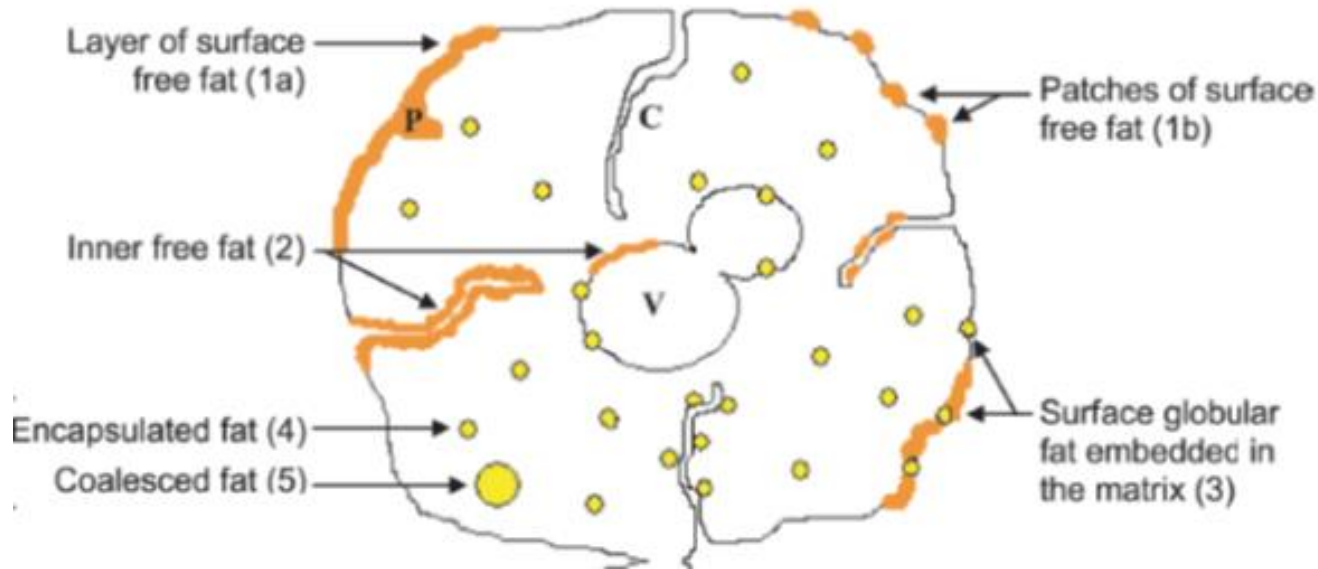
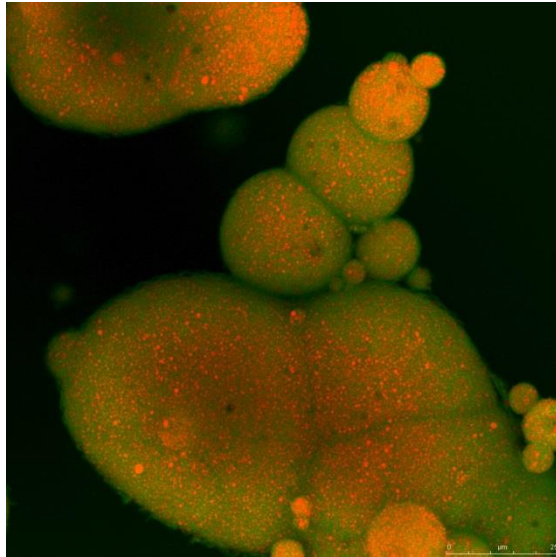
Factors affecting droplet size

Effect of	On droplet size
pressure ↑	↓↓↓ increase of out flow velocity
spray angle ↑	↓↓ film sheet thickness gets smaller
capacity size of spray nozzle ↑	↑↑ larger orifice dimensions
viscosity of liquid ↑	↑↑ higher inner friction results in larger droplets (fibers)
surface tension ↑	↑ larger wave length
liquid density ↑	↑ decrease of out flow velocity for const. liquid pressure

Controlling different processes during atomization



Fat distribution in dairy powders



- Fat in dairy powders distributed in various fractions on surface and in core of powder particles
- Emulsion is partially destabilized during drying

Table I. Types of fat within a powder particle: main associated analytical methods. Identification numbers and letters refer to captions in Figure 2: 1a: layer of surface free fat; 1b: patches of surface free fat; 2: inner free fat; 3: surface globular fat; 4: encapsulated fat; 5: coalesced fat. Abbreviations: CLSM: confocal laser scanning microscopy; SEM: scanning electron microscopy; TEM: transmission electron microscopy; XPS: X-ray photoemission spectrometry.

	Quantitative method	Qualitative method
Surface free fat 1a + 1b	Short-time solvent-extraction method with apolar solvent	SEM before/after extraction
Total free fat 1a + 1b + 2	Solvent-extraction method with apolar solvent	CLSM
Surface fat 1a + 1b + 3	XPS	SEM before/after extraction
Total fat 1a + 1b + 2 + 3 + 4 + 5	Solvent-extraction method with polar solvent	CLSM TEM

Surface properties

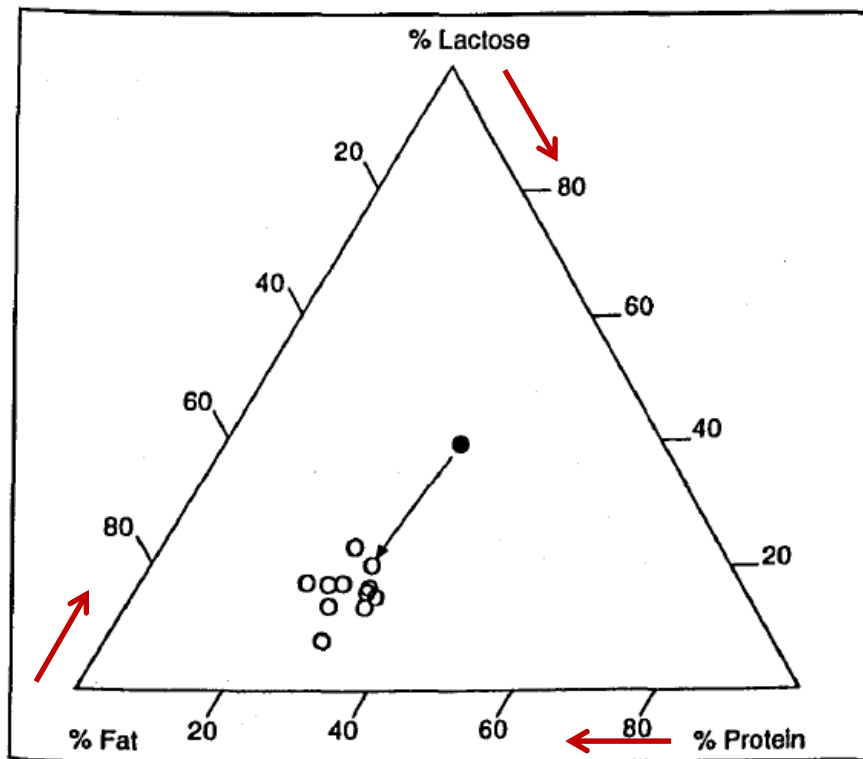
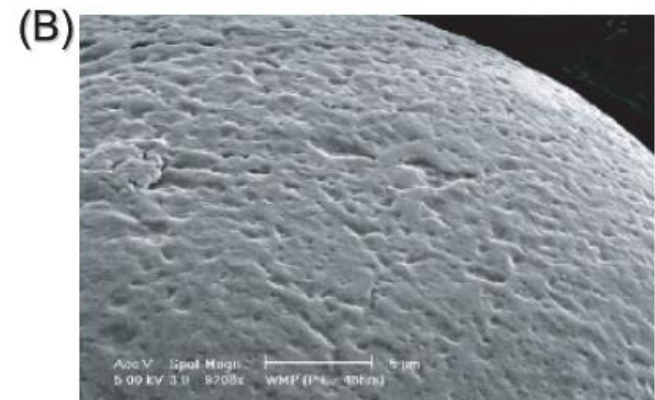
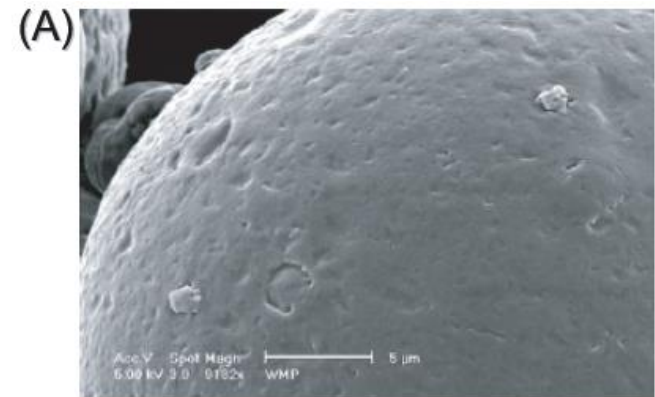


Fig. 2: Surface composition estimated by ESCA for powders produced in the production plant after fluid bed. ● composition of whole milk ○ surface composition of whole milk powder.

Before fat extraction



After fat extraction

- Fat is over-represented on the surface of milk powder particles (35% of product, 70% of surface)

Surface fat in dairy powders

Table 1.4 Surface composition^a of industrial spray dried dairy powders and skimmed milk powders with different lactose levels.

Powders	Bulk composition (g 100 g ⁻¹)			Surface composition ^b (%)			References
	Lactose	Protein	Fat	Lactose	Protein	Fat	
SMP	58	41	1	36	46	18	Kim <i>et al.</i> (2005)
WMP	40	31	29	2	—	98	
CP	13	12	75	1	—	99	
WPC	8	86	6	6	41	53	
SMP:lactose (3:1)	63	26	0.8	29	61	10	Shrestha <i>et al.</i> (2007)
SMP:lactose (1:1)	75	17	0.5	31	58	11	
SMP:lactose (1:3)	88	9	0.25	39	57	5	

WMP = whole milk powder; SMP = skimmed milk powder; CP = cream powder; WPC = whey protein concentrates.

^aAssuming that dairy powders are composed of three main components, namely, lactose, protein and fat.

^bBased on data from X-ray photoelectron spectroscopy (XPS) or electron spectroscopy for chemical analysis (ESCA).

Fat is over-represented on the surface of powder particles

Controlling free fat

- Controlling free fat is about controlling:

- Emulsion stability
 - Smaller fat globules more stable
 - Protein-covered fat globules more stable

- Powder particle matrix

- Higher inlet temperatures reduce free fat
- Lower outlet temperatures reduce free fat
- Lactose crystallization can damage fat globules

- Changes in emulsion and powder particle matrix during storage

- Crystallization of fat during storage can damage fat globules
- Lactose crystallization during storage can damage fat globules

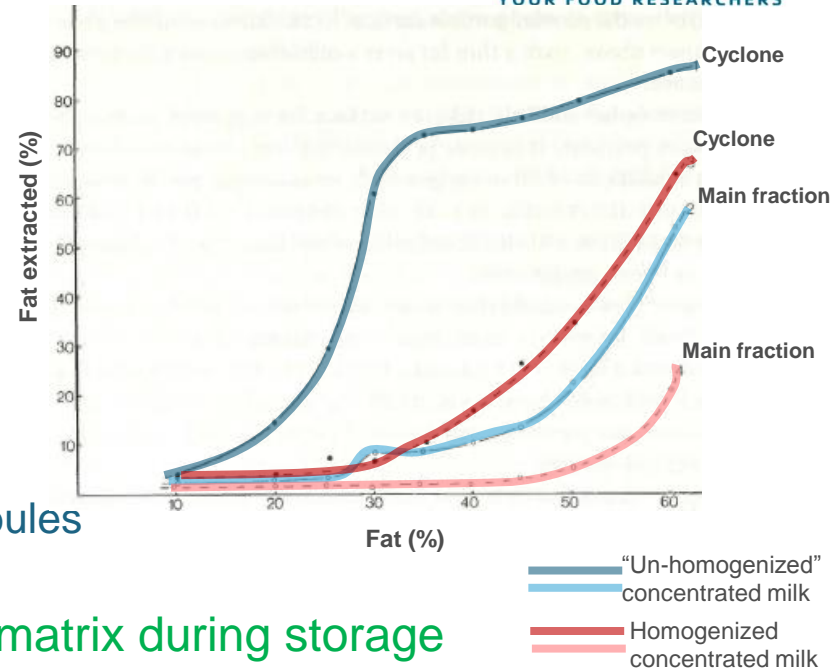
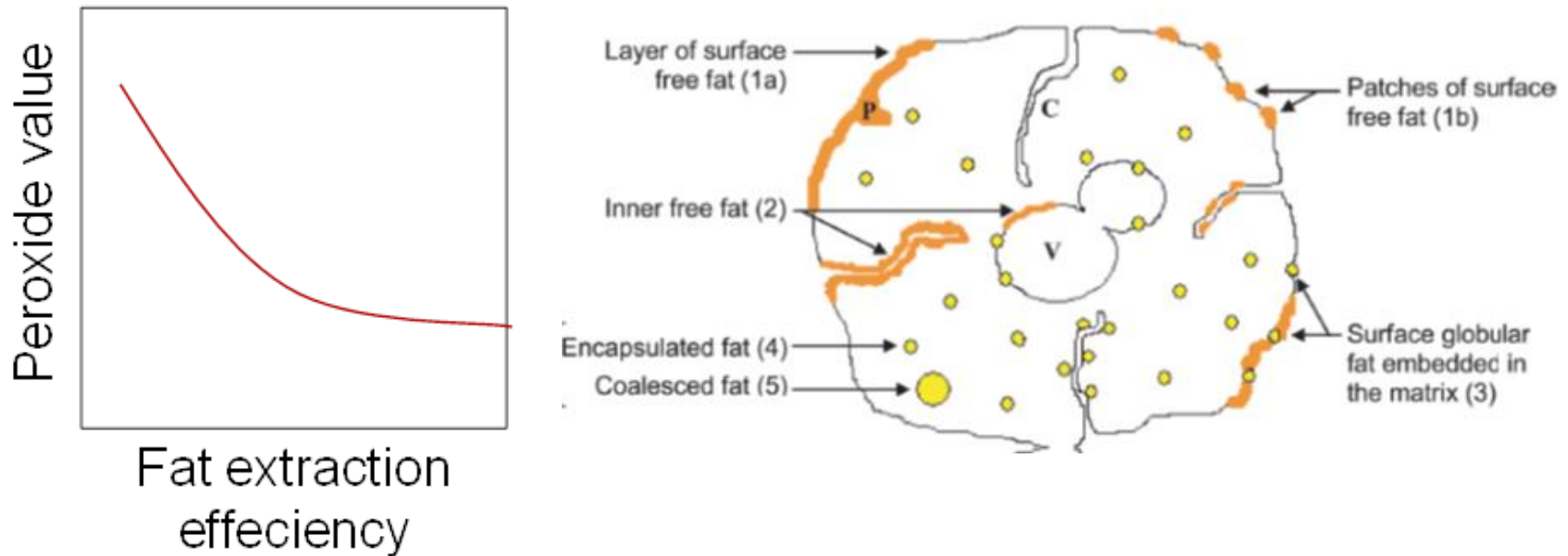


Table 1. The percentage of fat extracted by two methods from five whole milk spray powders and the percentage of fat present as fat globules with diameters $> 2 \mu\text{m}$.

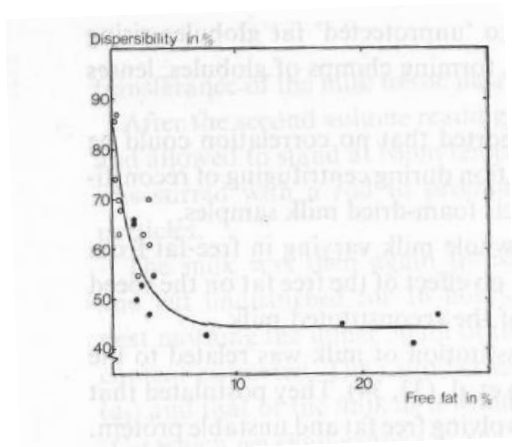
Sample number	Percentage of fat extracted			Percentage fat in globules $> 2 \mu\text{m}$
	10 min 22°C	20 h 40°C	difference	
1	1.6	2.5	0.9	0.3
2	9.9	10.1	0.2	0.6
3	8.0	14.2	6.2	11
4	9.1	25	15.9	15
5	16.8	59	42.2	27

- Peroxide value (PV) and p-anidine value (pAV) important indicators for oxidation

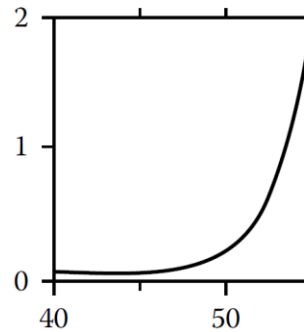


- Surface fat more readily oxidized
- Reliable values are only obtained if fat extraction is good

Solubility issues

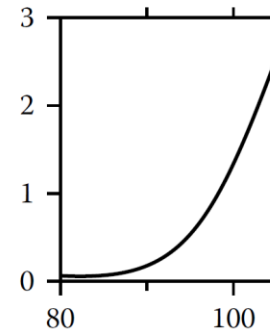


Insolubility



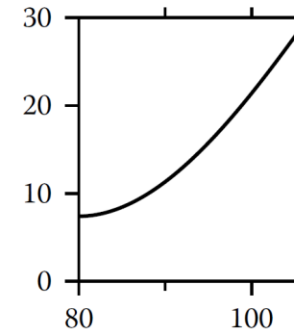
Dry matter (%)

Insolubility

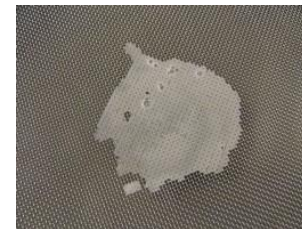


Temperature (°C)

Extractable fat (%)



Temperature (°C)



- Free fat strongly impairs the dispersibility of whole milk powder
- Hydrophobic surface of powder particles gets wetted poorly by water

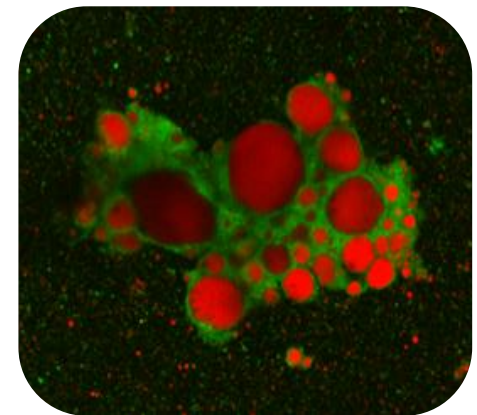
Insolubility development

Thermal stability of skim milk concentrate/powder^a

Moisture content (%)	ISI ₀ (ml)	T (°C)	t _{ISI=0.3} (s)
3	<0.05	70	1.8 × 10 ⁵
7	<0.05	70	1.1 × 10 ⁴
3	<0.05	100	1200
7	<0.05	100	720
9	<0.05	95	20
12	<0.05	95	<7
14	8.5	–	–
25	9.0	–	–
35	<0.05	–	–
40	<0.05	–	–
50	<0.05	85	275
50	<0.05	95	50

^aISI₀ = insolubility index before heat treatment; t_{ISI=0.3} = heat-holding time needed to increase ISI to a value of 0.3 ml.

- Rapid insolubility development between 10 and 30% dry matter
- Protein aggregation as combination of:
 - High temperature
 - High protein
 - Low pH
 - High ionic strength
- In whole milk powder, insolubility may also show as aggregation of protein-stabilized emulsion droplets



Insolubility development during drying

Table 2
Insolubility index from experiments on pilot-scale dryer^a

		$T_{ai} / T_{ao} (^{\circ}\text{C})$			
		94/77	104/86	114/96	134/114
Moisture content (%)	Powder	6.80	6.85	5.36	4.02
	Fraction 1	6.71	6.77	5.41	4.84
	Fraction 2	6.65	6.68	5.33	4.39
	Fraction 3	7.21	7.18	5.80	4.31
$d[v, 0.5]$ (μm)	Powder	40	57	45	58
	Fraction 1	21	22	22	27
	Fraction 2	69	68	67	68
	Fraction 3	144	156	139	143
ISi (ml)	Powder	<0.05	<0.05	0.35	2.80
	Fraction 1	<0.05	<0.05	0.05	0.55
	Fraction 2	<0.05	<0.05	0.20	2.50
	Fraction 3	<0.05	0.15	0.80	4.80

^a T_{ai} = inlet air temperature; T_{ao} = outlet air temperature; $d[v, 0.5]$ = volume median diameter; ISi = insolubility index; fraction 1: $d < 45 \mu\text{m}$; fraction 2: $45 < d < 90 \mu\text{m}$; fraction 3: $d > 90 \mu\text{m}$.

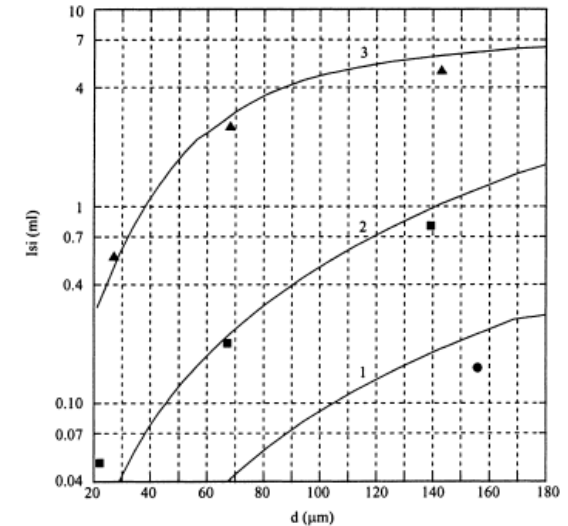
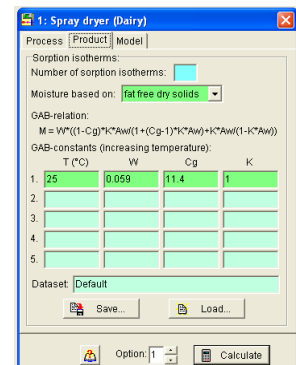
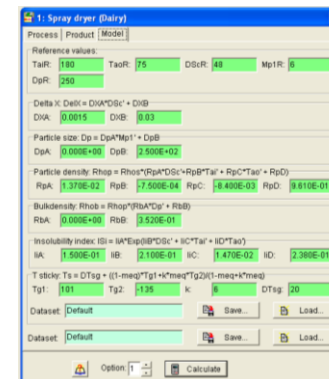
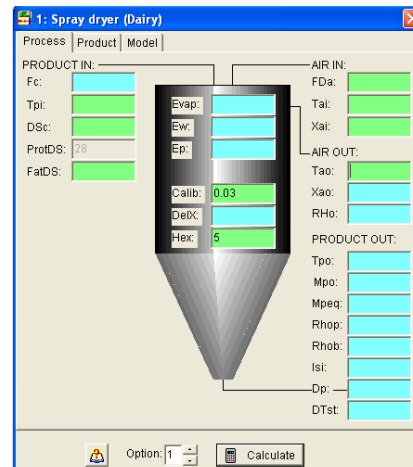


Fig. 2. ISi versus particle diameter. Experimental results (symbols) and model calculation (lines). Temperatures: 86°C (1, ●); 96°C (2, ■) and 114°C (3, ▲). Model constants: $k_0 = 0.054 \text{ ml/s}$; $E_a = 2.7 \times 10^5 \text{ J/mol}$; $T_0 = 348 \text{ K}$.

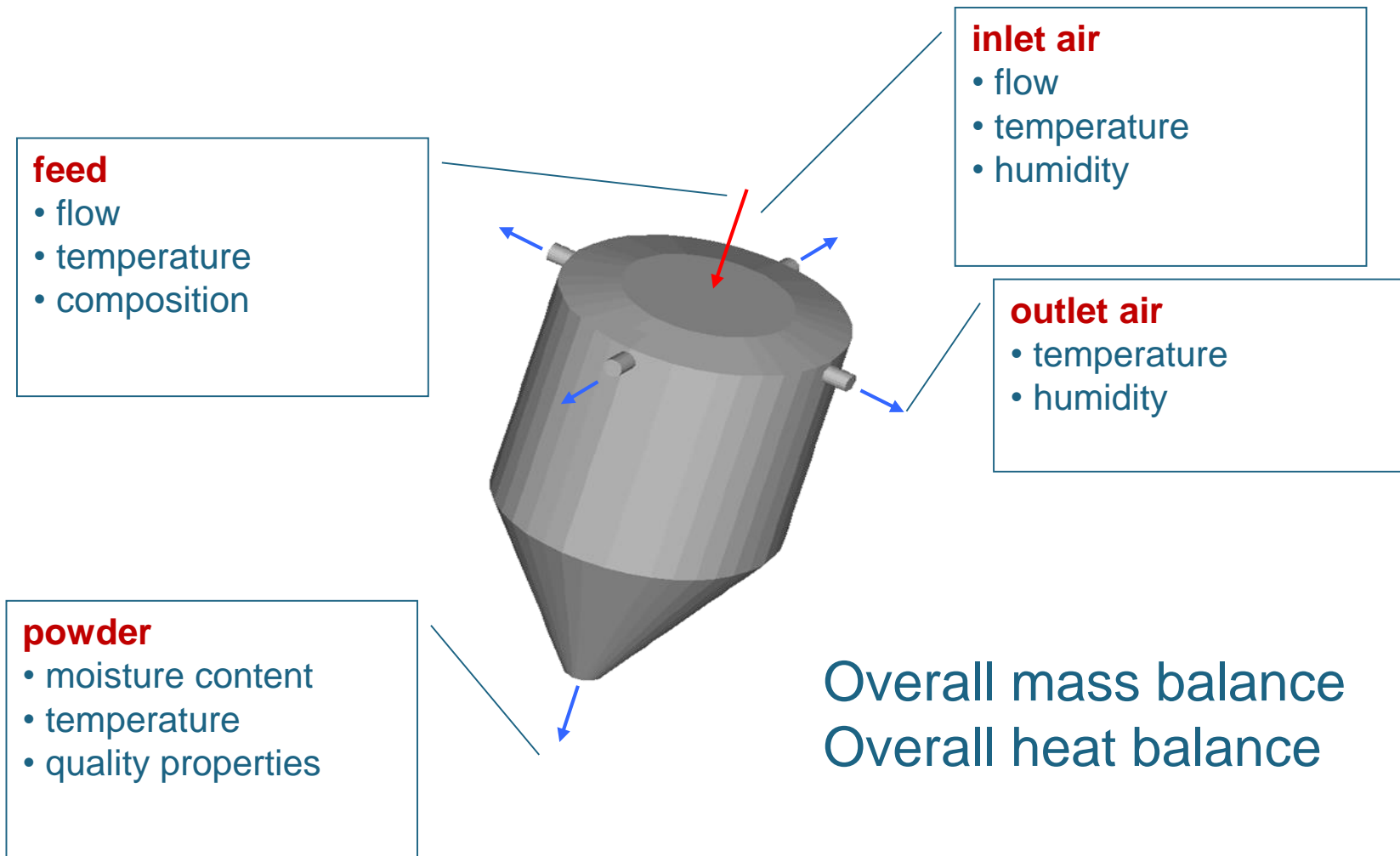
- Relation between particle properties and insolubility index can be used to model ISi as a function of droplet diameter and temperature
- ISi increases strongly with particle diameter
- Apply for predictive modeling of insolubility during drying

Model-based process optimization

- **Dryer settings optimized by trial-and-error**
 - Changing weather conditions...
 - Time consuming
 - Err on side of caution to prevent fouling
- **NIZO Premia Dryspec**
 - Enables process experts to quickly find opportunities in current food production lines



DrySpec 3



Insolubility index

➤ Increase heat load in protein containing product



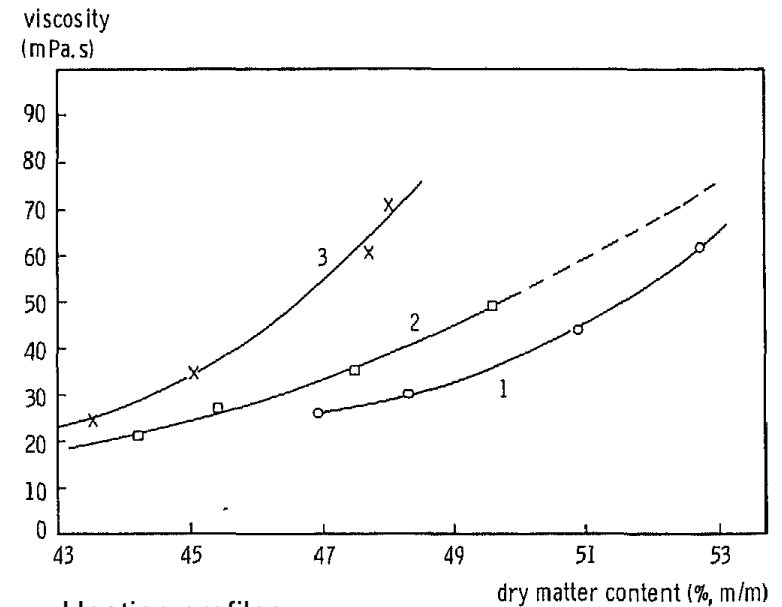
• Microbial control improved



• Feed viscosity increases



• Insolubility index (Isi) increases..



Heating profiles

1: 10s, 70 C; 2: 1 min, 85 C; 3: 5 min, 95 C

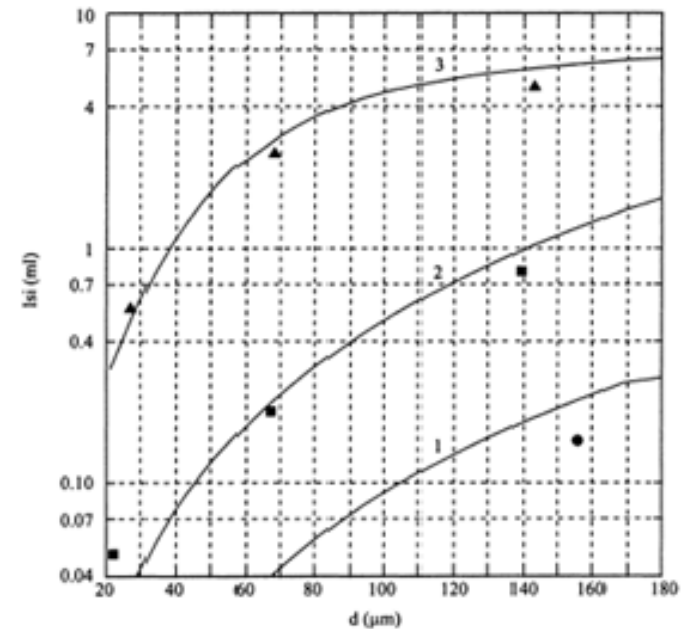
Solution: apply the theory

Understand:

- ISI as a function of the temperature-moisture history of the particles
- higher viscosity \rightarrow larger droplet \rightarrow higher heat load \rightarrow higher ISI

Optimize:

- kinetic Arrhenius type model coupled with CFD calculation
- ISI model: improve powder solubility while maintaining microbial quality



ISI versus particle diameter.

Experimental results (symbols) and model calculation (lines).

Temperatures: 86 °C (1, \blacklozenge); 96 °C (2, \blacksquare) and 114 °C (3, \blacktriangle).

Controlling water relations in milk powder properties to ensure efficient processing and handling and storage stability



Fouling of dryers



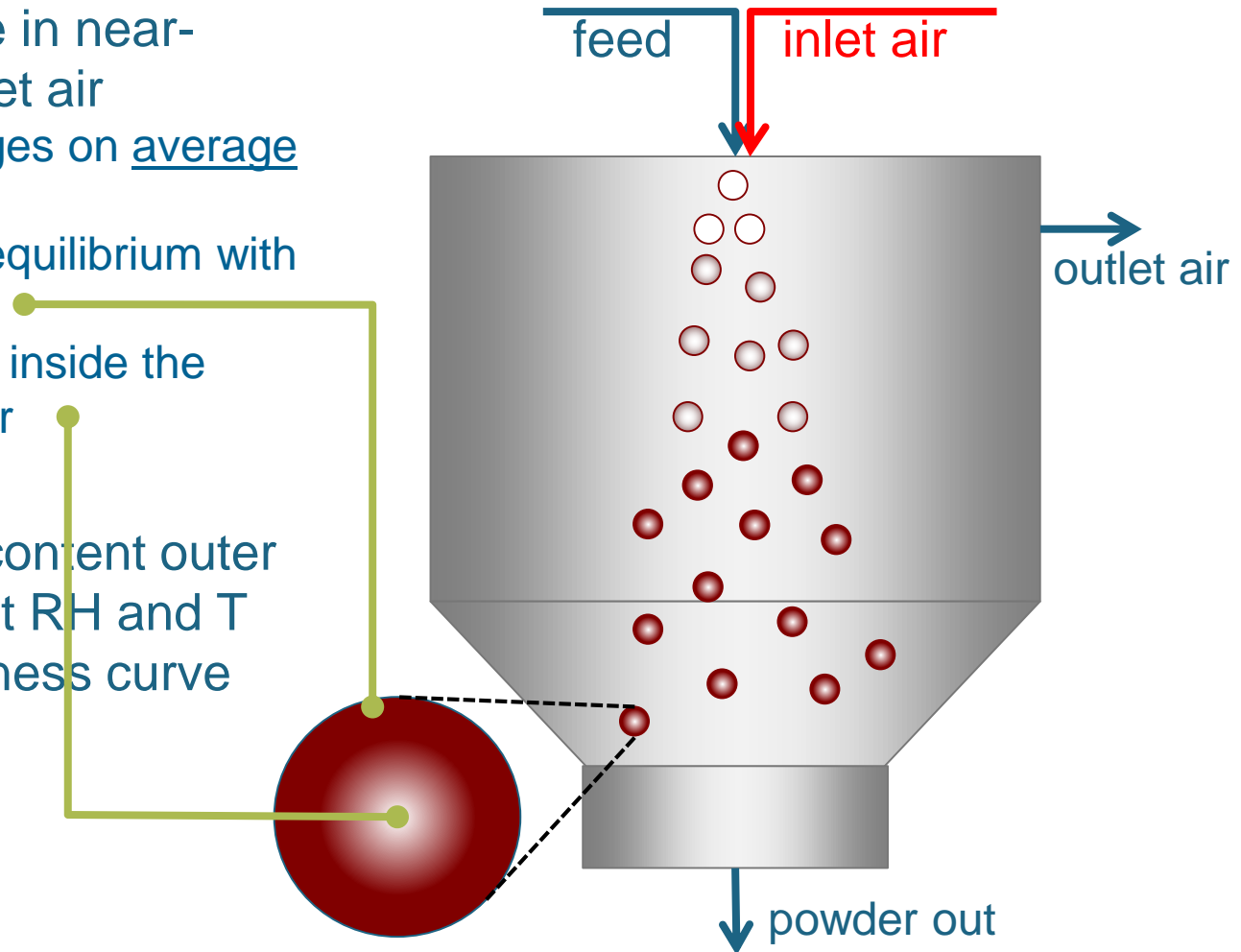
Impaired flow



Caking and
browning

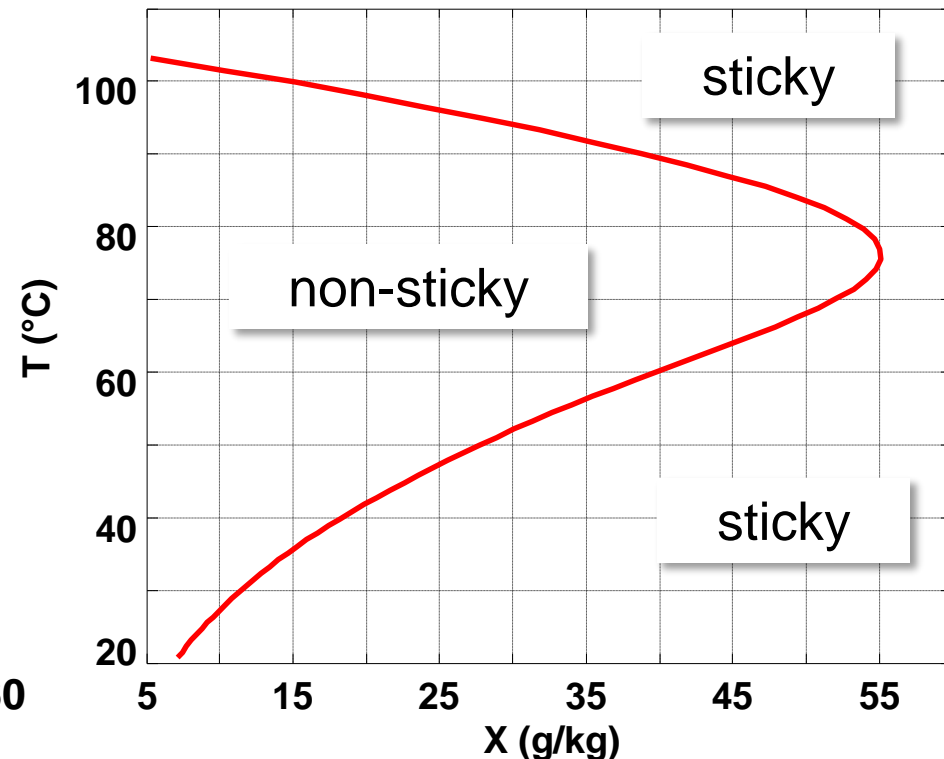
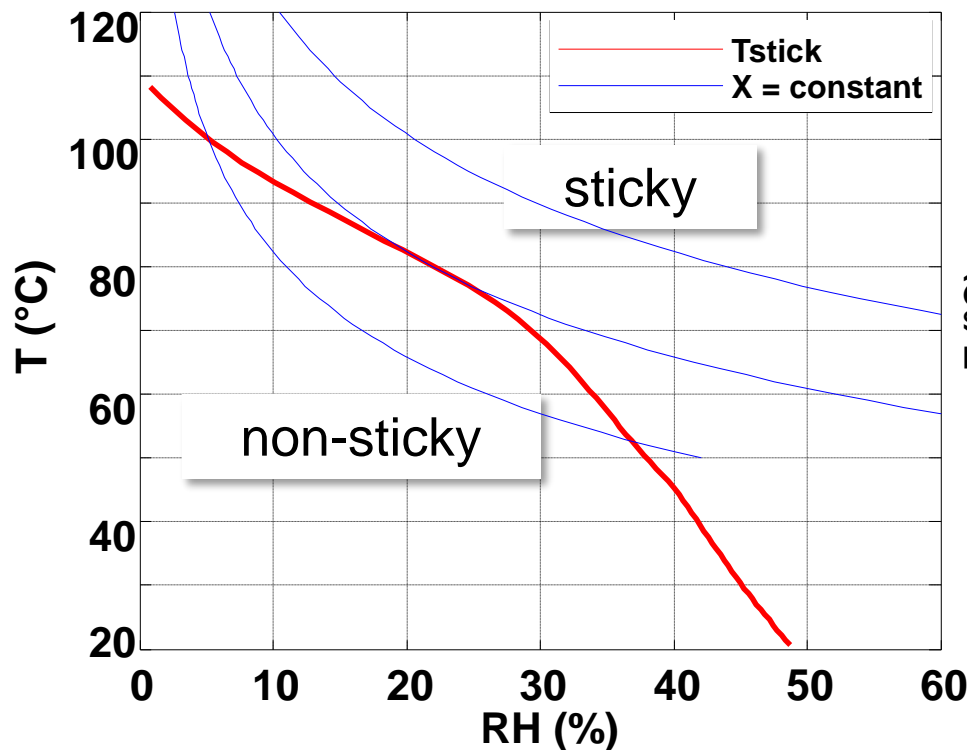
Stickiness

- Powder particles are in near-equilibrium with outlet air
 - DrySpec calibrages on average moisture content
 - outer layer is in equilibrium with outlet air
 - moisture content inside the particles is higher
- Calculate moisture content outer layer based on outlet RH and T
- Compare with stickiness curve



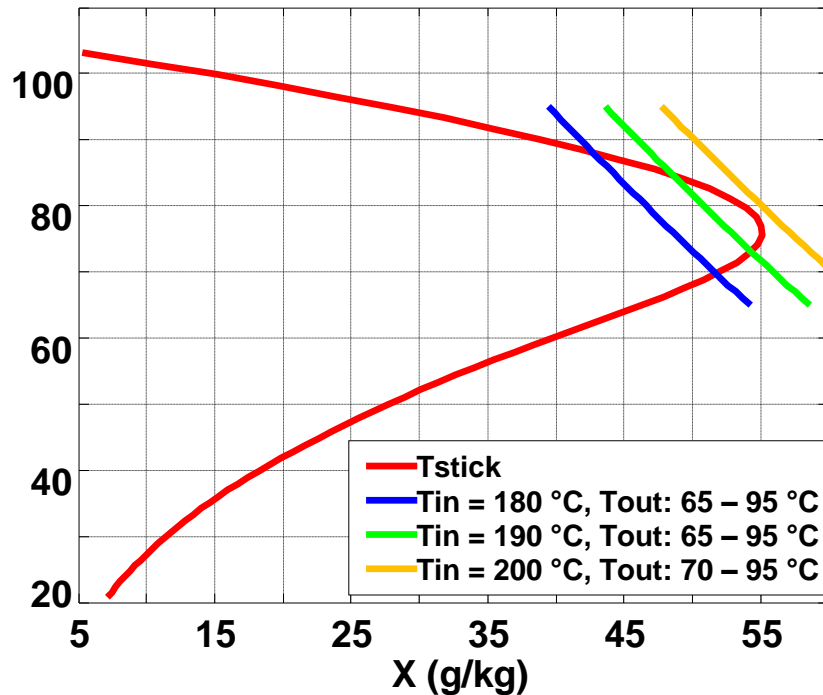
Stickiness measurement

- Powder stickiness as a function of air outlet temperature and air moisture content for powder in equilibrium with dryer air
- Measurements: Static (climate chamber, NIZO applied) or dynamic (e.g. fluid bed)



DrySpec3 example calculation

maximizing capacity at constant processing conditions

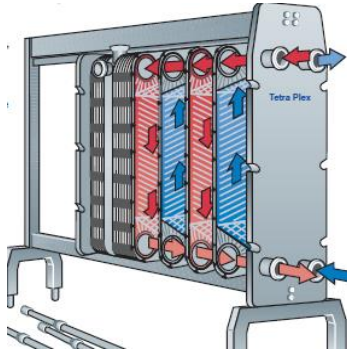


The screenshot shows the NIZO-Premia simulator interface for a spray dryer. Key parameters and results are as follows:

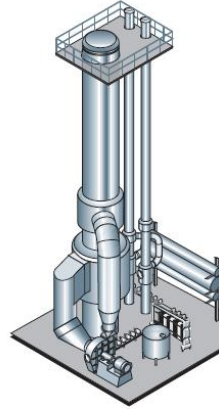
Parameter	Value
Drying model	1. SD: Near eq. model; no IFB
Calc. option	1. Input T air out
AIR DRYING CHAMBER IN: T (°C)	180
AIR DRYING CHAMBER IN: X (g/kg)	6
PRODUCT IN: F (kg/h)	8355.18
PRODUCT IN: T (°C)	70
PRODUCT IN: DS (%)	50
PRODUCT IN: Prot ds (%)	28
PRODUCT IN: Fat ds (%)	26
SPRAY DRYING CHAMBER: V (m3)	830
SPRAY DRYING CHAMBER: t (s)	0.0791292
SPRAY DRYING CHAMBER: Calib_ne	0.0761292
SPRAY DRYING CHAMBER: DetX	0.0791292
SPRAY DRYING CHAMBER: Calib_km1	10
SPRAY DRYING CHAMBER: Calib_km2	-22.64
SPRAY DRYING CHAMBER: Hex (kJ/kg)	4.5204699
SPRAY DRYING CHAMBER: Mp1 (%)	2.71
SPRAY DRYING CHAMBER: Mp1 eq (%)	1.45
SPRAY DRYING CHAMBER: DTst (K)	-6.3
SPRAY DRYING CHAMBER: CPU (ms)	0
AIR OUT: T (°C)	80
AIR OUT: X (g/kg)	46.6
AIR OUT: RH (%)	14.7
PRODUCT OUT: T (°C)	80.0
PRODUCT OUT: Mp (%)	2.71
PRODUCT OUT: Mp eq (%)	1.45
PRODUCT OUT: Rhob (kg/m3)	1457
PRODUCT OUT: Rhob (kg/m3)	513
PRODUCT OUT: Isi (ml)	0.75
PRODUCT OUT: Dp (um)	250
Evaporation and energy: Evap 1 (kg/h)	4061.1
Evaporation and energy: Evap ifb (kg/h)	
Evaporation and energy: Evap tot (kg/h)	4061.1
Evaporation and energy: Ew (kJ/kg)	4260
Evaporation and energy: Ep (kJ/kg)	4141

Efficient powder production: energy consumption, investment and labor

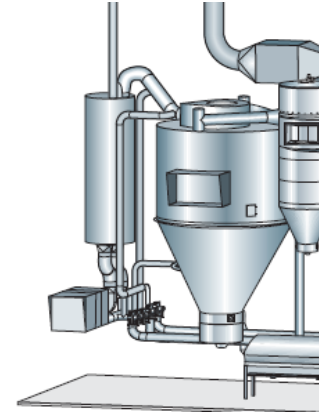
Heat treatment



Evaporation

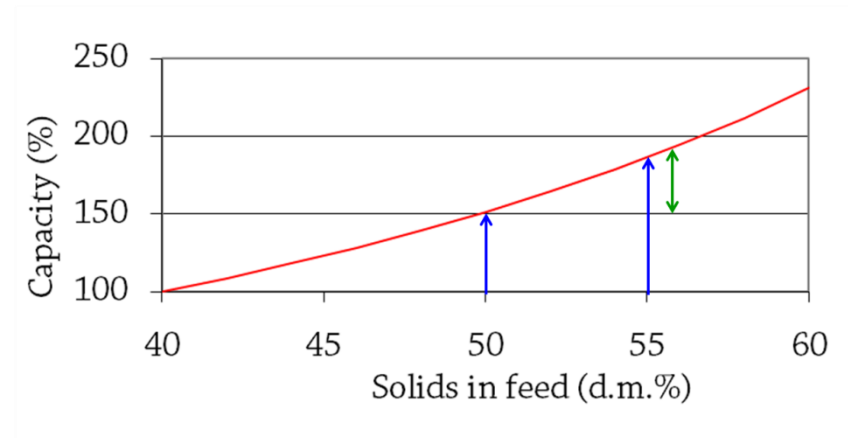
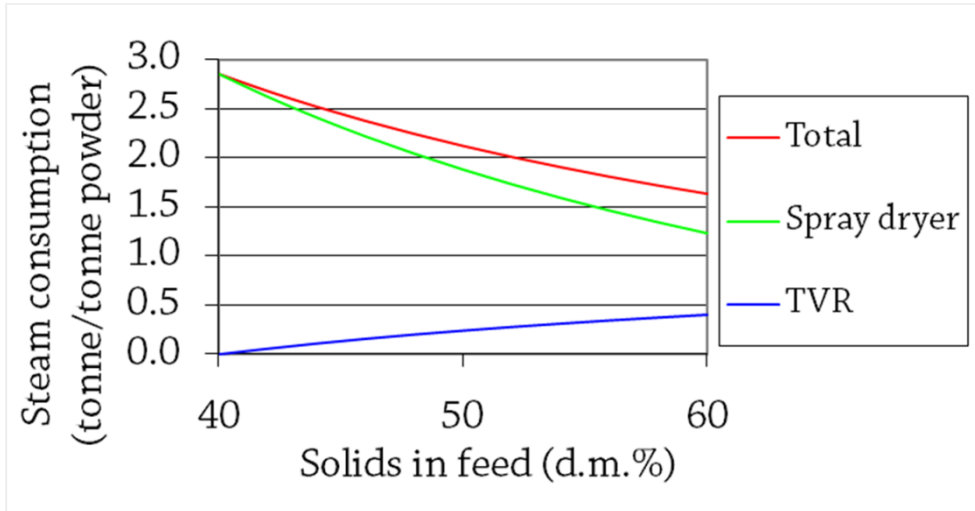


Drying



- Total energy consumption per ton powder: mainly evaporation and drying
- Largest energy consumption per ton water evaporation by far: drying
- Capex and Opex costs: Drying is the most costly by far

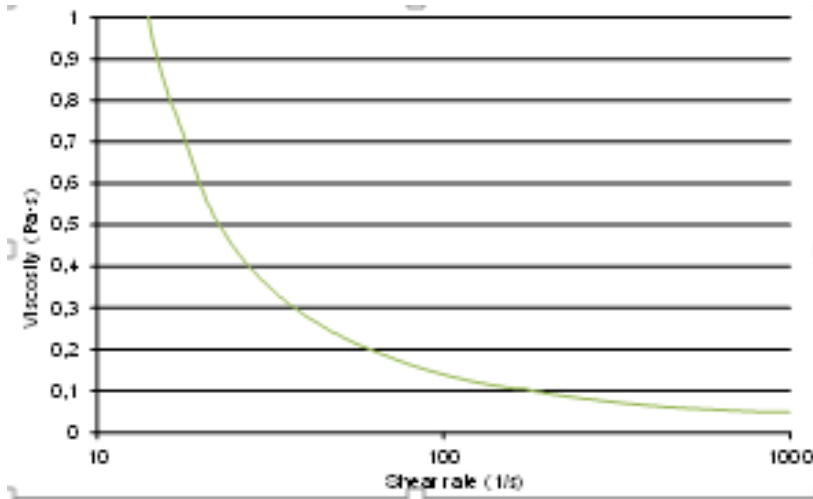
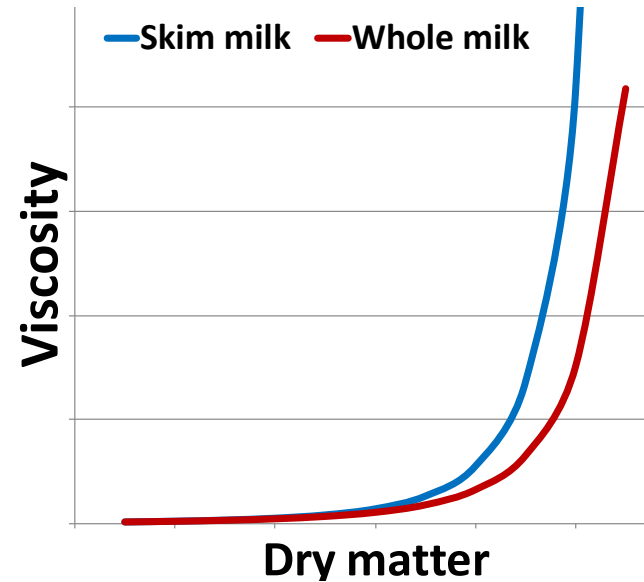
Steam consumption and capacity vs. dry matter content of feed



- Water evaporation capacity determines spray dryer output
- Power consumption by MVR to 40% d.m. not included
- Increasing the dry matter content of the output of the evaporator is usually done in a TVR system, as it can apply larger temperature gradients than MVR systems
- Steam consumption TVR applied = 0.5 tonne steam/tonne water evaporation
- Steam consumption spray dryer = 2 tonnes steam/tonne water evaporation

Viscosity of importance

- Viscosity depends on:
 - Shear rate
 - Composition
 - Temperature
 - Dry solids content
 - Protein denaturation
 - Age of product



- Viscosity decreases with shear rate
- In an evaporator
 - Low shear rates: **distribution plates**
 - High shear rates: tubes, inside pumps, atomizer

Viscosity of milk

Milk viscosity is determined by:

- Viscosity of the solvent (i.e., milk serum)
- Volume fraction of suspended particles
- Interactions between suspended particles

Volume fractions (ϕ) (at 25°C):

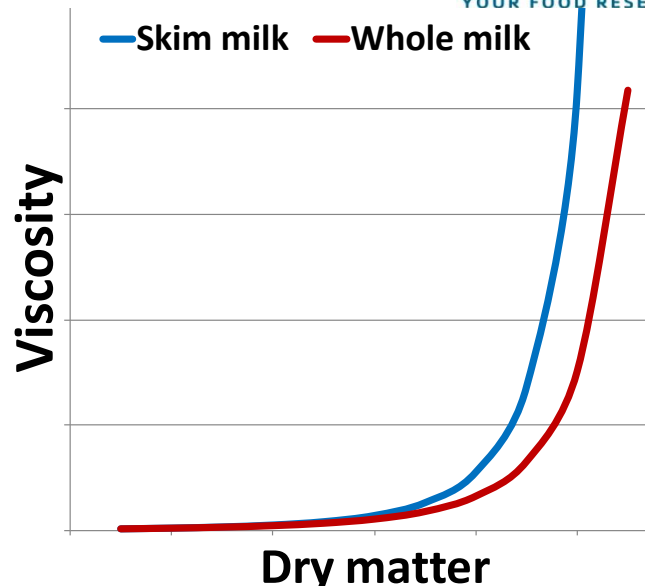
- Milk fat: 1.1 mL g⁻¹
- Casein micelles: 3.7 mL g⁻¹
- Whey proteins
 - Undenatured: 1.2 mL g⁻¹
 - Denatured: ~3 mL g⁻¹

$$\text{Total volume fraction} = \sum(c_i \times \phi_i)$$

Milk viscosity:

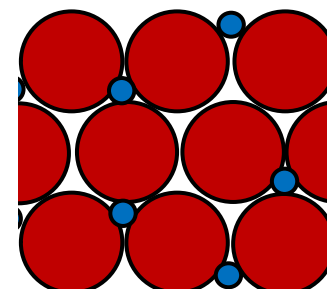
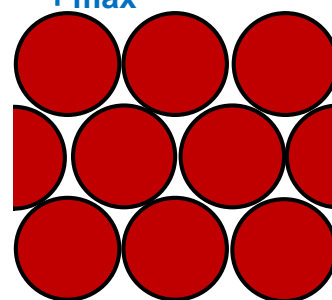
$$\eta_{concentrat} = \eta_{serum} \left(1 + \frac{1.25\phi}{1 - \phi / \phi_{max}} \right)^2$$

where ϕ_{max} = maximum volume fraction



Monodisperse spheres

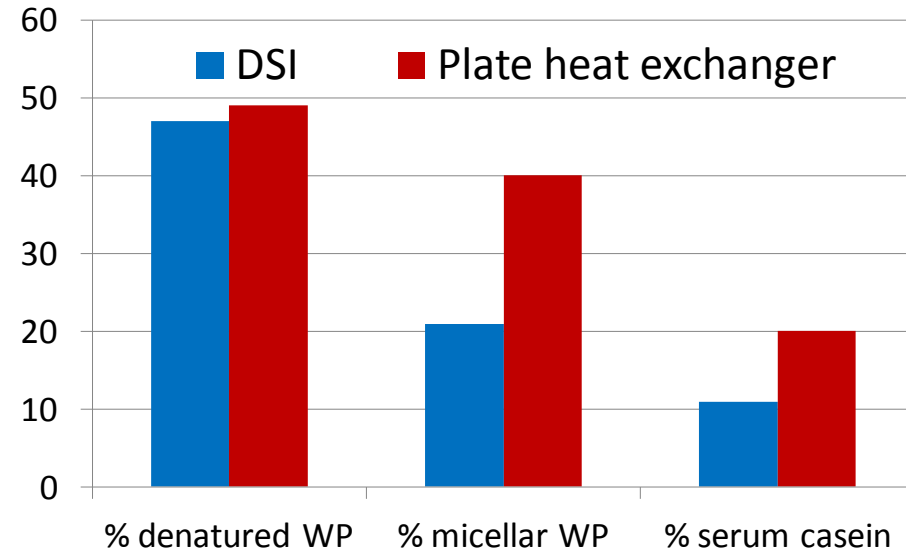
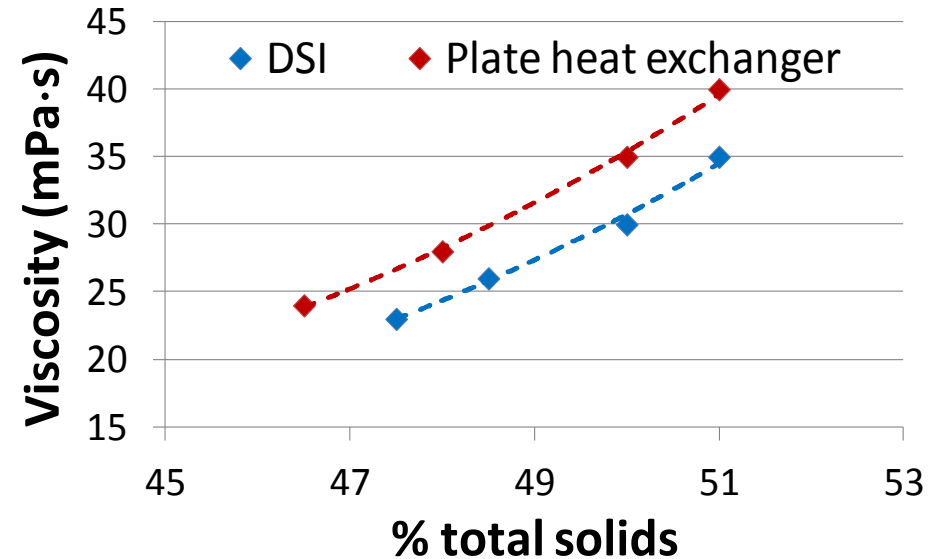
$\phi_{max} = \sim 0.74$



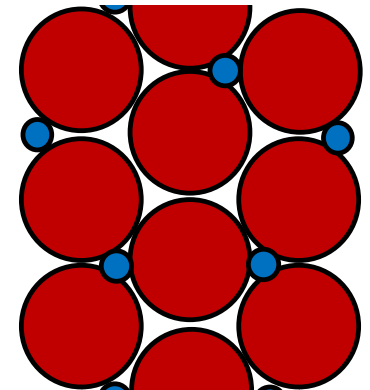
$\phi_{max} = \sim 0.80$

Polydisperse spheres

Effect of heating conditions

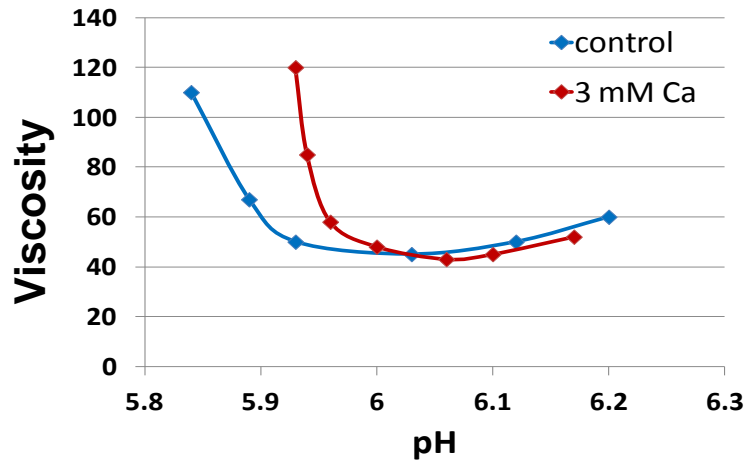


- Plate heat exchanger yields higher viscosity of milk concentrate at similar level of whey protein denaturation
- In milk heated with plate heat exchanger denatured whey protein largely on micellar surface → more voluminous micelles
- Non-micellar whey proteins can pack between micelles

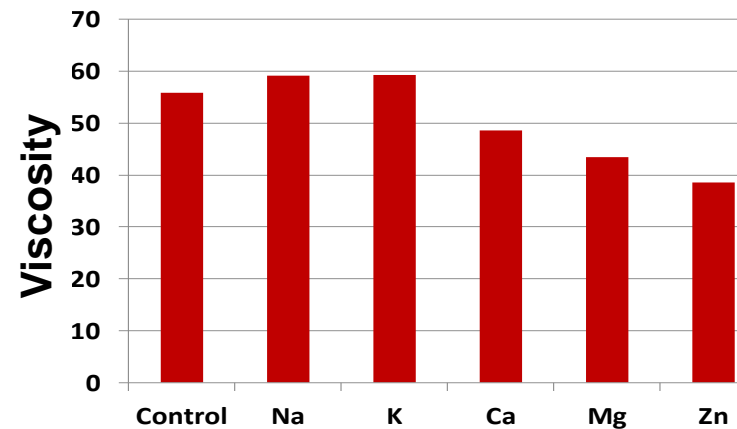


Mineral addition to milk prior to pre-heating and concentration

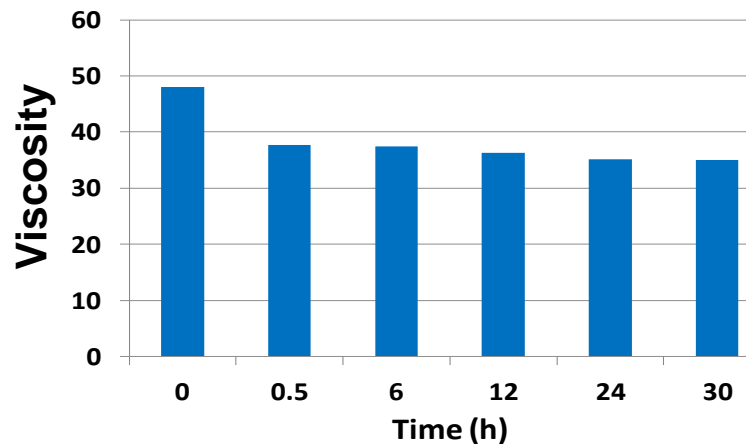
pH and calcium addition



Cation type



Time of calcium addition



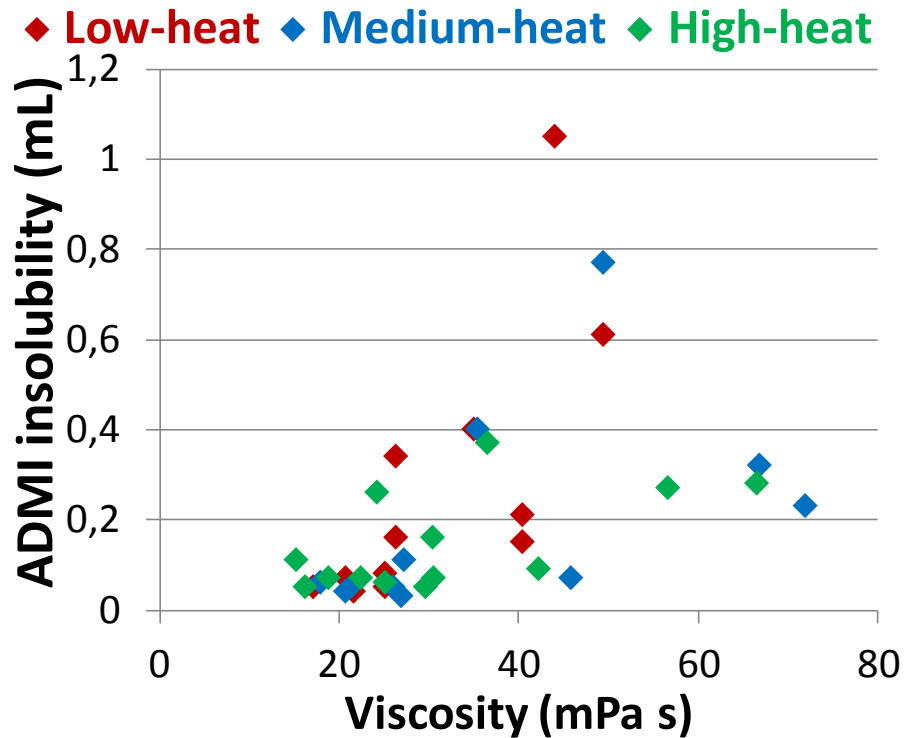
pH and divalent mineral can be used to minimize concentrate viscosity

Reduced viscosity because of:

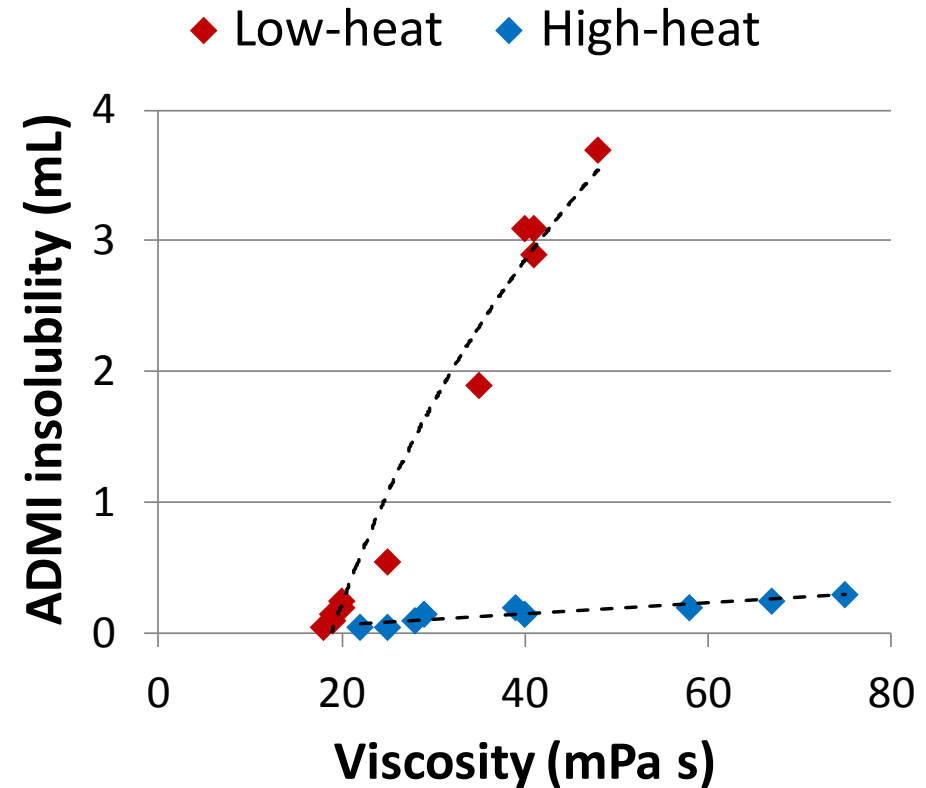
- Reduced micellar solvation
- Reduced non-micellar casein
- Association of denatured whey protein with casein micelles

Powder properties - insolubility

Constant drying conditions (180 / 95°C)



Constant moisture content (4%)

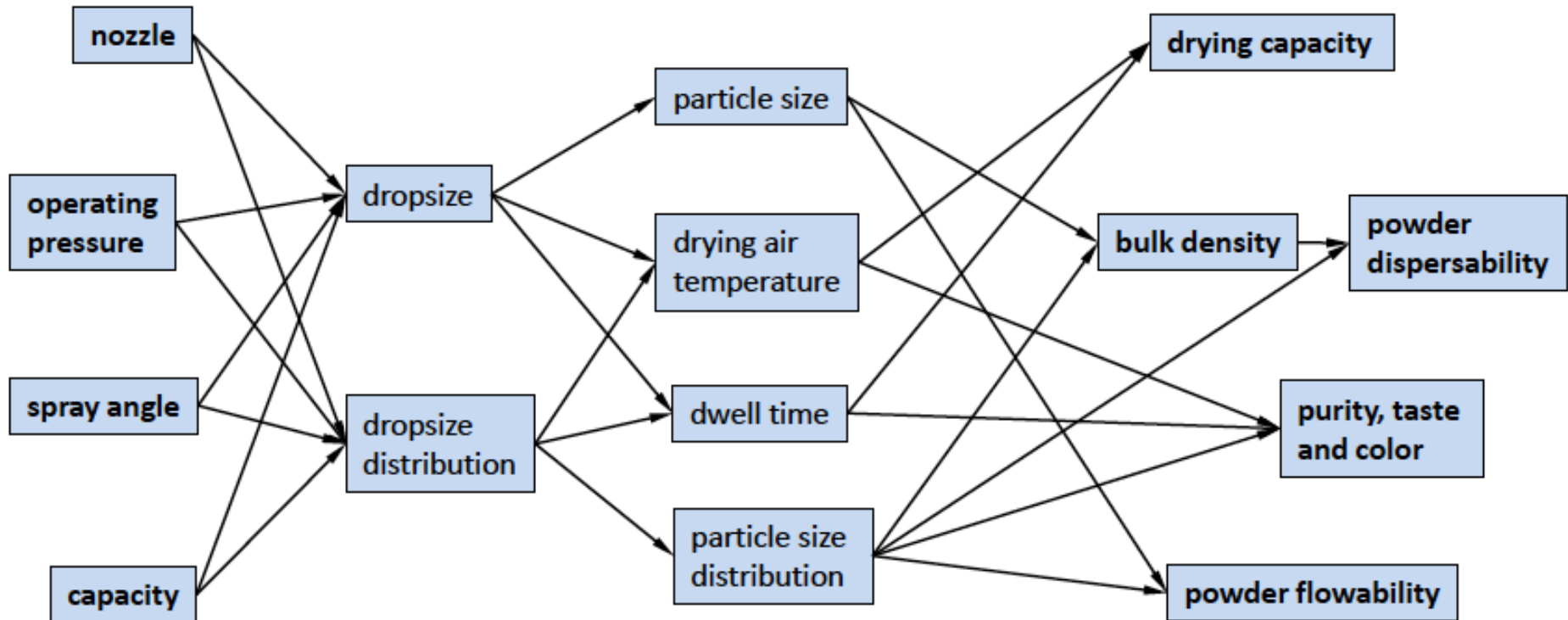


Need to adjust drying conditions and atomization

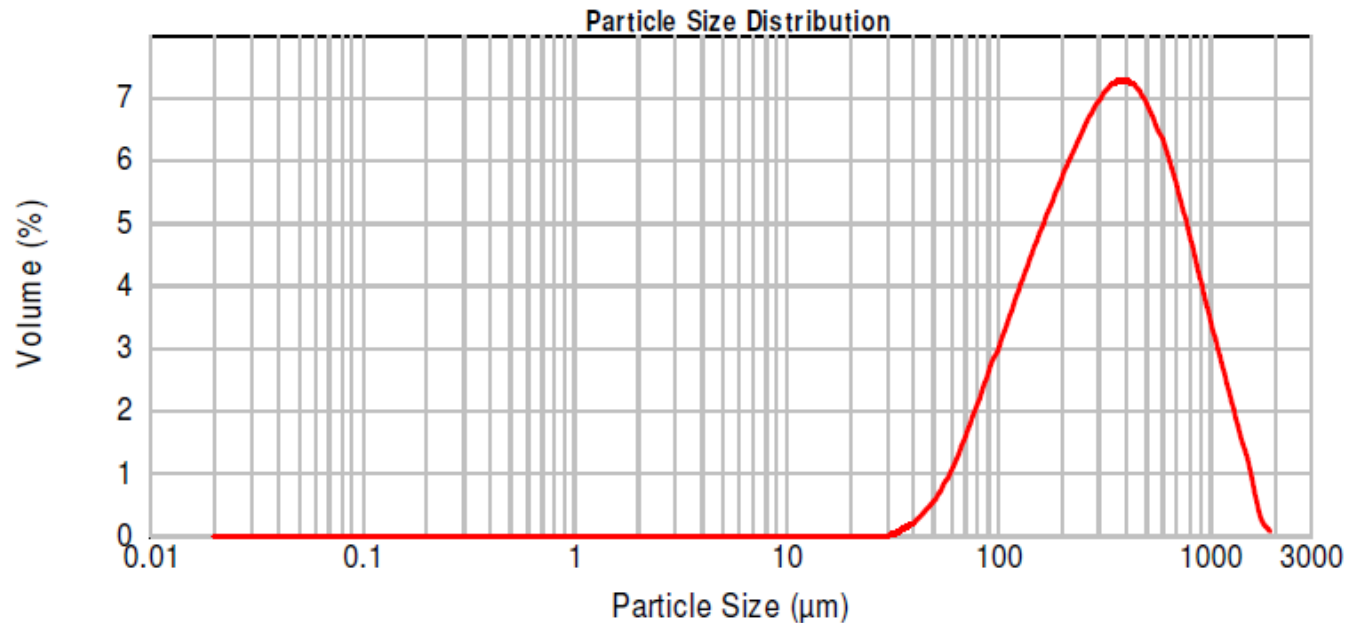
Increasing dry matter of feed is tricky

- Where to start?
 - Substantial investments will be needed upfront:
 - Evaporator adaptations to achieve high dry matter output
 - Higher capacity high pressure pump
 - Unlimited number of orifice – whirl chambers available
- What could happen?
 - Product properties may change:
 - Solubility may degrade
 - Bulk density can alter
 - Process performance may decrease:
 - Fouling of spray dryer could seriously interfere with daily production needs
 - Substantial product quantities can be at stake
- ➔ Success is not granted
- ➔ Need for a structured approach to ensure the dryer will perform at high dry matter feed

Atomization is key



Particle size distribution



- Small particles (D10)
 - Fines, blocking of cyclone, product quality
- Large particles (D90)
 - Long drying time, fouling!

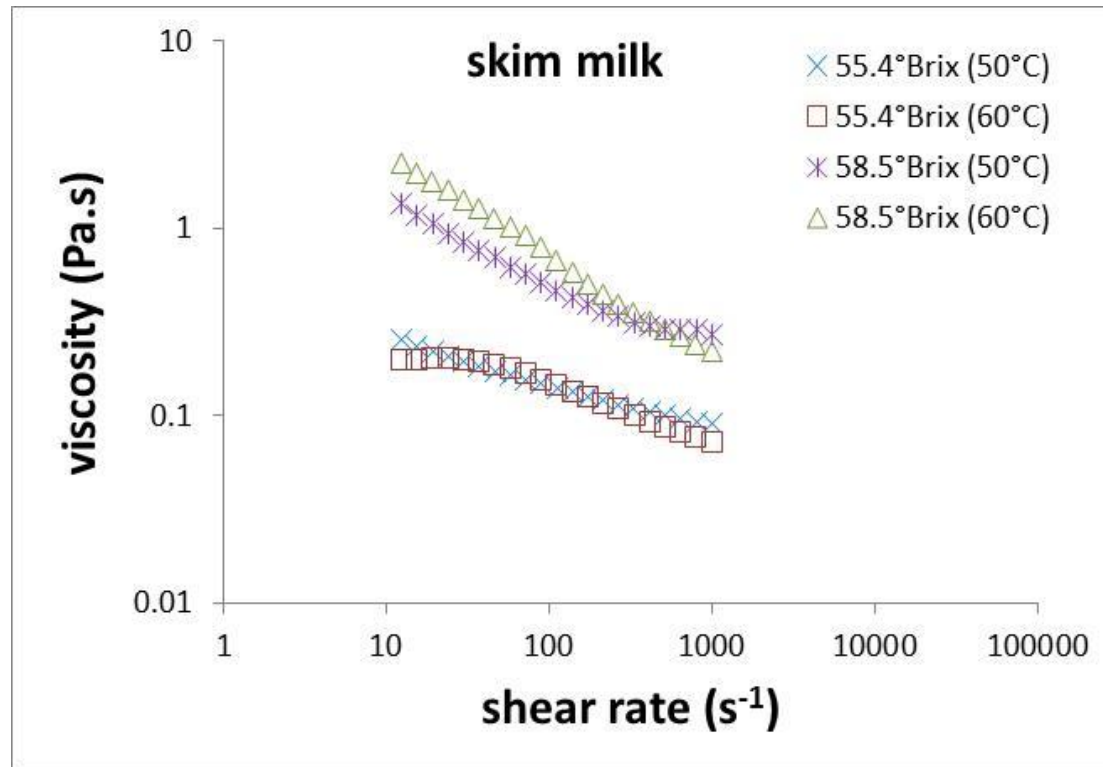
Four step approach toward high dry matter feed of spray dryers

1. Evaporate feed to reference and high dry matter content
 - a) Measure viscosity at 50 and 60°C as function of shear rate
 - b) Measure viscosity in-line at feed temperature
 - c) Extrapolate data to estimate viscosity at nozzle
2. Measure droplet size distribution spraying a Newtonian model liquid with inside the spray dryer as function of:
 - a) Viscosity
 - b) Process conditions
 - c) Nozzle type/configuration:
 - I. Pilot plant capacity
 - II. Full scale capacity
3. Select appropriate nozzle and process conditions to spray dry product at reference and high dry matter feed in pilot plant dryer:
 - a) Verify maximum droplet size using SEM
 - b) Compare powder characteristics
4. Apply selected full scale nozzles in production site plant

Testing facility used at NIZO

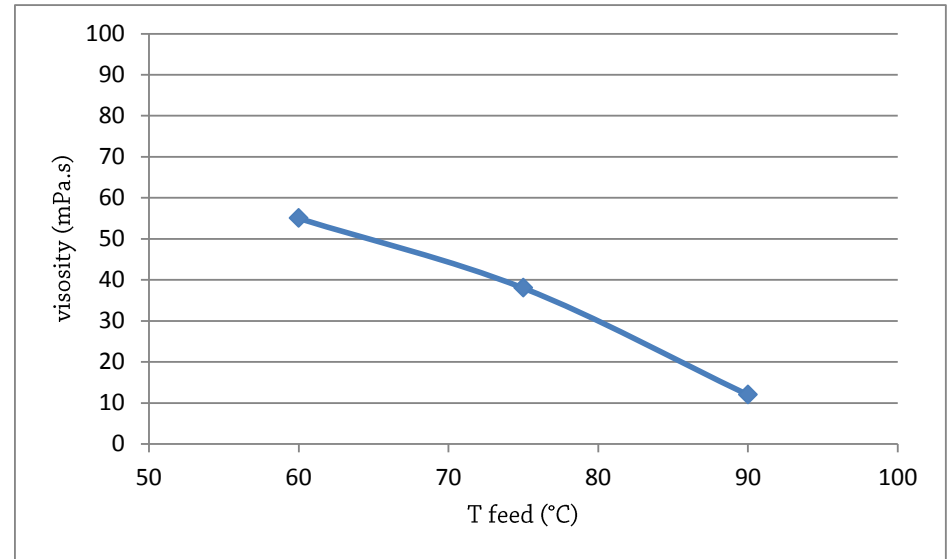
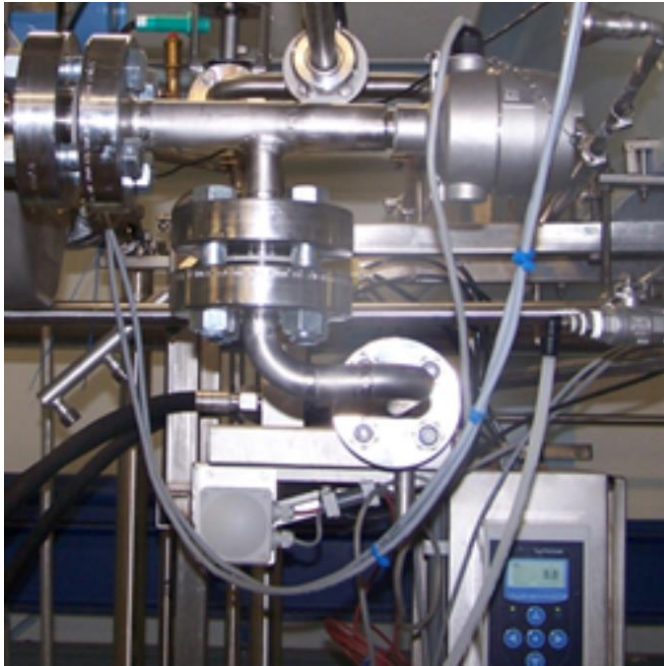


Measure concentrate viscosity as function of shear rate at 50 and 60°C



- Skim milk concentrate shows shear-thinning behavior
- Shear rate at nozzle is $\sim 100,000 \text{ s}^{-1}$
- Extrapolate lab data range to shear rate at the nozzle

Measure viscosity in-line just prior to atomization as function of temperature



- In-line viscometer measures at a shear rate of $\sim 1/1000$ s
- Combine data lab viscometer and in-line viscometer
- Extrapolation using temperature dependency and shear rate dependency to estimate viscosity at nozzle

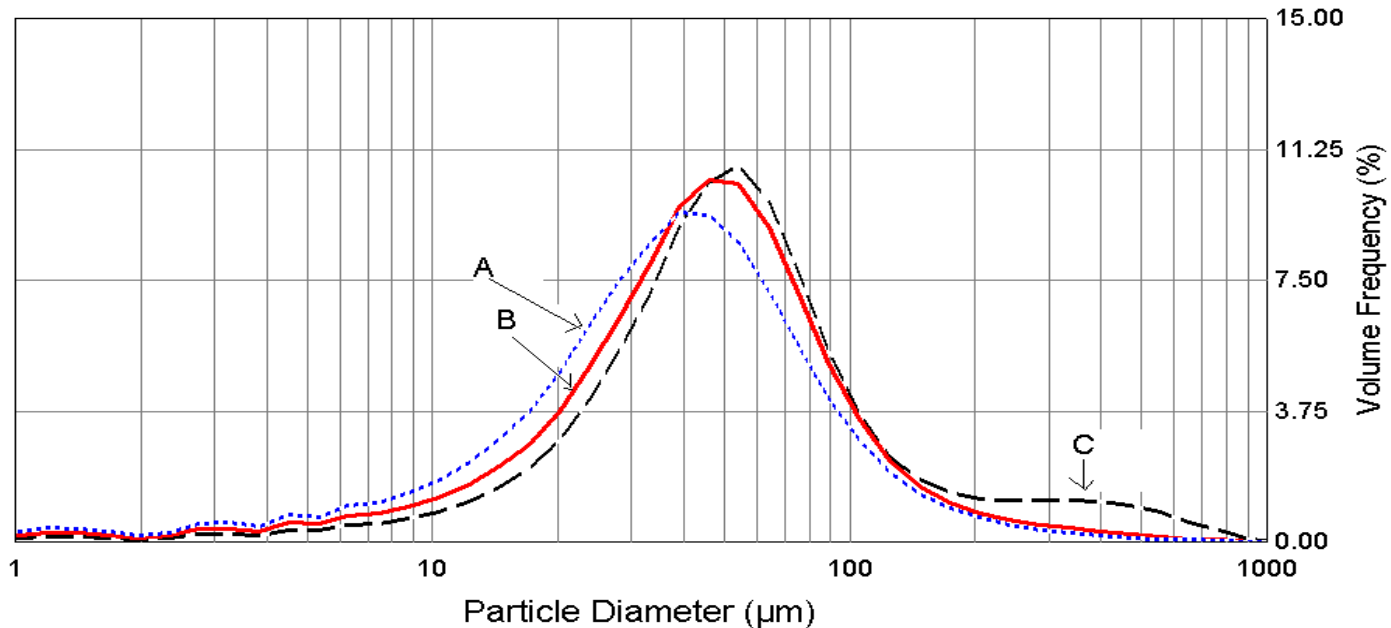
Droplet size distribution measurement inside the spray dryer



- Viscosity of glycerol by temperature adjustment/water addition
- Nozzle pressure/Feed rate
- Nozzle: swirl chamber/core + orifice combination



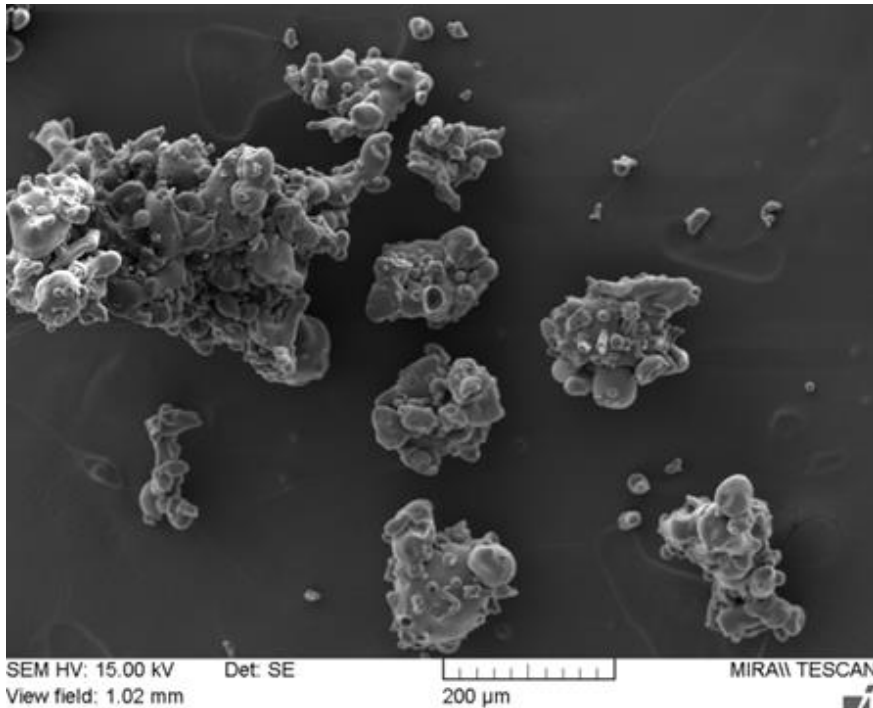
Measured particle size distribution of three nozzle configurations



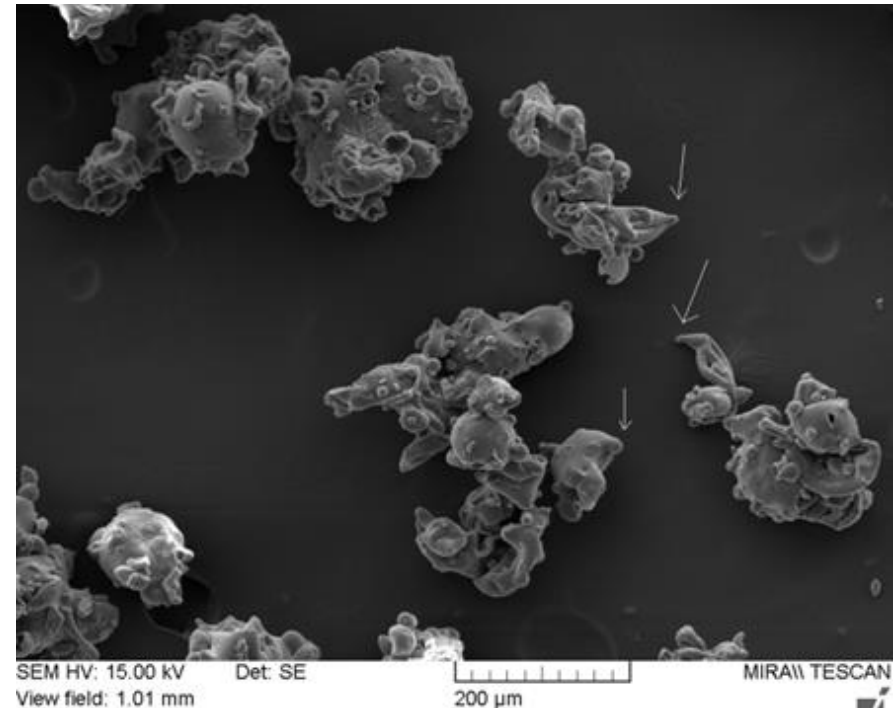
- All curves are made using glycerol at 30 mPa.s using different nozzles:
 - C gives a small quantity of undesired large droplets
 - B is preferred over A as number of fines is less

Observe spray drying of product at selected conditions using SEM

Safe processing



Getting to the limit

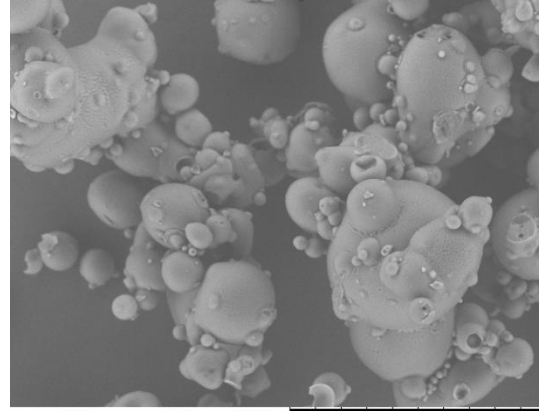


POWDER PARTICLE SIZE, SHAPE AND MORPHOLOGY

SEM pictures – morphology and size

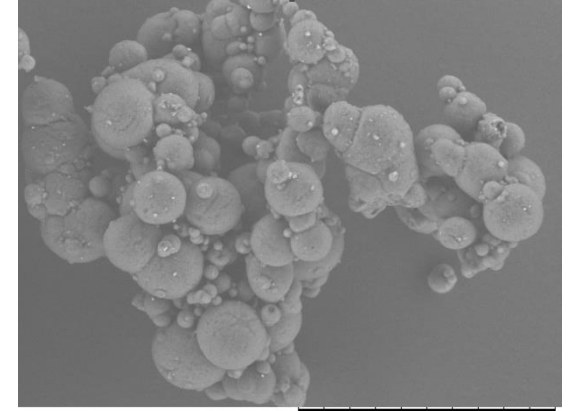


Buttermilk powder



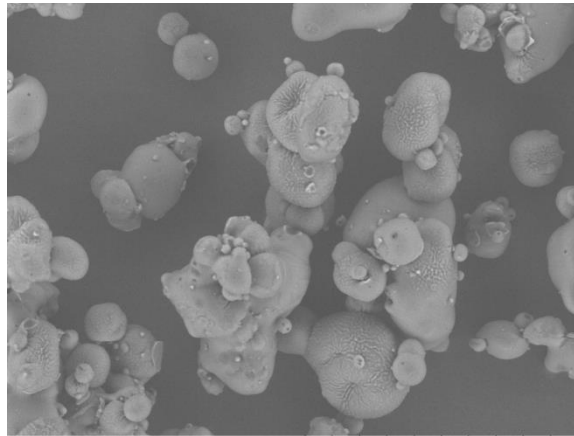
buttemp0003 2009/09/18 L D1.9 200 um

Cream powder

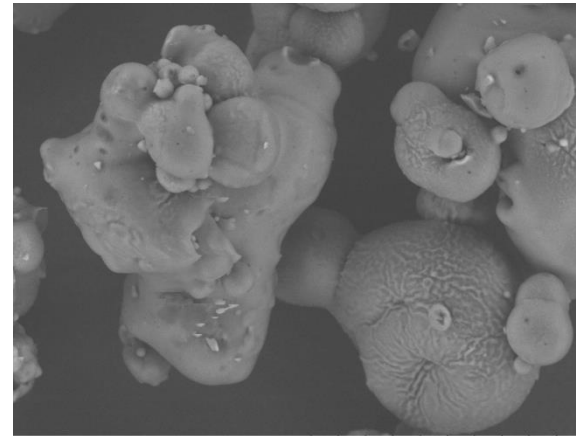


CREAM0006 2009/09/18 L D2.0 200 um

SMP



SMP0002 2009/09/18 L D2.0 200 um



SMP0005 2009/09/18 L D2.0 100 um

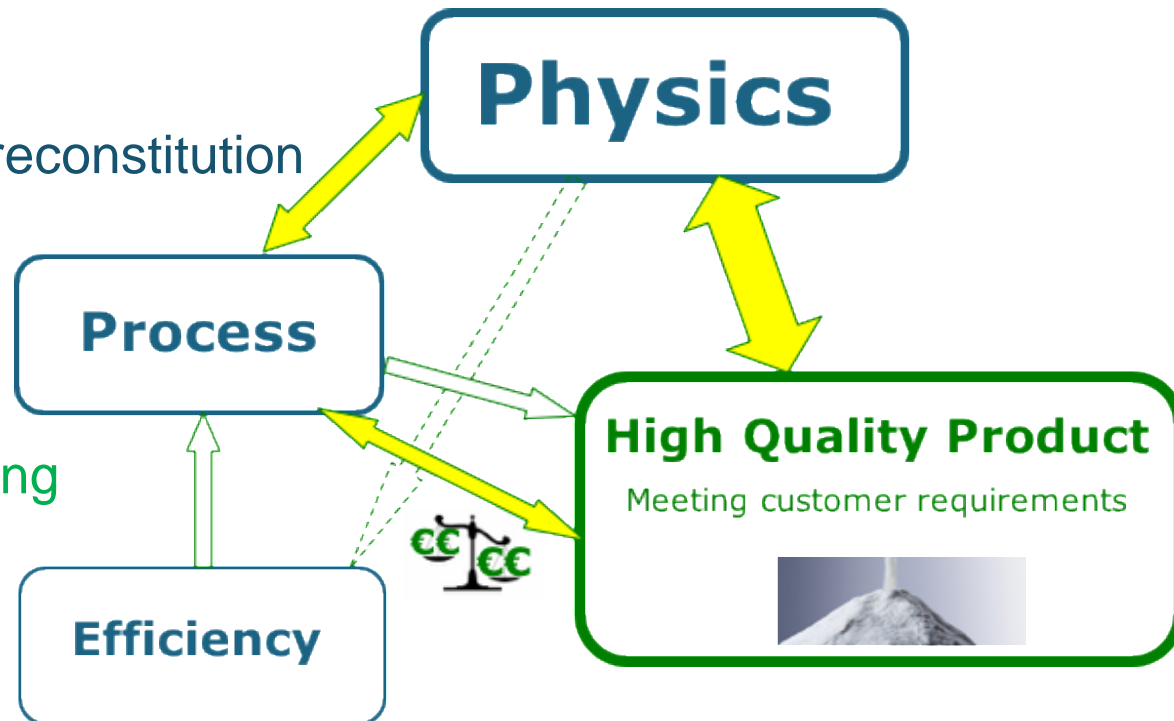
The ideal powder

For the user:

- Delivers all required nutrition
- Excellent physical appearance of powder
- Easy to handle
- Easy to dissolve
- No physical defects after reconstitution
- No off-flavor or off-taste

For the manufacturer:

- High capacity and yield
- No issues during processing
- Long shelf-life



What is needed to be on top of the game?

Know everything about **Product Characteristics**

- **Physics**, phase diagrams, sorption, T_g
- Sensitive ingredients
- Morphology in relation to bulk properties
 - Density, flow, reconstitution, etc.

Know everything about **Drying Technology**

- Unit Operations, process steering & control, Modeling processes
- Heat / Mass balances, Mollier Diagram
- Various drying techniques
- Air de-humidification

And being able to **combine and apply** this knowledge

Thank you for your attention

