Novel Ceramic Nanofiltration to Improve whey UF Permeate Quality and Increase its Utilization

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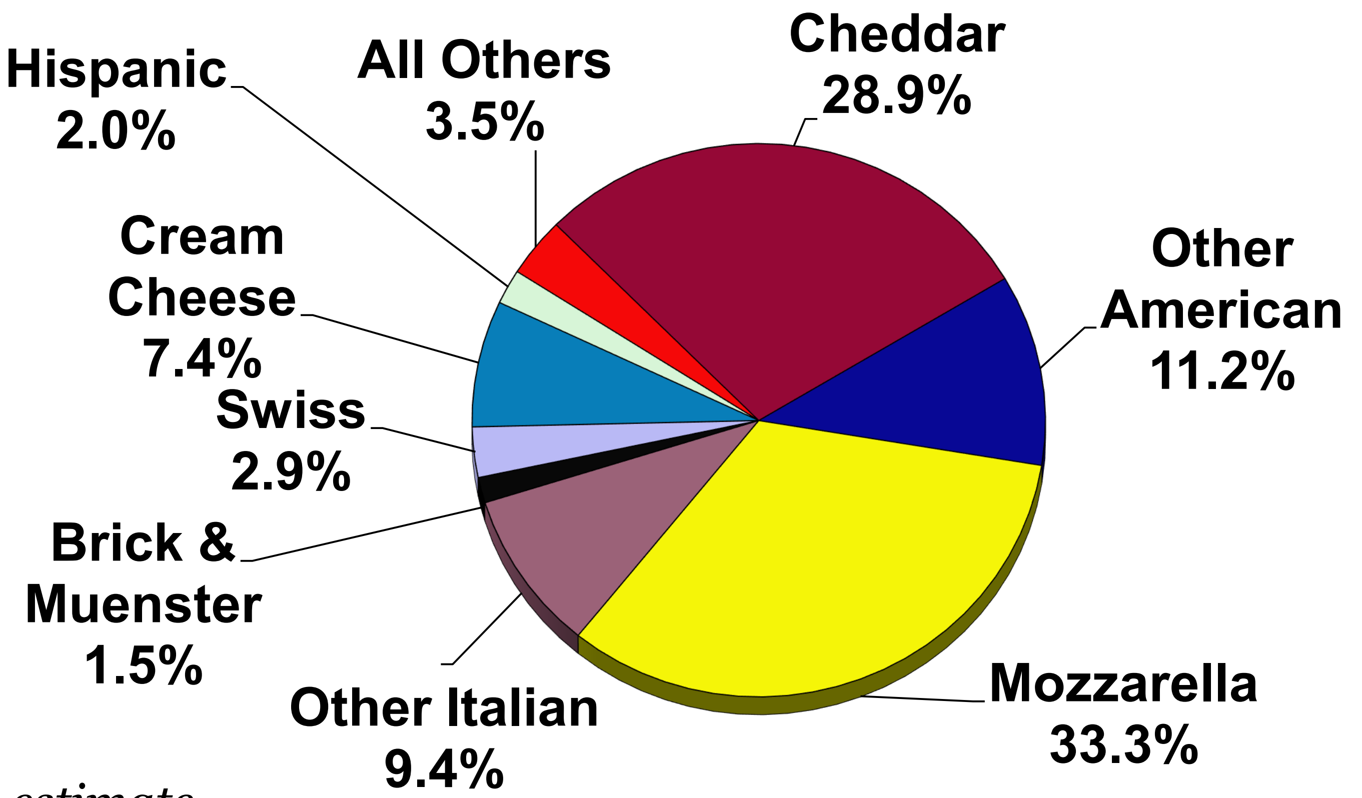
Center for Dairy Research *"Solution Based Research Backed by Experience, Passion and Tradition"*

Presentation Outline

- Noticing more frequent use of thermophilic cultures for cheese
- How the use of thermophilic cultures impacts the whey
- Ceramic nanofiltration element details
- Nanofiltration trials conducted to measure fractionation
- The experimental data and conclusions

U.S. Cheese[1] Production by Variety 2012*



**Preliminary estimate*

[1]Exludes Cottage Cheese

Source: *USDA, "Dairy Products Annual Summary"*

A Few Examples of Newer Varieties

- Cheddar and Parmesan ‘hybrids’
- Sweet Cheddar Types

Lots of Growth for Mozzarella and Hard Italian Varieties

The Change that Resulted in Additional Utilization of **Thermophilic** Strains

Bulk Starter made in-house mostly **Replaced** by Direct Vat Sets

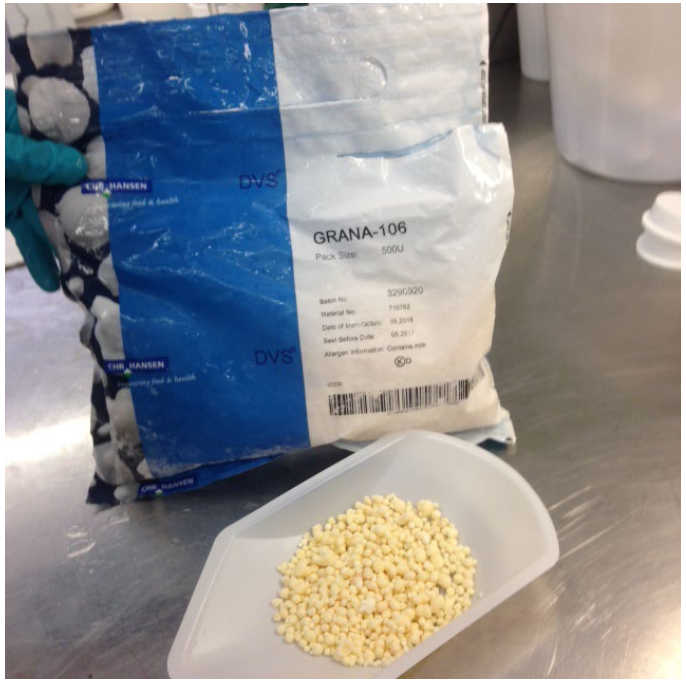
Even Cheddar, Monterey Jack, Colby & Related Varieties are Now Being Made with Some **Thermophiles**

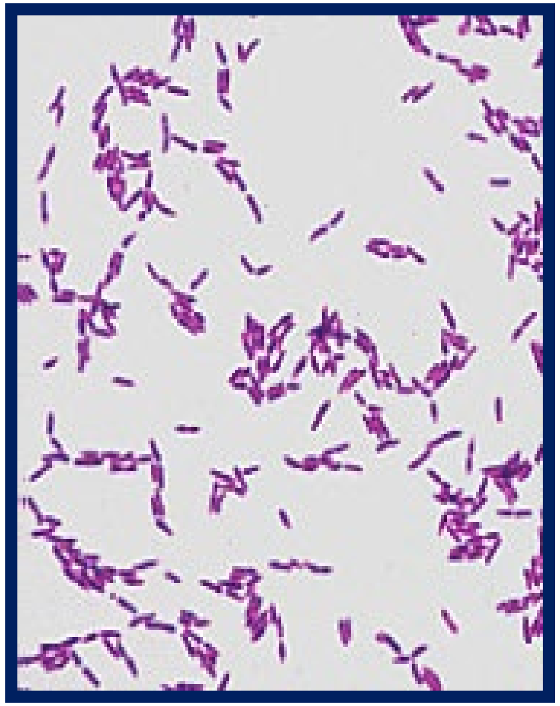
How Cottage Cheese & its Acid Whey Changed

Mesophiles are proteolytic given time. Because peptides do not make curd, **switching** to thermophiles provided a significant yield increase

Cottage Whey UF Permeate Composition	Peptides dry basis	Lactose dry basis	Galactose dry basis	Lactic Acid dry basis
Traditional (Mesophiles)	>1%	65%	Negligible	>10%
New Makes (Thermophiles)	Negligible	55%	>10%	>10%

If Starter Cultures are **the Engines** for Cheesemaking and the **Fuel** is Lactose, Which of those Engines are **Half** as Efficient?





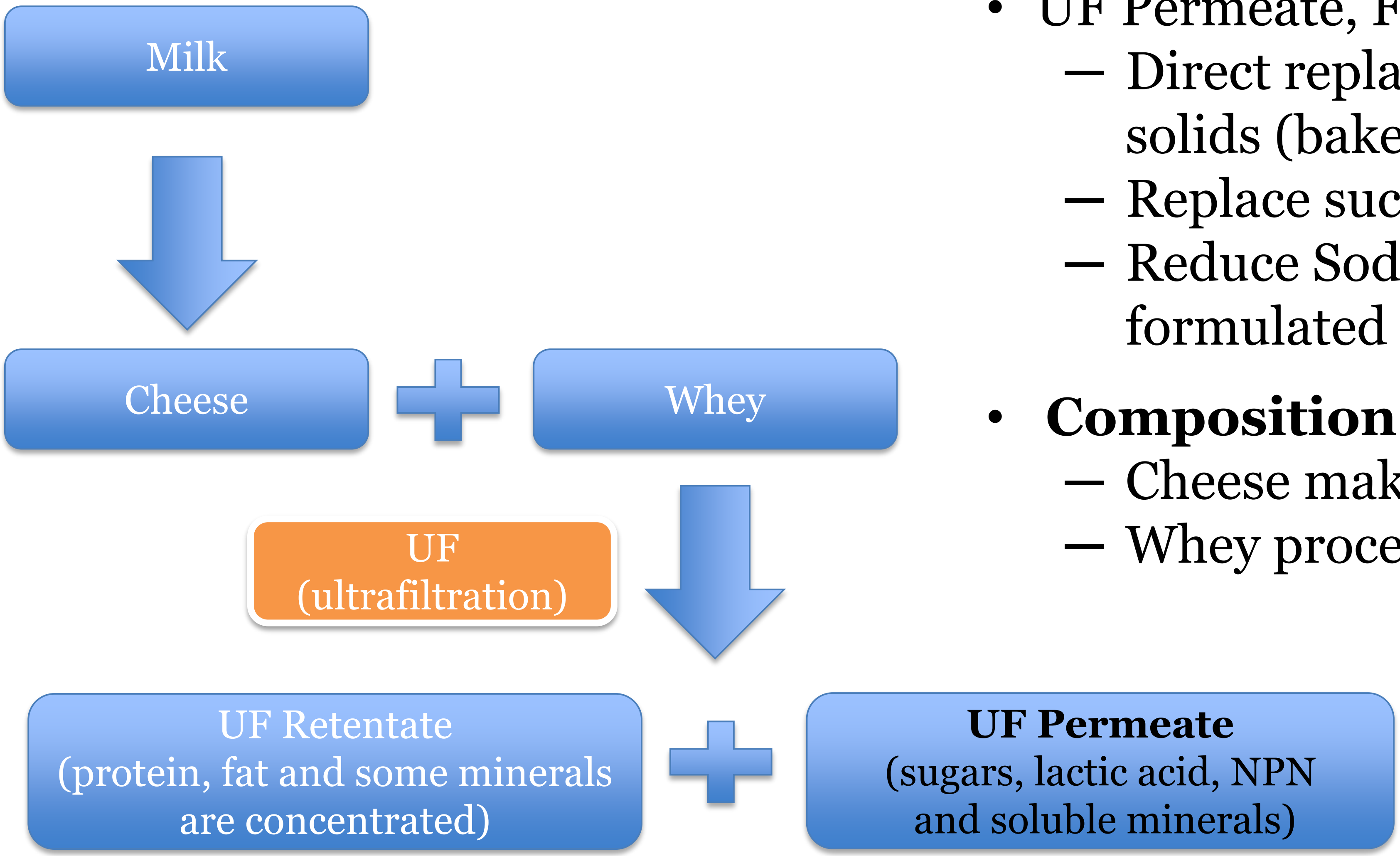
Lactic Acid is the Goal

One Lactose + one water molecule → **Mesophilic Cultures, 100% Efficient** → **Four** Lactic Acid molecules

One Lactose + one water molecule → **Thermophiles, 50% Efficient** → One **Galactose** + **Only Two** Lactic Acid

Most importantly, what to do with **galactose?**

More Background – How Whey UF Permeate is Utilized



- UF Permeate, Food Applications
  - Direct replacement of other dairy solids (bakery, confectionary)
  - Replace sucrose or corn syrups
  - Reduce Sodium level in formulated products
- **Composition varies** due to
  - Cheese making parameters
  - Whey processing efficencies

Whey Ultrafiltration (UF) Permeate composition Challenges Summarized

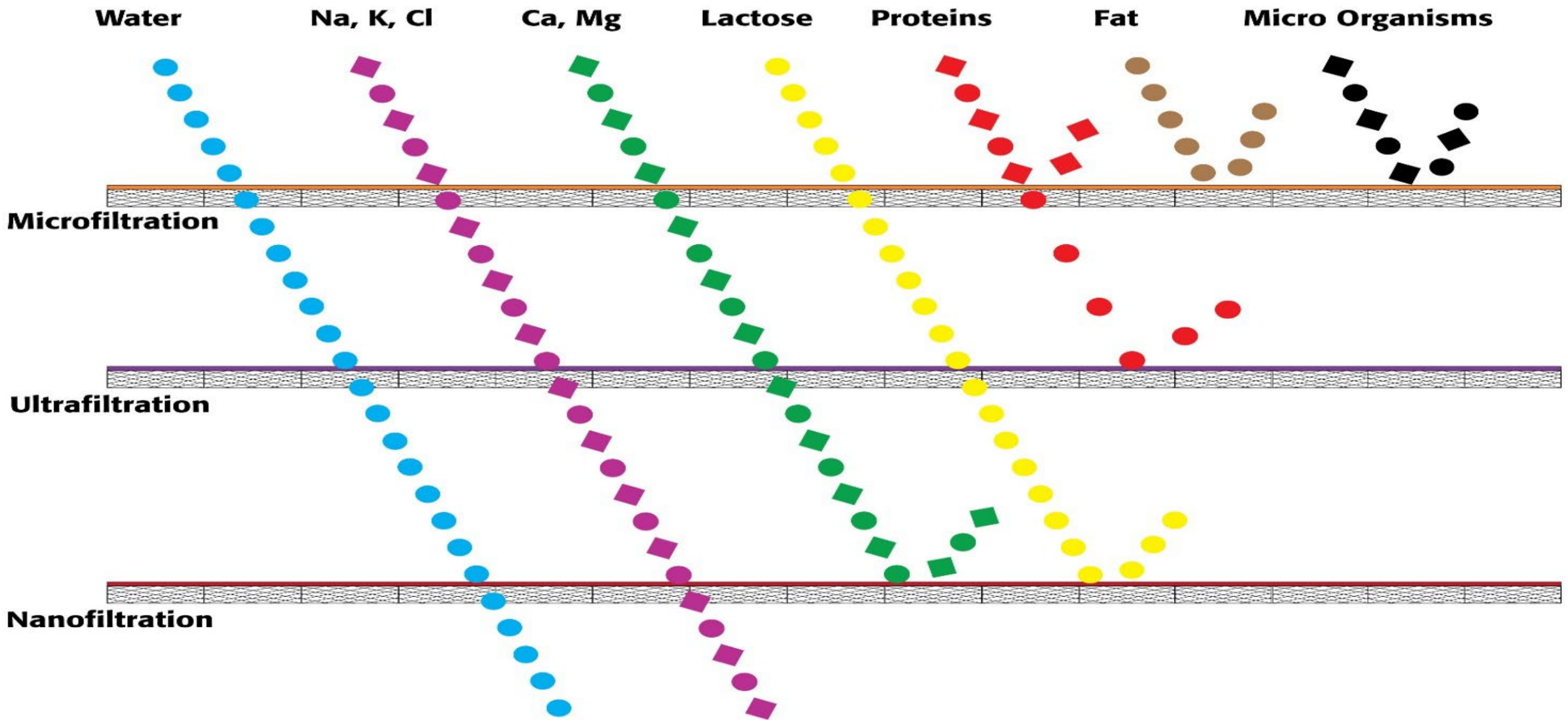
Lactic acid and **especially galactose** cause

1. Stickiness that reduces dryer throughput
2. And results in Maillard browning

Variable/High Salt (NaCl) content is common

1. Salt meant for the cheeses, often ends up in the UF permeate
2. The monovalent minerals can be removed from whey and UF permeate using **Nanofiltration**

The Three Categories of sanitary Membranes that Fractionate



-Note this research is focused on Nanofiltration

Previous Works Utilizing Spiral Nanofiltration Membranes to Fractionate Acid Whey

- Nanofiltration was very effective to fractionate deproteinized Greek and Cottage acid whey (Crowley et al., 2018):
  - Significantly reduced the galactose and lactic acid content
  - Significantly reduced the ash content
  - Effectively retained the lactose which is important
  - Concentrated the calcium phosphate for our process to create and purify milk minerals, a calcium supplement with very good bioavailabily

* Synder Spiral Nanofiltration Data

highlighting Effective Greek yogurt Acid whey **Fractionation**

	Ash dry basis	Lactose dry basis	Galactose dry basis	Lactic Acid dry basis
NF Feed (Greek Yogurt UF Permeate)	12%	55%	10%	12%
NF Retentate; Synder NFX (standard NF)	8.5%	67%	9%	6%
NF Retentate; Synder NFS (unique NF)	8.5%	67%	9%	6%
NFS Permeate	40%	** Not Detected	12%	45%

* Synder Filtration Inc., Vacaville, California USA

** <0.1% HPLC Lactose Detection limit for permeate with 1% solids

Synder Spiral Nanofiltration Data

NFS is the Option to Permeate some Calcium

	Calcium dry basis	Sodium dry basis	Potassium dry basis	Chloride dry basis
NF Feed (Greek Yogurt UF Permeate)	2.0%	0.6%	2.4%	1.5%
NF Retentate; Synder NFX (standard NF)	2.2%	0.3%	1.1%	0.5%
NF Retentate; Synder NFS (Unique NF)	1.8%	0.3%	1.0%	0.5%
NFS Permeate	3.6%	3.6%	12%	10%

Ceramic NF Research Hypothesis

1. Ceramic NF membranes may have sharper MWCO than polymeric membranes.
   – That would serve to more effectively fractionate (separate) galactose from lactose
2. UF permeate solids concentrated via NF will contain significantly reduced (dry basis) quantities of galactose, lactic acid and sodium chloride.

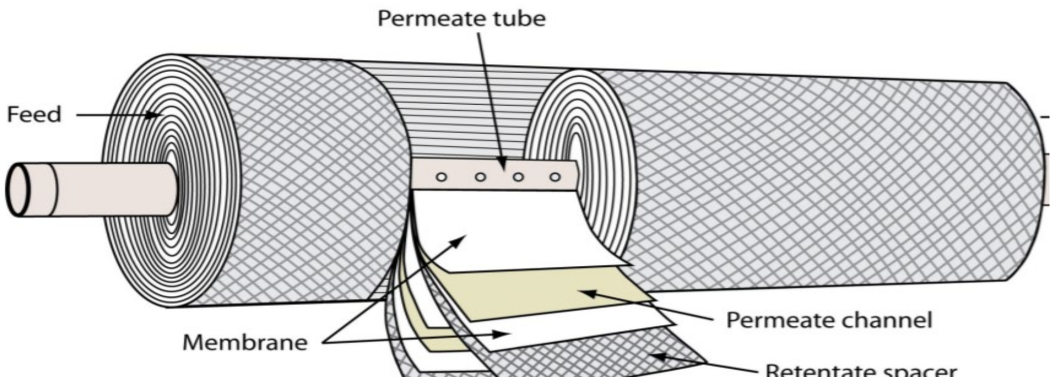
Research Objectives

1. Compare fractionation performance of various types of polymeric and ceramic NF membranes

2. Analyze the NF permeate and retentate samples for galactose, lactic acid, lactose, ash and individual minerals
3. Use the data to calculate and compare retention coefficients

Two Distinct NF Membrane Configurations

- Finding the right **pore size** Nanofiltration Membrane with two Goals
  - Allow for **galactose,** lactic acid and Na, K, Cl to readily pass through it
  - Also, it's critical retain a very high percentage of the lactose



Spiral-Wound

Ceramic

Benefits of Ceramic:

- High thermal resistance
- High chemical resistance
- Better cleanability due to the previous point
- Longer lifespan (elements are consumables)
- But does ceramic have both an **ideal** and **uniform** molecular weight cutoff (MWCO) to fractionate galactose from lactose (<2 fold MW Difference)

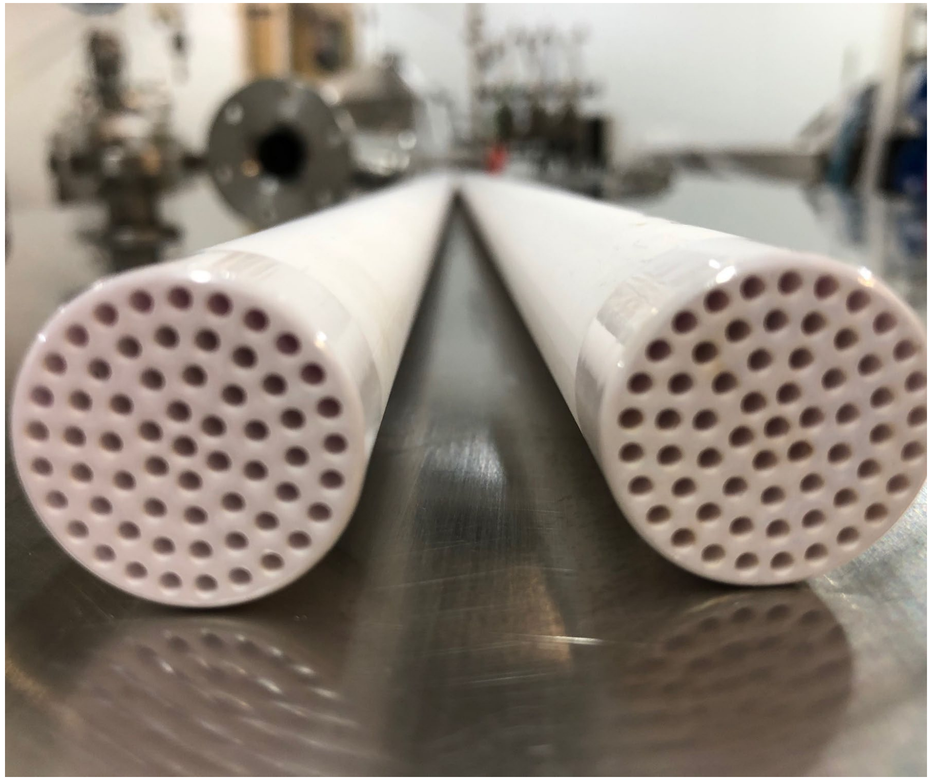
Inopor, the 1st Manufacturer? offering <u>true</u> NF Elements

- CDR Purchased all **three** of their NF membranes
- Their research **model EC** ceramic elements have;
  - 0.46 square meter of membrane
  - 250, 450 and 700 Dalton MWCO membranes
  - 25 mm (~1”) diameters and are 1.2 meters in length
  - Made with 61 (2 mm diameter) flow channels
  - Model EC requires crossflow of up to 15 gpm

(* 15 gpm corresponds to 5 M/sec. crossflow velocity)

Inopor Ceramic Elements & SS Housing from Germany

Each SS housing holds one element



Model EC Elements

Unique Points of Ceramic

Because not many dairy manufacturers are familiar with Ceramic Filtration

- The recommended Crossflow **is broad?**
  - Crossflow is critical for all sanitary filtration
  - 3 to 5 feet per second (9 to 15 gpm for model EC)
  - And 5 is **67%** faster than 3; thus a **broad range!**
- Ratio of Crossflow to Membrane area is HIGH
  - For Ceramic, that ratio is **7 fold** higher than spiral
  - Ceramic has less membrane & requires more flow!

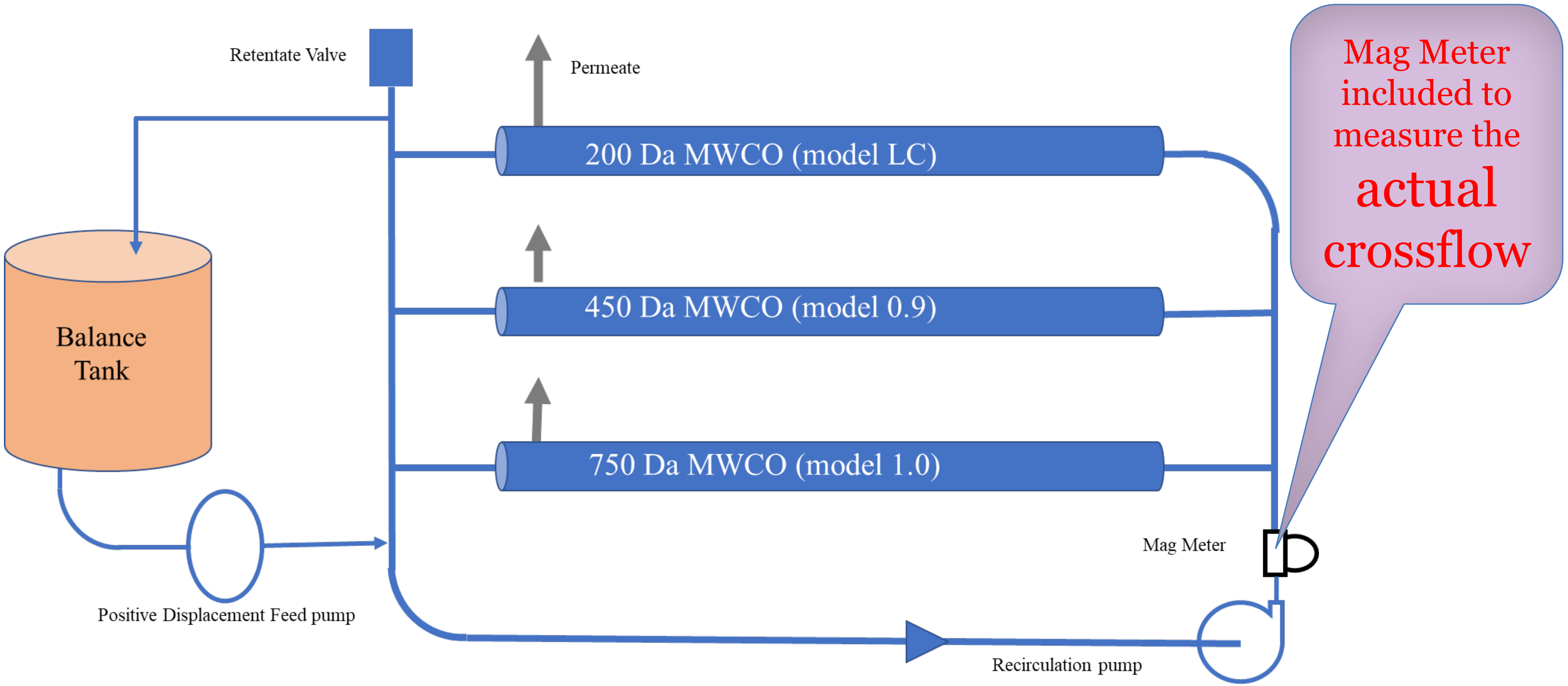
Our NF System within our Process Pilot Lab

Plus, those that occasionally stop by to give or take directions

Interviews regarding Acid Whey remain common

Our Ceramic NF Process Flow Diagram

Evaluating three unique membranes in parallel



Conducting the NF Evaluation Trials

1. To feed the process, it was ideal to make a consistent composition representative of whey UF permeate
2. Evaluate how membrane type and MWCO impact the NF permeate rate and composition.

Element Details:

- Five commercially available polymeric nanofiltration models were evaluated
- Three Inopor Ceramic elements were purchased and evaluated in parallel

Preparation of the consistent feed for NF runs

First, we prepared a simple sugar Syrup

- **CDR Simple sugars syrup:**
  - 10% lactose solution (pH adjusted to 7.0) + lactase
  - Hydrolyze at 39˚F for ~27 hours
  - Evaporate into syrup to ~66%TS:
    - ~6% lactose
    - ~30% galactose
    - ~30% glucose

NF Feed	Quantity
Commercial milk UF permeate powder	55 lbs
Simple sugars syrup	6 lbs
Lactic acid	1 lb
And the balance is Water	
Final Volume	**115 gal**

Comparing the Retention Coefficients (RC)
*RC = 1-(%X in Permeate/%X in the Retentate)

Molecular Wt. Cut Off	Galactose	Glucose	Lactic A.	Lactose
250 Ceramic *	0.75	0.76	0.76	0.96
450 Ceramic *	0.63	0.67	0.65	0.92
700 Ceramic *	0.64	0.63	0.64	0.91
Spiral (model A) **	0.93	0.93	0.79	1.0
Spiral (model B) **	0.82	0.85	0.71	0.99
Spiral (model C) **	0.90	0.93	0.75	1.0
Spiral (model D) **	0.94	0.96	0.78	1.0

* n=3 trials where the permeate and retentate were sampled together

** n=2 trials, calculations utilized averages of permeate & feed composites

Comparing the Retention Coefficients (RC)
*RC = 1-(%X in Permeate/%X in the Retentate)

Molecular Wt. Cut Off	Chloride	Calcium	Potassium	Sodium
250 Ceramic *	0.09	0.97	0.67	0.59
450 Ceramic *	0.02	0.94	0.55	0.45
700 Ceramic *	0.00	0.94	0.55	0.45
Spiral (model A) **	<0.1	0.99	0.50	0.47
Spiral (model B) **	<0.1	0.98	0.52	0.47
Spiral (model C) **	0.3	0.99	0.65	0.63
Spiral (model D) **	<0.1	0.99	0.57	0.54

* n=3 trials where the permeate and retentate were sampled together

** n=2 trials calculations utilized averages of permeate & feed composites

Comparing the Spiral Retentate Composition to the Feed Reported as dry basis, similar to powder composition

Molecular Wt. Cut Off	%Chloride	%Sodium	%Galactose	%Lactic Acid
Spiral (model A) **	0.33	0.47	3.3	0.59
Spiral (model B) **	0.33	0.55	1.7	0.38
Spiral (model C) **	0.28	0.56	2.4	0.35
Spiral (model D) **	0.28	0.52	2.5	0.48
NF Feed for Reference	1.54	0.90	2.4	0.52

** n=2 trials utilizing the retentate composite samples

Some Conclusions:

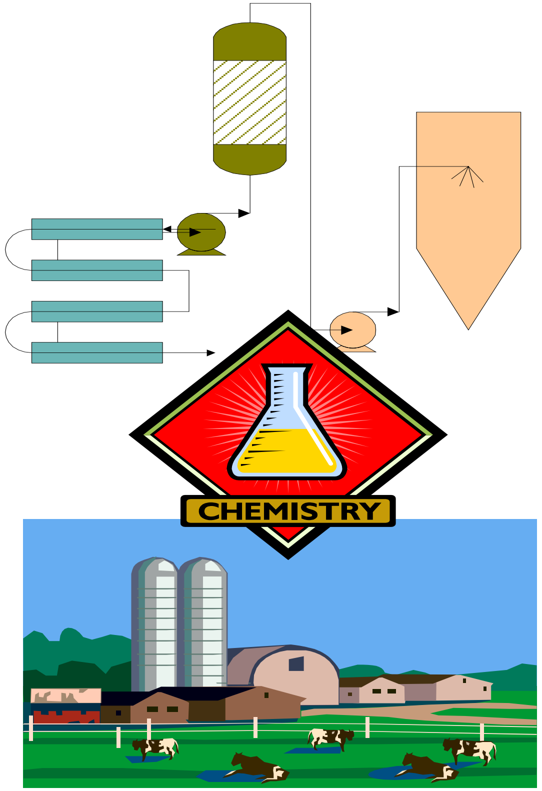
- There's less than a two-fold molecular weight difference between galactose and lactose. Thus, a challenging fractionation task.
- Ceramic did allow a bit more calcium to permeate than spiral does
- Ceramic did have the best (smallest) RC for galactose and Lactic acid. But ceramic also had the smallest lactose RC (too open, lactose leaks)
- Thus, relative to the galactose and lactose retention coefficients; ceramic does not appear to have a **narrow** molecular weight cut off
- Relative to membrane area, Ceramic requires dramatically higher crossflow velocity and more boost pressure than spiral (i.e. Ceramic require larger recirculation pumps & more or larger vessels (membrane)

Acknowledgements

National Dairy Council Funding

CDR Staff:

- Yanjie Lu for lots of Analytical support, etc.
- Dean Sommer for the cheese slides and coming to the lab to have your picture taken



Thank You

Project funding provided by the National Dairy Council managed by Dairy Management, Inc.