Properties of lactose as determinants of crystallization behaviour and of industrial applications

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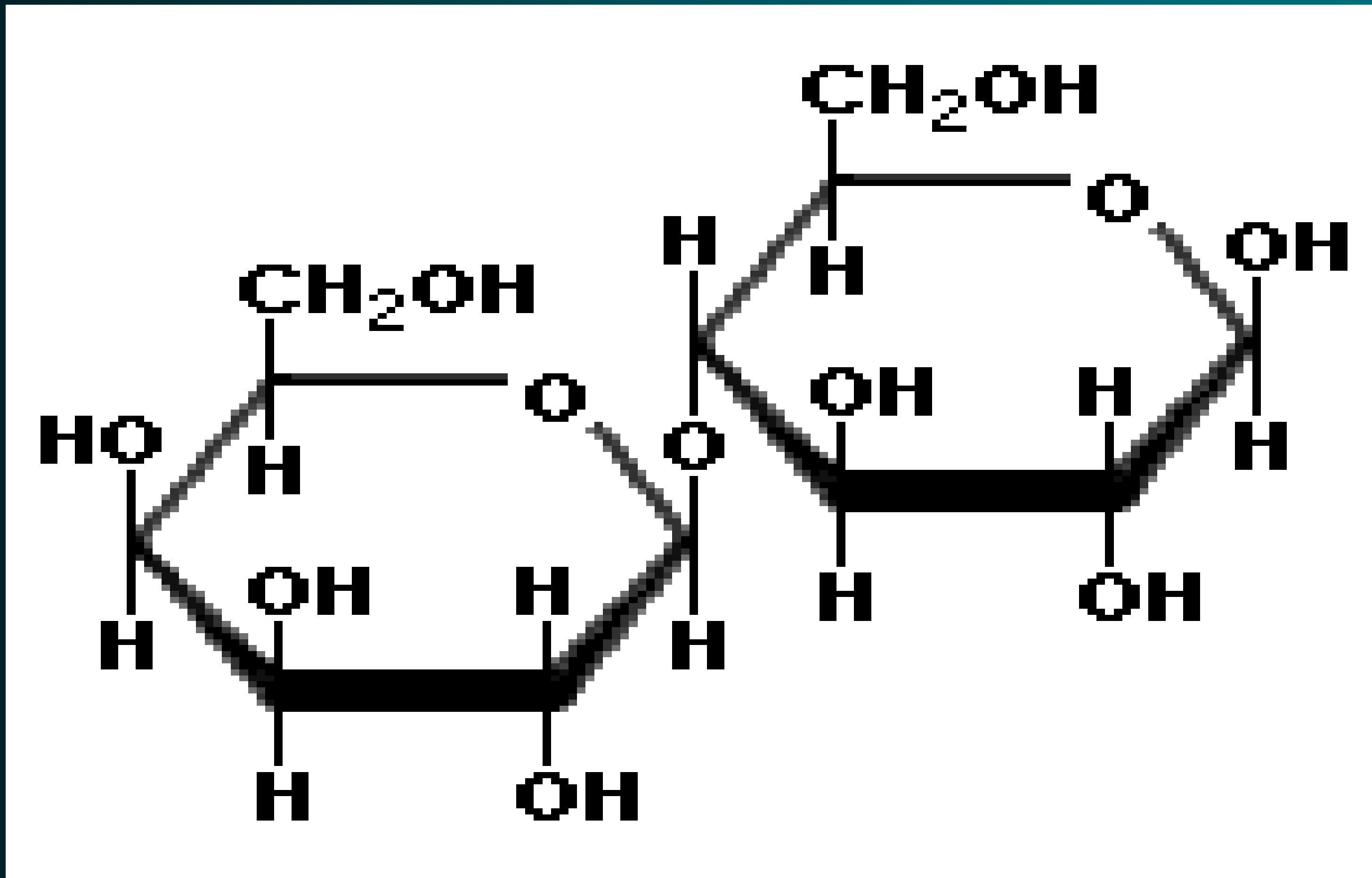
Lactose

- Milk of (almost) all mammals
- Primary source of energy for the neonate
- Highest concentration (7%) in human milk
- Principal component of cow's milk (4.5 - 5%; 53% of non-fat solids)
- Cheapest reducing CHO on the market

Concentration of lactose in milk of different mammals

Species	Lactose content (%)	H_2O content (%)
Human	7.1	87.1
Cow	4.6	87.3
Buffalo	4.8	82.8
Goat	4.3	86.7
Sheep	4.8	82.0

Schematic representation of the lactose disaccharide molecule



Important basic properties of lactose

- Disaccharide (glu-gal)
- Two stereoisomers (α- and β) > mutarotation
- Reducing sugar >>> browning reaction
- Low sweetness (20% of sucrose at 5% conc.)
- Low solubility (18% w/w at 20°C)

  > low osmotic pressure

  > slow crystallization

Forms of lactose

- α - lactose, β – lactose
- In solution the ratio of α : β is about 1 : 2
- Effect of temperature and mutarotation on
  - > solubility
  - > sweetness
  - > crystallization behaviour
- In crystalline state
  - > α – lactose contains one H_2O molecule
  - > α – lactose anhydride (heating above 130^oC)
  - > β – lactose (above 95^oC) contains no H_2O

Properties of lactose affecting the dairy industry

- Fermentable by lactic acid bacteria
- Crystallizes in highly concentrated dairy foods (sweetened condensed milk, ice cream, whey cheese mysost)
- Low sweetness – unsuitable as a sweetening agent, can be improved by hydrolysis
- Lactose malabsorption limits consumption of dairy foods by lactose intolerant consumers
- Fermentable by aquatic bacteria - high BOD of whey
- Unique crystallization behaviour

Focus of this presentation

- Crystallization phenomena
  - Model systems – experimental methods
  - Solubility
  - Real life dairy foods
- Sweetness phenomena
  - sensory impact in dairy foods
  - use for protein standardization
- Lactose hydrolysis
- Lactose derivatives and pharma-lactose

Centuries of dairy science: lactose

- 1633 – Bartoletto isolated and described lactose as “essential salt without nitrogen”
- 1688 - Ettmueller isolated lactose from whey and purified by recrystallization
- 1814 – 1820 analytical work by Berzelius
- 1902 – 1942 fundamental work by Hudson
- 1936 – 2007 lifetime achievements of Prof. Andrei Georgievich KHRAMTSOV

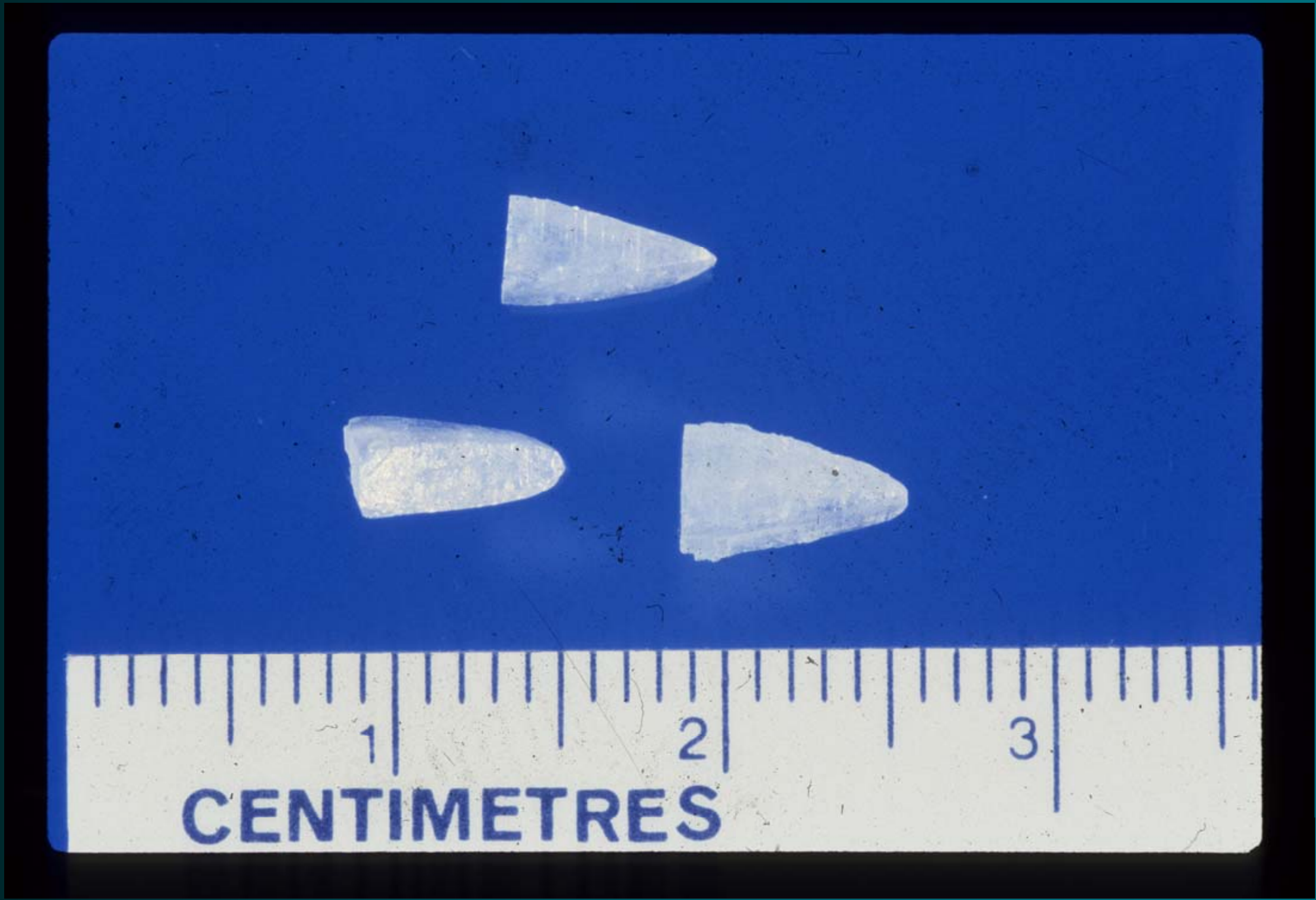
Lactose crystallization

- Supersaturated solution
- Concentrated dairy systems (condensed milk, ice-cream, whey cheese)
- Whey or whey permeate >> evaporation and cooling for production of lactose commodity
- Crystalline habit and crystal growth mechanism (α-lactose)

Single crystal method to study the crystallization phenomena

- Method described by Professor J.A. Kucharenko from Polytechnical Institute, Kiev, for study of sucrose crystallization
- Series of 12 articles published in “The Planter and Sugar Manufacturer” (New Orleans), from May 12 to July 28, 1928 (volumes 80 and 81)
- Crystallization velocity, density, effects of impurities, solubility, supersaturation

Single crystal method adapted to study lactose crystallization



Lactose crystal growth: single crystal experiments

- Van Kreveld (1966 – 1969): crystals grow from the apex of a pyramid down
- Jelen (1971 – 74): effect of supersaturation and growth promotion by mineral impurities
- Visser (1980 – 1983): crystal growth retarders, structure of lactose crystal
- Jelen (1996) – crystallization velocity with assumption of bottom plane growth

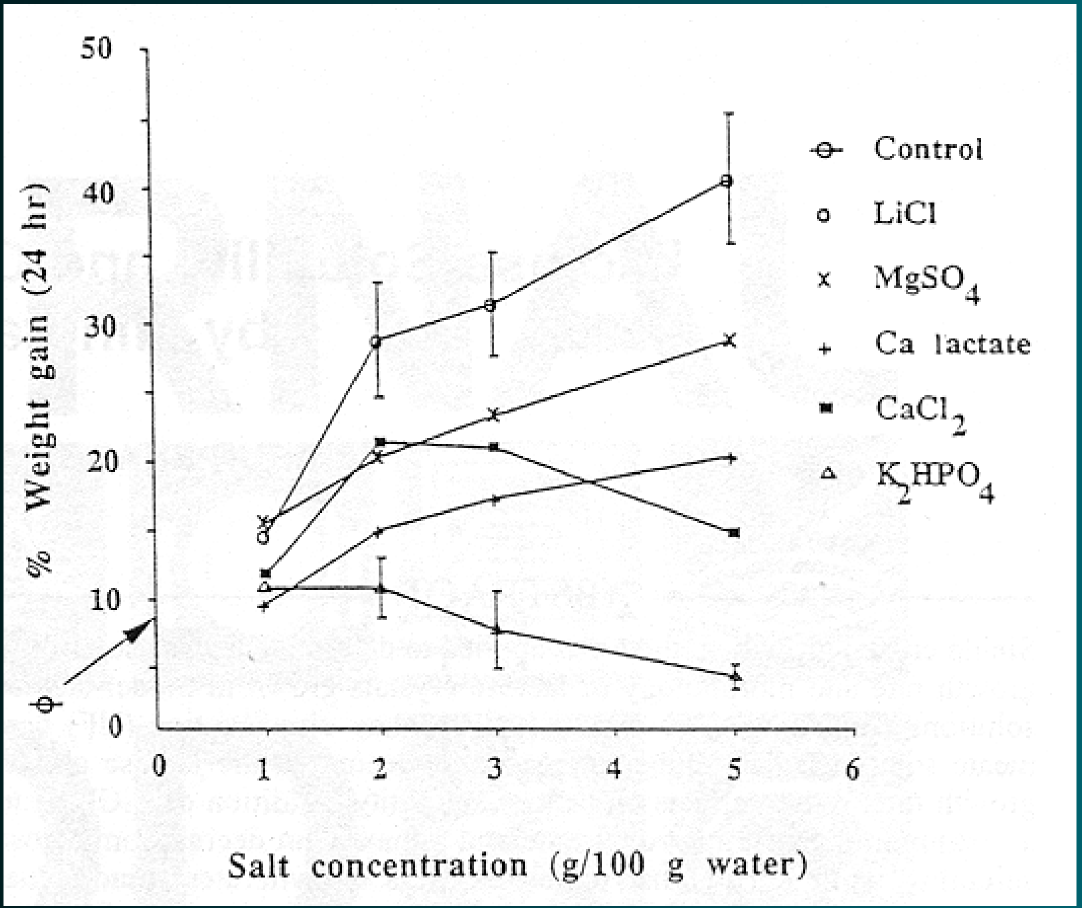
Lactose solubility

Temperature °C	Concentration of saturated solution (g / 100 g H_2O)
30 °C	24.0
50 °C	44.0
70 °C	77.8
80 °C	98.9

Effect of minerals on lactose solubility at 30°C

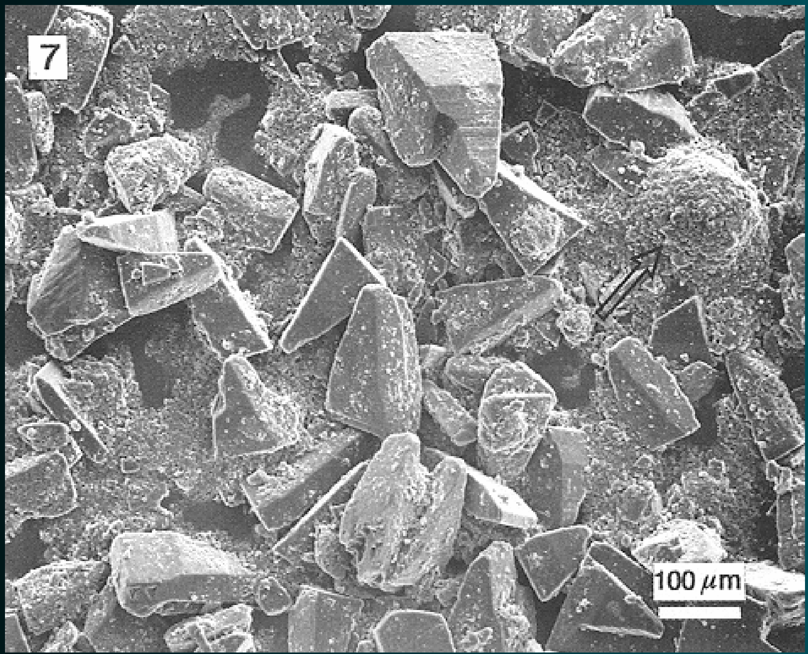
Salt added (5 g / 100 g H_2O)	Estimated solubility (g / 100 g H_2O)
Control (no salt)	24.4
Calcium chloride	24.3
Magnesium sulphate	23.0
Lithium chloride	21.9
Potassium phosphate	26.5

Lactose crystallization velocity

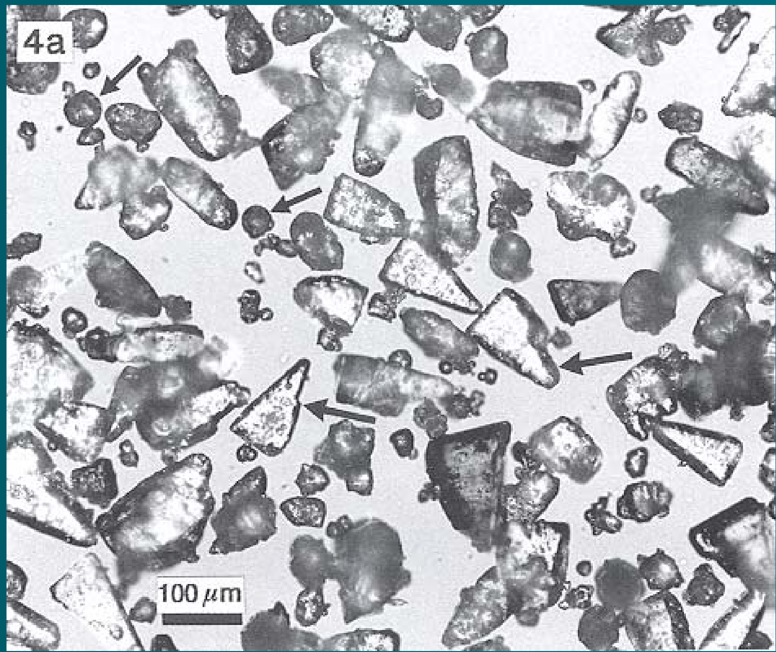


Average growth rates of lactose crystals in model lactose solutions with or without the addition of salts.

Lactose crystals under microscope



Scanning electron microscopy of spray dried demineralized permeate powder (courtesy Dr. Kalab, Ottawa).



Light microscopy of demineralized whey permeate powder (courtesy Dr. Kalab, Ottawa).

Growth rates of lactose crystals (30^{o}C, supersaturation 9 g)

Exposed face	Crystallization rate (mg m^{-2} min^{-1})
All	160.9 ± 12.1
Sides	2.5 ± 3.0
Top (truncated)	-0.6 ± 3.4
Bottom	211.8 ± 69.3

Growth rates of lactose crystals (30^{o}C, supersaturation 10g)

Assumptions	Approximate Crystallization rate (mg m^{-2} min^{-1})
Growth on all sides	80 **(Jelen 1972)**
Growth on bottom side only	350 (**Bhargava/Jelen, 1996**)
Sucrose	320 **(Smythe, 1971)**

Promoters and inhibitors of crystal development

Promoters

- Lithium chloride
- Calcium chloride (at low concentrations)
- Some phosphates
- Other electrolytes (?)

Inhibitors

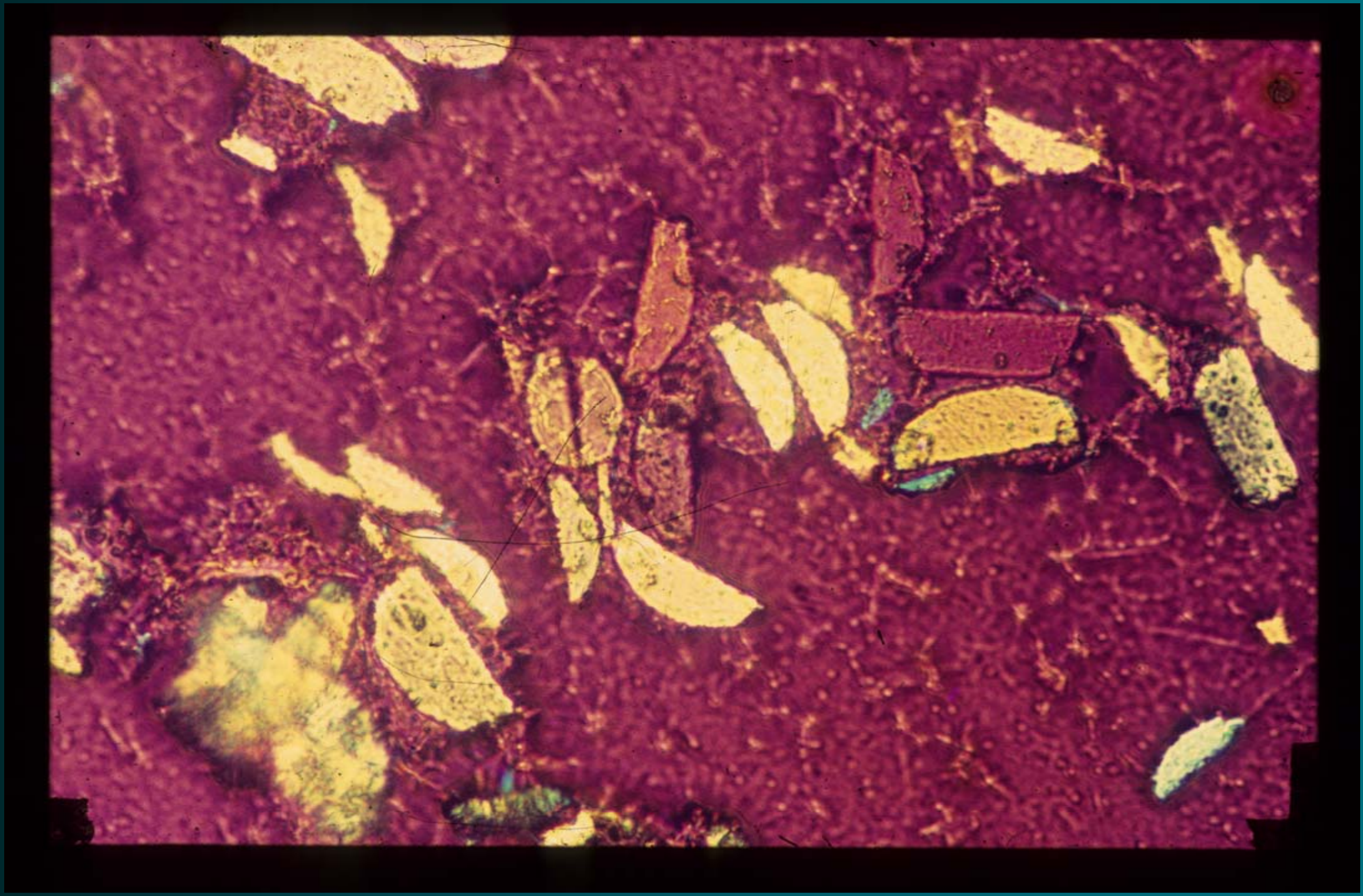
- β-lactose
- Riboflavin
- Galactose
- Gelatin
- Potassium salts

Lactose solubility and crystallization in whey, UF permeate or dairy foods

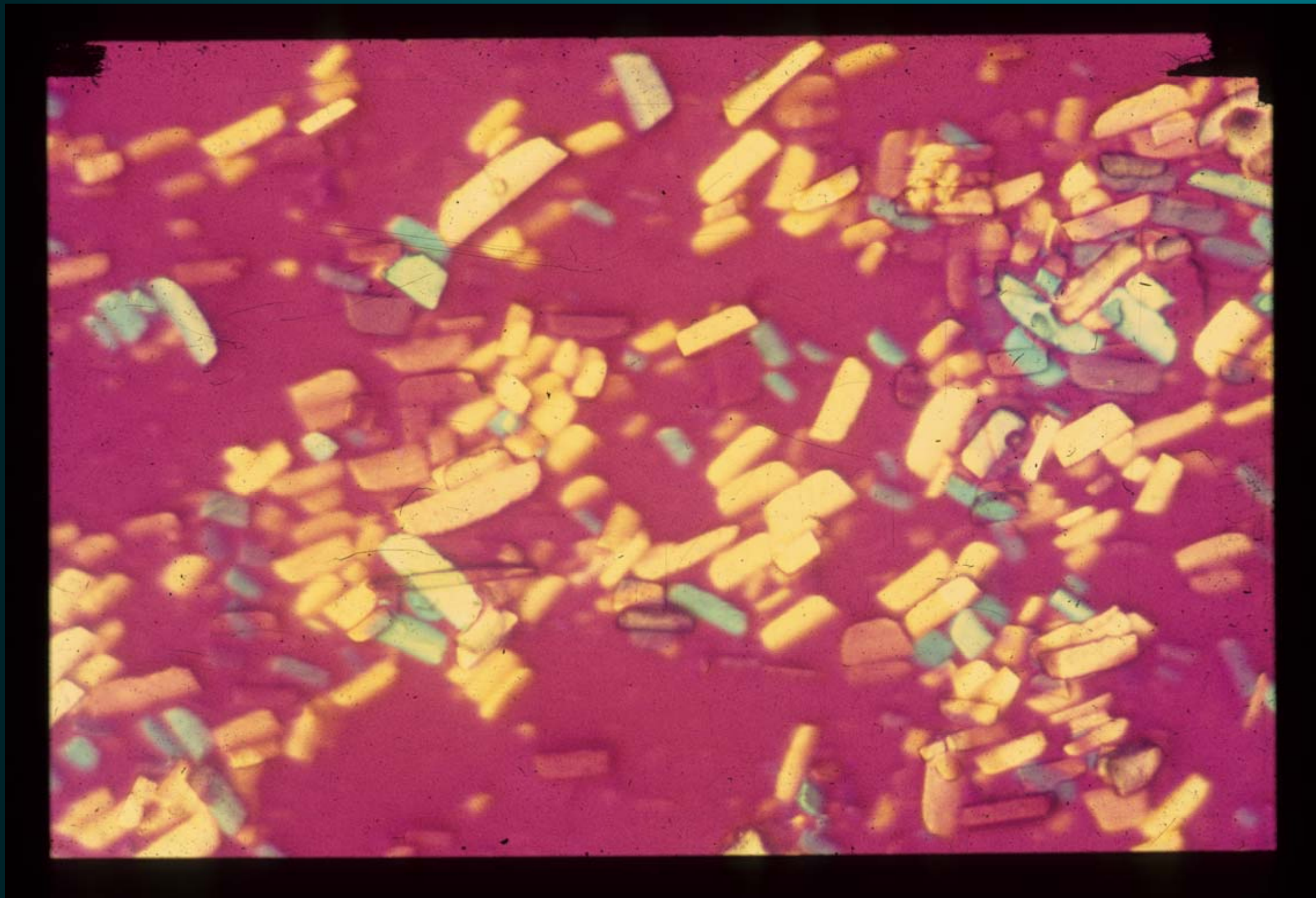
- Nucleation vs crystal growth
- Components affecting solubility and crystal growth
- Very high supersaturation – nucleation favored
- Ice cream, frozen desserts, Sw. Cond. Milk
- Norwegian whey cheese Mysost

Lactose crystals in whey cheese Mysost

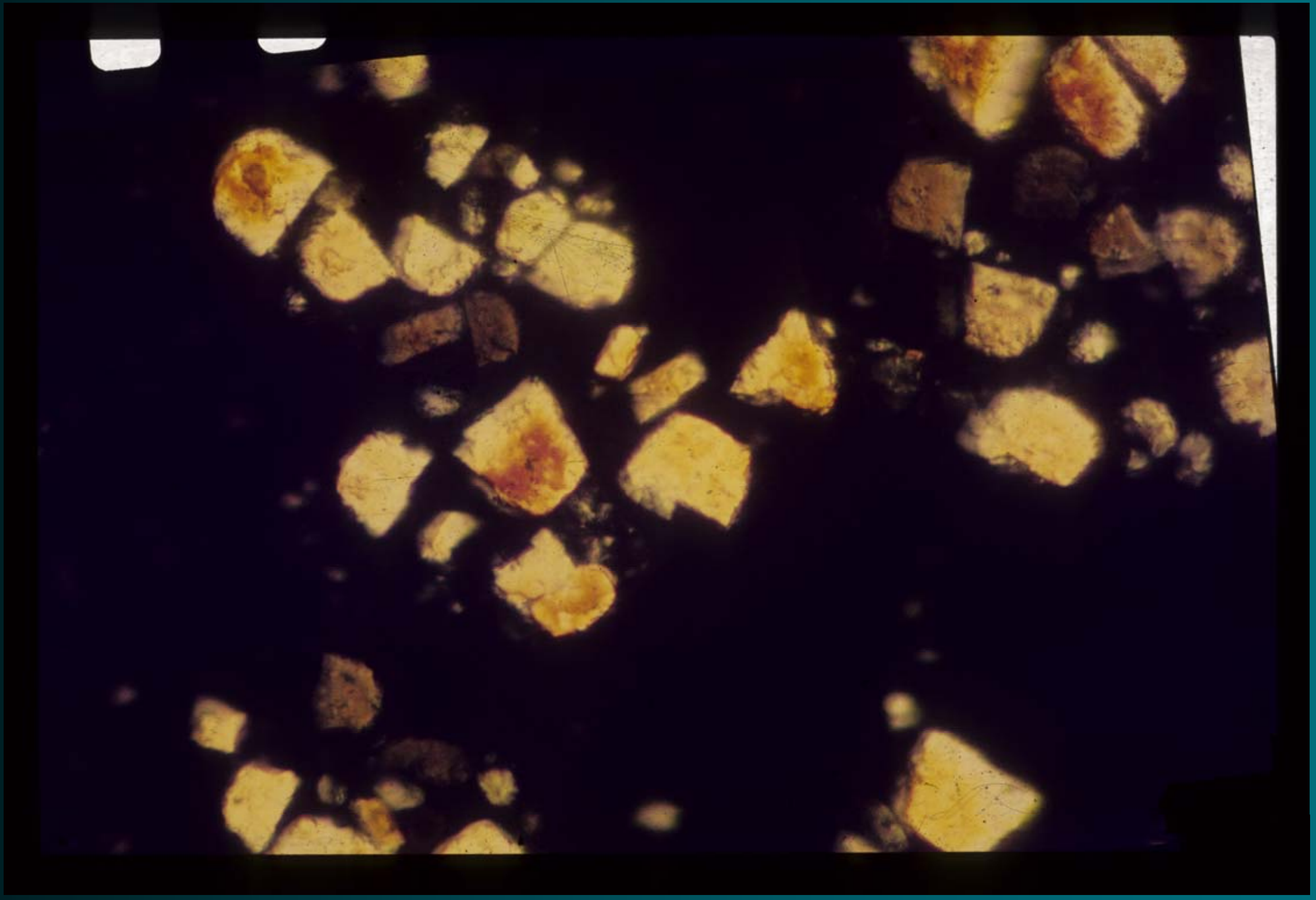
Lactose crystals in whey cheese Mysost



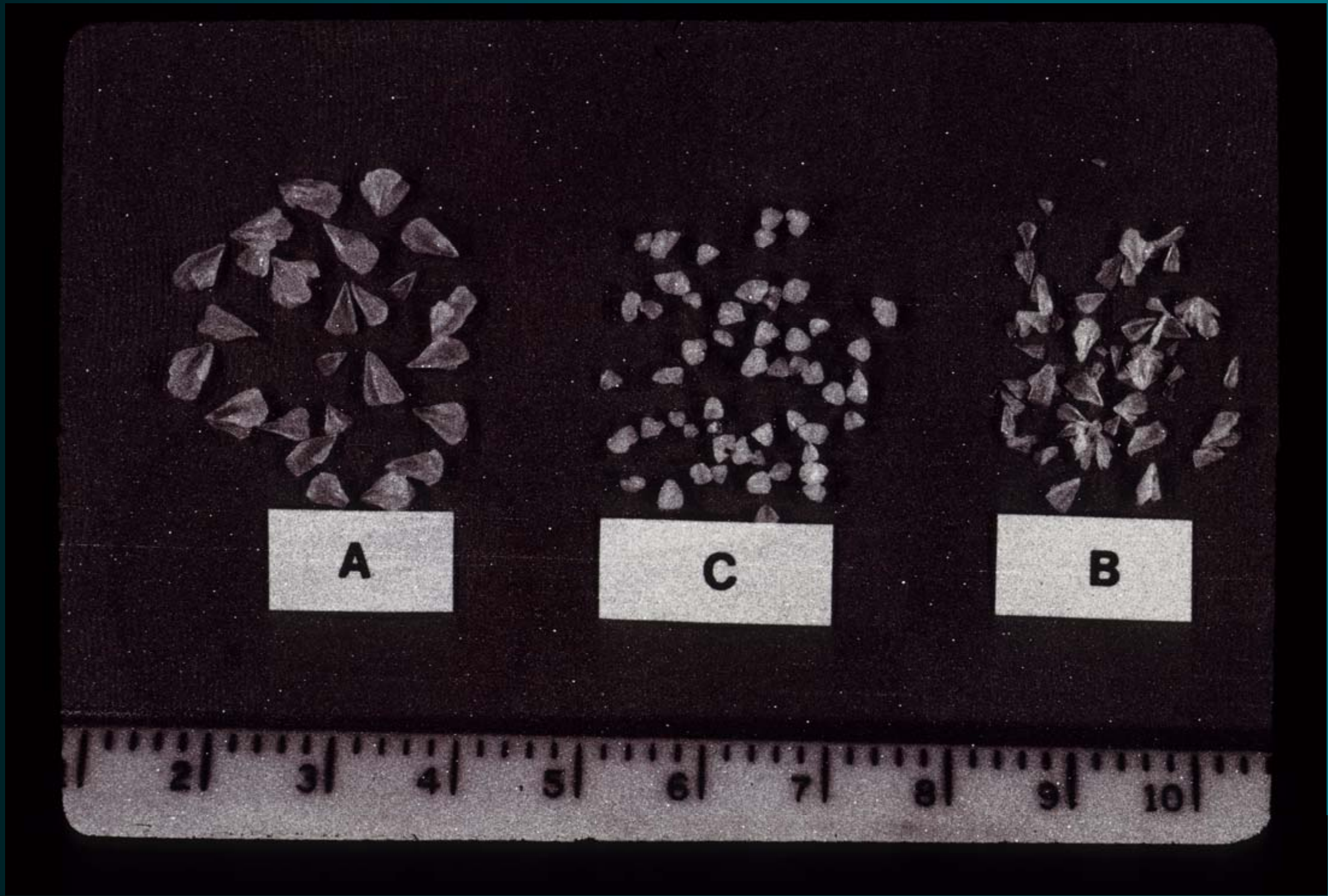
Lactose crystals in whey cheese Mysost



Lactose crystals in whey cheese Mysost



Effect of mineral impurities on lactose crystals



Sweetness of lactose

- Much lower than sucrose (20 % at 5% conc.)
- Effect of temperature and concentration
- Addition of less that 1% lactose to milk clearly noticeable (Jelen & Michel, 1999)
- Lactose used for down standardization of protein in dry milk
- Sweetness can be increased by hydrolysis

Lactose hydrolysis

Reasons	Methods
Low solubility (sandiness)	Acid + heat
Low sweetness	Free enzyme
Lactose intolerance	Immobilized enzyme
Oligosaccharides	Disrupted bacteria

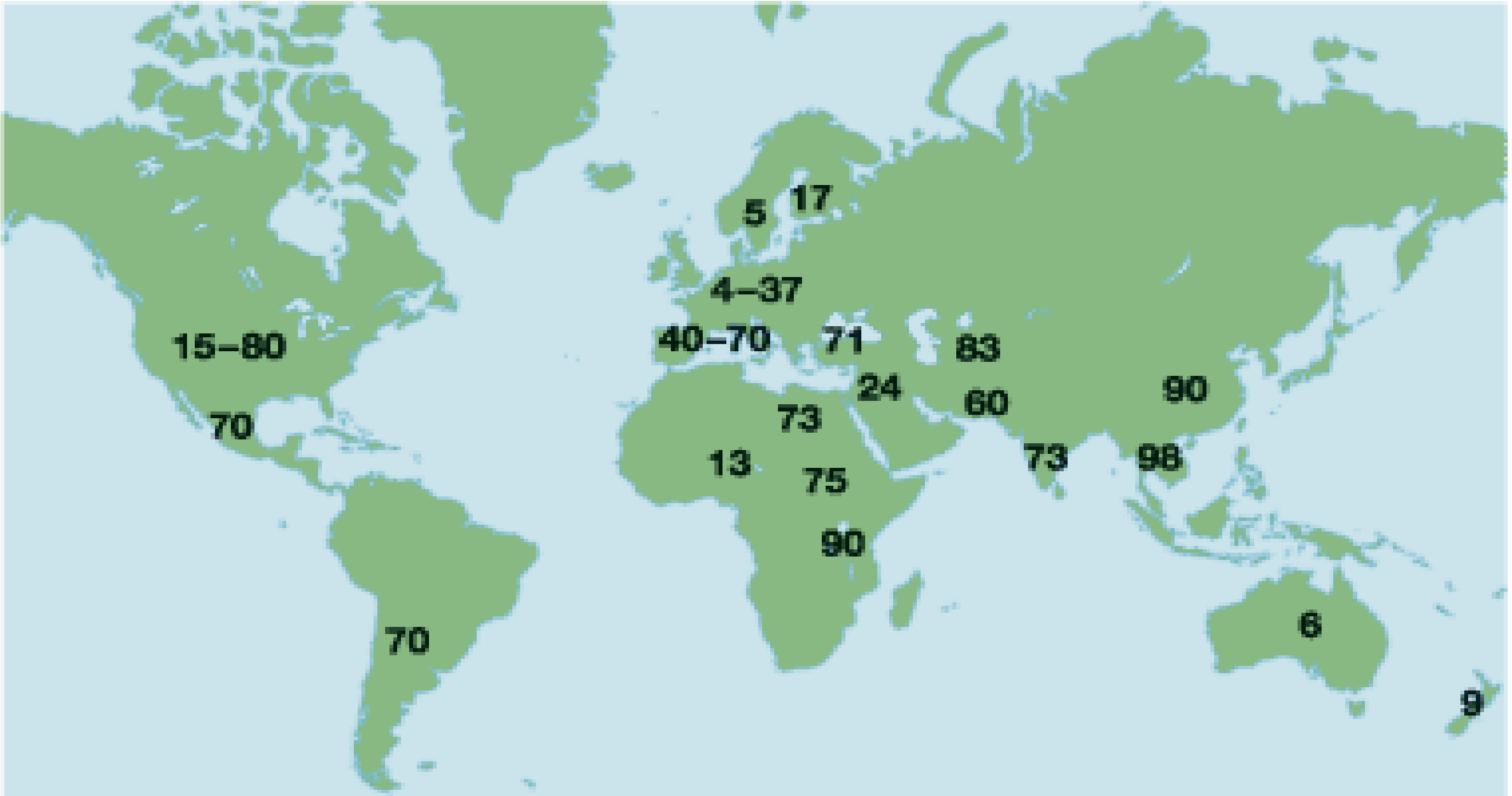
50 Mpersons market in USA alone

Characteristics of lactose hydrolysis methods

- Acid-catalysed hydrolysis
  - technological and material problems
- Immobilised enzyme technology
  - rarely succesful (Valio)
- Membrane - based enzyme reactors
  - theoretically interesting, rarely used
- Free (soluble) purified enzymes
  - used in industrial practice
  - Tetra-lacta process
  - home use in milk or as a dietary aid (pill)

How widespread is lactose intolerance in the world?

Lactose intolerance around the world
by percentage of population



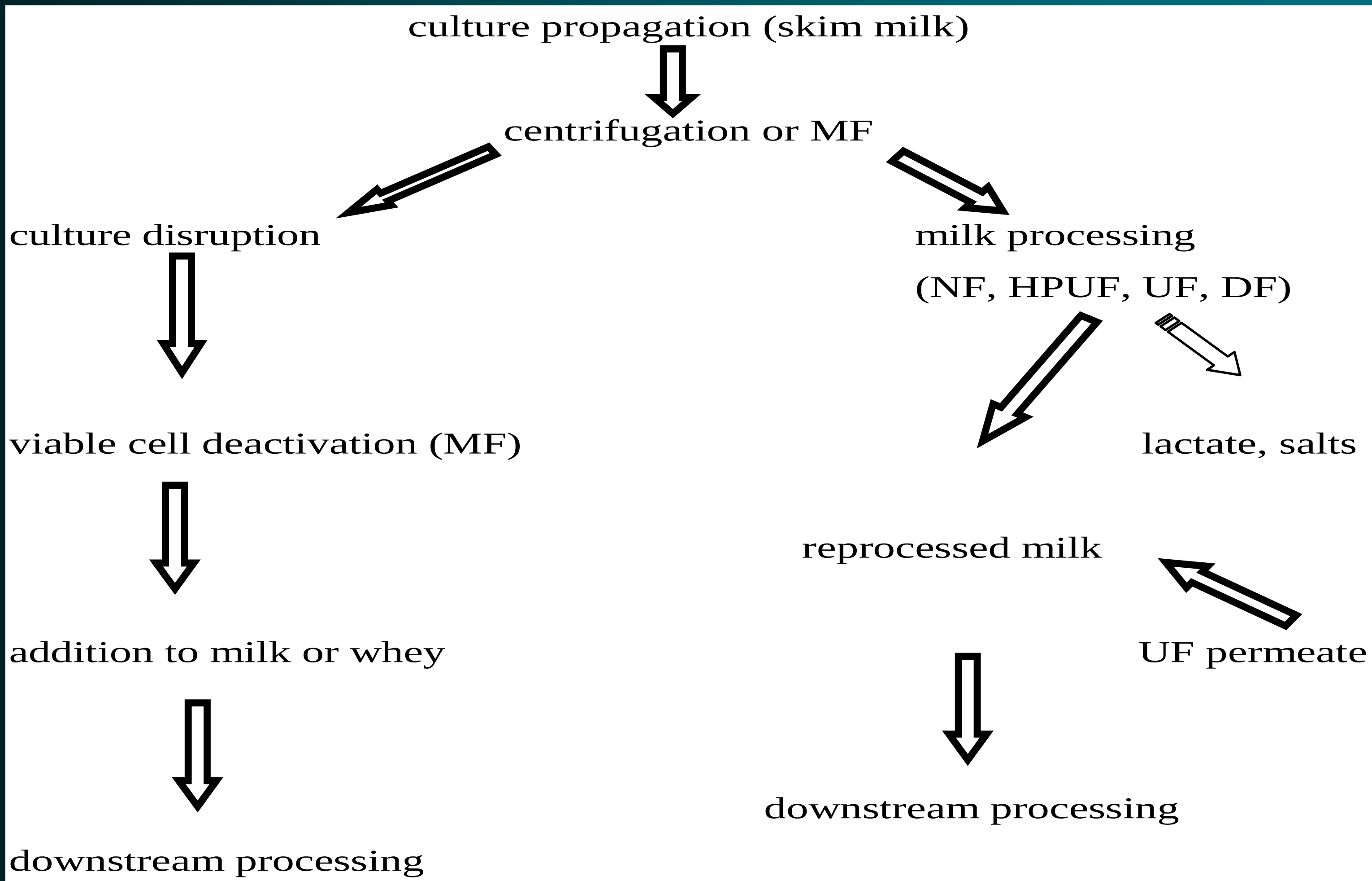
Lactose hydrolysis by disrupted lactic acid bacteria

- framework: Canadian research network on lactic acid bacteria for applications in dairy industry (University of Alberta).
- objective: to investigate a “simple” approach to the lactose hydrolysis problem using lactase enzyme produced by common dairy bacteria after their disruption as crude enzyme extracts
- (CEE) obtained by microfluidization

Components of the proposed lactose hydrolysis process

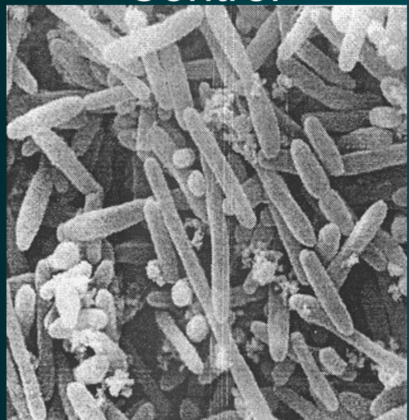
- well defined (GRAS) **enzyme source** **(*Lactobacillus bulgaricus*** 11842**)**
- well defined (GRAS) **medium** **(skim milk, whey)**
- culture production, separation and **disruption** **(**bead mill, high pressure homogenizer, Microfluidizer)
- use of the “dirty” CEE for lactose **hydrolysis** without additional purification

Schematic representation of the proposed process

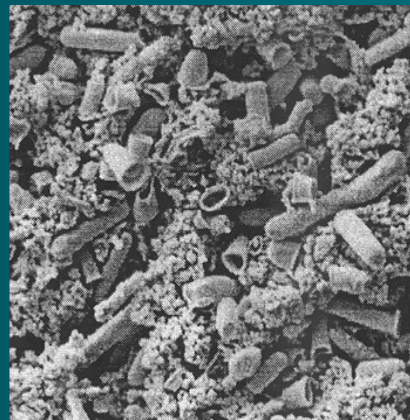


SEM images of Disrupted *L. bulgaricus 11842*

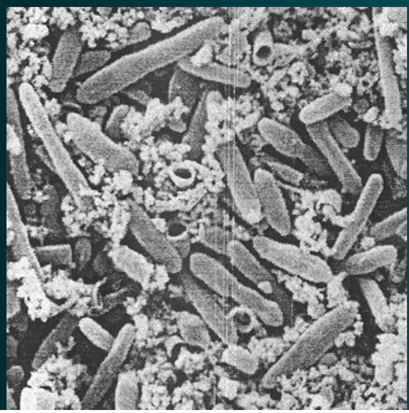
Control



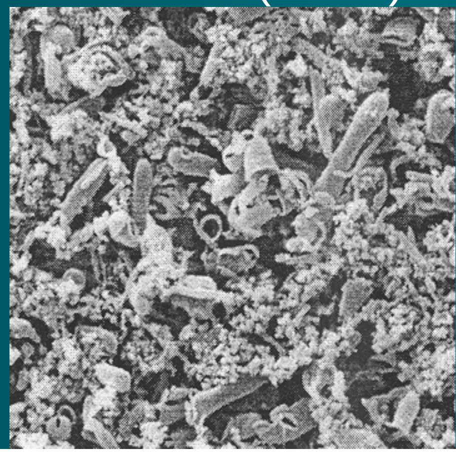
1 pass (homogenizer)



Sonication (6 min)



Bead Mill (6 min)



Lactose derivatives: GALACTO - OLIGOSACCHARIDES

Properties

- Di-, tri-, tetra- or higher -saccharides
- Intermediate sweetness
- Highly heat and acid stable
- Bifidogenic factor
- Non-digestible

Applications

- Probiotic foods
- Nutraceutical (FOSHU) foods (anticarcinogenic)
- Non-cariogenic foods
- Competing against inulin

Lactose derivatives as value added products

- Lactulose: 16 kt/year
- Lactitol: 10 kt/year in 1 plant
- Lactobionic acid: potential for 1kt/year in 1 German plant
- Oligosaccharides:
  - Galactooligosaccharides: n/a
  - Lactosucrose: 1.6 kt/year

Pharmaceutical lactose products

α-lactose:

- 100 mesh (>125 µm)
- Agglomerated / granulated
- Spray dried (80% crystals, 20% amorphous)

Anhydrous lactose:

- α -lactose (heated >130°C)
- β-lactose (crystallized >93°C)

Pharmaceutical lactose processes

Whey or UF permeate (crystallization)

↓

Crude lactose

↓

Refining recrystallization

↓	↓	↓	↓
Refined α-lactose	Heating >130°C	Spray drying	Crystallization on roller driers >93 °C
	↓	↓	↓
	Anhydrous α -lactose	Spray dried α-lactose	β-lactose

Industrial processors of pharma-lactose

Manufacturer	Product trade name	Lubricant
Meggle	Tablettose Cellactose	- Cellulose
BASF	Ludipress	PVP
DMV	Pharmatose	Lactitol

New aspects of lactose science and industrial applications

- Use of microcrystalline lactose as a flavour carrier
- Lactose effects in microencapsulation of fat by WPC
- Caking in bulk lactose
- Use of lactose for protein standardization in non-fat-dry-milk and fluid milk

Traditional uses of isolated lactose

Food (1996 total 425 kt)	Other (1996 total 175 kt)
Infant foods	Pharmaceuticals
Confectionery	Fermentation
Other (bakery, dry mixes)	Feeds
Dairy (protein standardization)	Derivatives

THANK YOU!