Recent improvements in lactose crystallization and in drying parameters for improving quality and uses of acid whey and of related powders

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Background and Objectives

Lactose Crystallization and Thickening

Stickiness

Conclusions
Acidification
Cooling
Heat Treatment

Milk

Microfiltration
Ultrafiltration

Standardization
Renneting
Mineral addition

Casein
Retentate
WPC / I

Ultrafiltration

Permeate
MF / UF

± acid / sweet
± rich in minerals
± rich in proteins
± rich in lactose
± rich in residual fat
± rich in µ-organisms

Cheese

Whey
## Biochemical composition of whey and derivates

<table>
<thead>
<tr>
<th>Component</th>
<th>Range</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Proteins</strong></td>
<td>0 to 6 g.L(^{-1}) ± denaturated</td>
</tr>
<tr>
<td><strong>Minerals</strong></td>
<td>0 to 6 g.L(^{-1})</td>
</tr>
<tr>
<td><strong>pH</strong></td>
<td>2 to 7</td>
</tr>
<tr>
<td><strong>Lactic acid</strong></td>
<td>1 to 7 g.L(^{-1})</td>
</tr>
<tr>
<td><strong>Lactate</strong></td>
<td>1 to 7 g.L(^{-1})</td>
</tr>
<tr>
<td><strong>Lactose</strong></td>
<td>30 to 50 g.L(^{-1})</td>
</tr>
<tr>
<td><strong>Glucose, Galactose, etc.</strong></td>
<td>1 to 5 g.L(^{-1})</td>
</tr>
<tr>
<td><strong>EPS, etc.</strong></td>
<td>Traces</td>
</tr>
<tr>
<td><strong>Residual fat, Phospholipides</strong></td>
<td>Traces</td>
</tr>
<tr>
<td><strong>NPN (0 to 2 g.L(^{-1})), aa, NH(_3)</strong></td>
<td></td>
</tr>
</tbody>
</table>
Permeate and Whey

Minerals

pH

Lactic Acid

Lactate

Fat residual, Phospholipides

Proteins

± denaturated

NPN, aa, NH₃

Lactose, Glucose, Galactose, EPS, ...

D I E T
A G R I C U L T U R E
E N V I R O N M E N T
Processing of whey powders & derivates

Whey
- Heat Treatment
- Whey Fractionation (MF, UF, NF, IEC, ED)
- Vacuum Evaporation

Concentrated Whey
- Lactose Crystallization

Crystallized Concentrated Whey
- Spray Drying

Whey Powder
- Storage

Whey Powder
TS = 98% (w/w)

Concentrated Whey
TS = 55% (w/w)

Whey
TS = 5% (w/w)
Processing of whey powders & derivates

Whey Powder | TS = 98% (w/w)

- Heat Treatment
- Whey Fractionation (MF, UF, NF, IEC, ED)
- Vacuum Evaporation

Spray Drying

Crystallized Concentrated Whey

Lactose Crystallization

Concentrated Whey | TS = 55% (w/w)

- Heat-Induced Protein Denaturation
- CaHPO₄ Precipitation

Variability in Kinetics and Crystal Size

Extensive Thickening

Fouling

Storage

TS = 5% (w/w)

Variability in Kinetics and Crystal Size

Caking

Maillard Reactions

Loss of Solubility

Stickiness
Background and Objectives

Lactose crystallization and Thickening

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Conclusions
Viscosity = \frac{\text{Shear stress}}{\text{Rate of Shear Strain}} = \frac{k \cdot \text{Torque}}{k' \cdot \text{Angular Velocity}} \text{Maintained Constant}

Controlled conditions of T°C and stirring speed / geometry

AR 2000 Rheometer head

Four blade paddle stirrer

Fill level

Water jacket

30 mm diameter cup

\text{Torque} = f \text{ (time)}
Thickening at lab scale

Concentrated Acid Whey

Refraction index (°Brix)  Lactose Crystallization  Torque (µN.m)

1. Decrease in the concentration of soluble phase
2. Lactose crystals counterbalance viscosity decay
3. Sharp increase in viscosity = Thickening

(Mimouni et al. 2007)
Separation of proteins and lactose crystals from concentrated whey

Lactic Acid Whey

- Concentration
- Lactose Crystallization
- Centrifugation
- Filtration

Proteins

UF membrane

Lactose crystals

Lactose Crystal-Free Concentrated UF of Acid Whey

(Mimouni et al. 2007)
Thickening occurred regardless of the presence of proteins or crystals of lactose.
Viscosity of particle suspensions:
- increases with volume fraction of particles (Petrie, 1999)
- strongly increases with the particle aspect ratio (i.e. elongation) (Pabst, 2006)
Influence of pH

Torque Amplitude (µN.m) (®)

(Mimoun et al. 2007)
Influence of pH

Lactic Acid Whey

- $\text{H}_3\text{PO}_4$ (pH 3)
- $\text{H}_2\text{PO}_4^-$ (pH 4)
- $\text{HPO}_4^{2-}$ (pH 5)
- $\text{PO}_4^{3-}$ (pH 7)

Anions

- $\text{RCOO}^-$
- $\text{RCOOH}$

Association constant with Ca$^{2+}$ (L.mol$^{-1}$)

- $\text{RCOO}^-$: 15
- $\text{H}_2\text{PO}_4^-$: 11
- $\text{HPO}_4^{2-}$: 642
- $\text{PO}_4^{3-}$: $2.88 \times 10^6$

(Holt et al., 1981)
(Mimouni et al., 2007)
Influence of Ph Example

<table>
<thead>
<tr>
<th>pH</th>
<th>Phosphate concentration (g.100 g^{-1} H_2O)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.5</td>
<td>5.0 ± 1.5</td>
</tr>
<tr>
<td>5.1</td>
<td>60.4 ± 0.9</td>
</tr>
<tr>
<td>6.1</td>
<td>64.7 ± 2.4</td>
</tr>
<tr>
<td>1.5</td>
<td>4.4 ± 0.5</td>
</tr>
<tr>
<td>40.2 ± 4.1</td>
<td></td>
</tr>
<tr>
<td>25.0 ± 4.7</td>
<td></td>
</tr>
</tbody>
</table>

Torque amplitude (ΔF, 10^{-4} N.m) of calcium lactate supersaturated solutions ([Ca] = 1.34 g.100 g^{-1} of H_2O), at different pH, during stirring at 120 rad.s^{-1} and 20°C, with and without phosphate ions.

(Mimouni et al. 2007)
Background and Objectives

Lactose crystallization and Thickening

Stickiness

Conclusions
Acid lactic whey / permeate, mono & disaccharides, polyols, hydrolyzed compounds, minerals

Low $T_g$ / ^ Stickiness

- Inlet $\theta$ & Flow rate
- Outlet air $\theta$ & AH
- $\theta$ Droplet & Powder

$T_g$ Lactic acid = -60°C

$T_g$ Lactose = + 90°C

Integration of $T_g$
Materials

Pilot workshop: Research and development for evaporation/drying

«MSD type» drying tower
80 kg of water evaporated per hour
**Spray drying**

### [Acid lactic whey] at ≈ 55% TS, at ≈ 80% crystallized lactose

<table>
<thead>
<tr>
<th></th>
<th>Inlet $\theta$°C</th>
<th>Outlet $\theta$°C</th>
<th>AH g.kg$^{-1}$DA</th>
<th>[C] kg.h$^{-1}$</th>
<th>Powder kg.h$^{-1}$</th>
<th>€ / ton Water</th>
<th>€ / ton Powder</th>
</tr>
</thead>
<tbody>
<tr>
<td>[ALW]</td>
<td>233</td>
<td>89</td>
<td>41</td>
<td>258</td>
<td>185</td>
<td>78.5</td>
<td>61.6</td>
</tr>
<tr>
<td>[ALW]</td>
<td>155</td>
<td>75</td>
<td>26</td>
<td>129</td>
<td>92</td>
<td>99.9</td>
<td>78.5</td>
</tr>
</tbody>
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Lactose crystallization and Thickening

Stickiness

Conclusions
Origin of thickening is due to the formation of calcium lactate crystals.

Concentrated lactic acid whey is likely to thicken because of:

- High [Ca] + High [Lactate] + pH ~ 4.5

Origin of stickiness during spray drying is due to the low T_g value of lactic acid (-60°C).

To improve the quality of the lactic acid whey powder:

- Modify the calcium lactate supersaturation (pH; Phosphate; Citrate; TS; Temperature)
- Modify the spray drying parameters (C flow rate, inlet and outlet air θ, outlet air AH)
MERCI

THANK YOU