Complimentary Calcium Fractionation Techniques to Increase Coproduct Solids Value and Utilization

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Wisconsin Center for Dairy Research

Center for Dairy Research “Solution Based Research Backed by Experience, Passion and Tradition”
Presentation Outline

1. Background
2. Project Philosophies
3. Calcium Fractionation
4. Trials & Data
5. Conclusions
Background

- Co-Products are UF Permeates derived from either Milk, Sweet Whey or Acid Whey
- AW contains 3X more Ca than Milk Perm.
- Calcium becomes Insoluble by Increasing any combination of three Variables; pH, Temperature and/or Ca Concentration
- Processing Examples that exceed Calcium Solubility are 2X NF/RO of Milk Permeate due to its High pH or AW Evaporation due to heat & concentration even with pH < 4.6!
Lots of Acid Whey Research Activity
Stokes Law for Sedimentation Velocity

\[ V = \frac{2}{9} \frac{(\rho_p - \rho_f)}{\mu} g R^2 \]

- Defines the Variables to consider for particle separation
- Particle Radius (R) Squared has a very significant impact
  - Thus double the Radius and it will travel 4X faster
- Density Difference is Necessary \([ \rho \text{ (particle)} - \rho \text{ (fluid)} ]\)
  - The particle density may be fixed, but measure it
  - The fluid solids (density) has an upper limit
- The Force applied (g) or use more force Mechanically
This Study’s Philosophy

• The High Calcium Phosphate content of Acid Whey is both a problem and an opportunity
• Fractionating Coproducts increases the Purity of both streams created by the separation
• Generate Process Rate and mass balance Data available for Economic Evaluations
• Create the Prototypes which are necessary to explore New Applications
Ca Fractionation Options

1. Mechanical via Hydrocyclones

2. Filtration Concentration of;
   a. Soluble Ca via Nanofiltration
   b. Insoluble Calcium utilizing High Temperature compatible, open pore size UF elements from Germany
Generating Calcium Phosphate (CaP)
CaP Yield Potential is 7% of the AW Permeate Solids

Acid pH UF Permeate
NF or not?

Standard NF to Retain Lactose

NF Retentate
The Ca & Lactose are concentrated

Heat to 150 F

Alkali to pH 7

NF Permeate is mostly Lactic acid, Na, K, Cl & Galactose

NF lowers the costs to make & process the CaP
The Hydrocyclone Design
Our First Hydrocyclone Test Run

The Full Process

Hydrocyclone mounted horizontally & the Yield

The CaP harvested
# Hydrocyclone Apex Size Options, Flow Rates and Volume Split

<table>
<thead>
<tr>
<th>Pump speed (Hz)</th>
<th>Apex nozzle diameter in mm &amp; inches</th>
<th>Feed flow (L/min) via Mag flow meter</th>
<th>BF flow (L/min) via beaker &amp; timer</th>
<th>BF as a % of Feed Flow</th>
<th>OF flow (L/min) by Difference</th>
</tr>
</thead>
<tbody>
<tr>
<td>60</td>
<td>4.8 &amp; 3/16</td>
<td>52.6</td>
<td>0.27</td>
<td>0.5%</td>
<td>52.3</td>
</tr>
<tr>
<td>60</td>
<td>6.4 &amp; 1/4</td>
<td>53.0</td>
<td>0.74</td>
<td>1.4%</td>
<td>52.3</td>
</tr>
<tr>
<td>60</td>
<td>7.1 &amp; 9/32</td>
<td>53.0</td>
<td>0.82</td>
<td>1.5%</td>
<td>52.2</td>
</tr>
<tr>
<td>60</td>
<td>7.9 &amp; 5/16</td>
<td>53.0</td>
<td>1.44</td>
<td>2.7%</td>
<td>51.6</td>
</tr>
<tr>
<td>60</td>
<td>9.5 &amp; 3/8</td>
<td>53.0</td>
<td>&gt;4.0</td>
<td>&gt; 7.5%</td>
<td>&lt;49.0</td>
</tr>
</tbody>
</table>
Another Hydrocyclone Test
This test was done on Ca lactate? before the DMI project started

The Large beaker contains HC Overflow (OF). Note how its been aerated and a flocculated precipitate was not removed.
The Small beaker contains HC Bottom flow (BF) which is the a concentrated stream of Calcium Precipitate
How Turbulence Impacts the CaP Particle Size Range Generated

NF Concentrate from AW UF Permeate

Ca depleted
Use for edible lactose

Special high temperature compatible Ultrafiltration

Heat Neutralize

Co-mingled OF

Decant to waste

NF Concentrate from AW UF Permeate

Heat Neutralize
CaP Particle Size Determined by
1. Low Turbulence Neutralization (LTN)
2. High Turbulence Neutralization (HTN)
Uniform Size Classification

The HC Overflows (solids lines) have very similar Particle Size Profiles
Effect of Washing CaP
The Large CaP Particle (the red profile) Washed by decanting to achieve purified CaP (the green profile)
Impact of Apex Diameter Utilized

Hydrocyclone Apex Nozzle Size did not impact Size Separation Efficiency, but only how much large particles are concentrated.

The three Apex Sizes listed in the key are millimeter diameters.
Milk Minerals Powder

The two overflow streams were combined, run through Special UF, Diafiltered & spray dried

<table>
<thead>
<tr>
<th></th>
<th>Total solids (%)</th>
<th>Ash (% solids)</th>
<th>Calcium (% solids)</th>
<th>Phosphorus (% solids)</th>
<th>Ca:P (-)</th>
<th>(% Crude Protein)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>96.0</td>
<td>81.3</td>
<td>29.3</td>
<td>15.4</td>
<td>1.5</td>
<td>1.85</td>
</tr>
</tbody>
</table>
## Composition of Milk Permeate after Demineralization

“% Reduction” is the percentage of Ash or Protein Removed compared to Milk Permeate, the Process Feed material.

Note the High Lactose Purity Achieved

<table>
<thead>
<tr>
<th>Trial start Date</th>
<th>%Ash Dry Basis</th>
<th>%Protein Dry Basis</th>
<th>%Lactose Dry Basis</th>
</tr>
</thead>
<tbody>
<tr>
<td>1/22/15</td>
<td>5.0%</td>
<td>1.5%</td>
<td></td>
</tr>
<tr>
<td>% Reduction</td>
<td>43%</td>
<td>51%</td>
<td></td>
</tr>
<tr>
<td>7/27/15</td>
<td>5.9%</td>
<td>1.9%</td>
<td>91.7%</td>
</tr>
<tr>
<td>% Reduction</td>
<td>38%</td>
<td>37%</td>
<td></td>
</tr>
<tr>
<td>7/28/15</td>
<td>5.0%</td>
<td>1.6%</td>
<td>95.9%</td>
</tr>
<tr>
<td>% Reduction</td>
<td>43%</td>
<td>47%</td>
<td></td>
</tr>
</tbody>
</table>
Acid Whey Coproducts Demineralized with NF and Special UF

“% Reduction” is the percentage of Ash or Protein Removed compared to Milk Permeate, the Process Feed material

<table>
<thead>
<tr>
<th>Final Coproduct (from Greek AW)</th>
<th>% Calcium Dry Basis</th>
<th>% Sodium Dry Basis</th>
<th>% Potassium Dry Basis</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>.037%</td>
<td>2.3%</td>
<td>1.2%</td>
</tr>
<tr>
<td>% Reduction</td>
<td>82%</td>
<td>From NaOH</td>
<td>53%</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Final Coproduct (from Cottage AW)</th>
<th>% Calcium Dry Basis</th>
<th>% Sodium Dry Basis</th>
<th>% Potassium Dry Basis</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>.012%</td>
<td>2.5%</td>
<td>1.3%</td>
</tr>
<tr>
<td>% Reduction</td>
<td>95%</td>
<td>From NaOH</td>
<td>51%</td>
</tr>
</tbody>
</table>
Conclusion- CaP is in a better place

The Problem of Ca Soil blinding an Evaporator Plate Is Resolved by Producing Milk Minerals & then Edible Lactose
Acknowledgements

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