

# New approach for high purity lactose - utilising the by-products from whey

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A joint venture of CSIRO &  
the Victorian Government



# Whey disposal issues

- Cheese manufacture creates 9L whey for each kg cheese.
- Cheese production increasing - up 50% since 1996.
- 40% of whey currently disposed as waste.
- Australia produces only edible lactose and imports more expensive grades.



# Traditional lactose technology

Traditional lactose processes have many difficulties & give poor returns

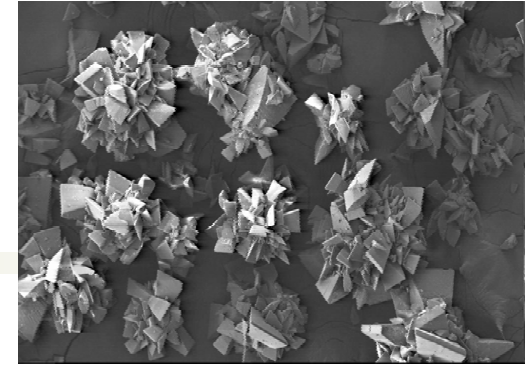
- Evaporator fouling – short runs & high cleaning wastes
- Crystalliser overloaded with impurities
- Water wasted from washing yellow, “salty” crystals
- Low yields at 65-75%
- Mother liquor – high in salt & lactose, waste problem

# The Challenge

- The Dairy Research & Development Corporation issued *a challenge* in 1993 ~  
*“Who can help solve the lactose problem?”*
- UWS & FSA came together to form the Food Fractionation Group for research into lactose.
- To develop new ways for processing lactose to:
  - minimise waste,
  - reduce costs,
  - maximise by-product utilisation,
  - produce safe, commercially viable, high value products.



# [ UWS/FSA Lactose research



- New methods for the purification of whey and lactose ~ patented IEL process (Durham).
- Lactose crystallisation; mechanisms of control & improving industrial lactose crystallisation (Vu).
- Novel patented AAC lactose crystallisation process ~ rosette structures with unique properties (Dalziel/Lifran/Sleigh).
- Lactose phosphate and mechanisms of lactose crystallisation inhibition (Lifran/Sleigh).
- Mechanisms of lactose caking & studies on the properties of anomeric forms of lactose (Listiohadi).

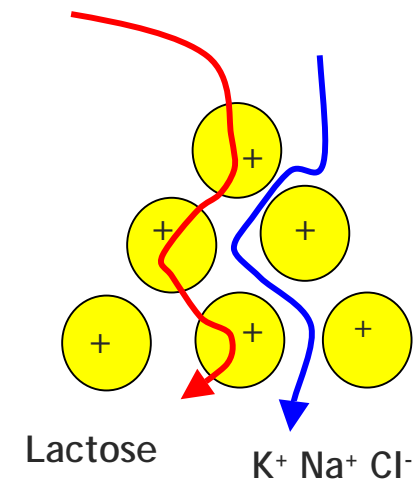
# [ New approach for high purity lactose ]

Based on chromatographic separation of minerals and lactose

## *Principles of chromatography:*

- Resin saturated with counter ions in same composition as those in whey.
- Ionic species (minerals) repelled to the exterior of the resin beads, so quickly move through the interstitial spaces.
- Non-ionic species (lactose) not excluded, and slowly permeate through the interior of the resin beads.
- Water used to elute lactose and mineral fractions through the long resin filled columns.

Whey permeate containing  
Lactose & Minerals



# Chromatographic experiments - Evaporated whey permeate

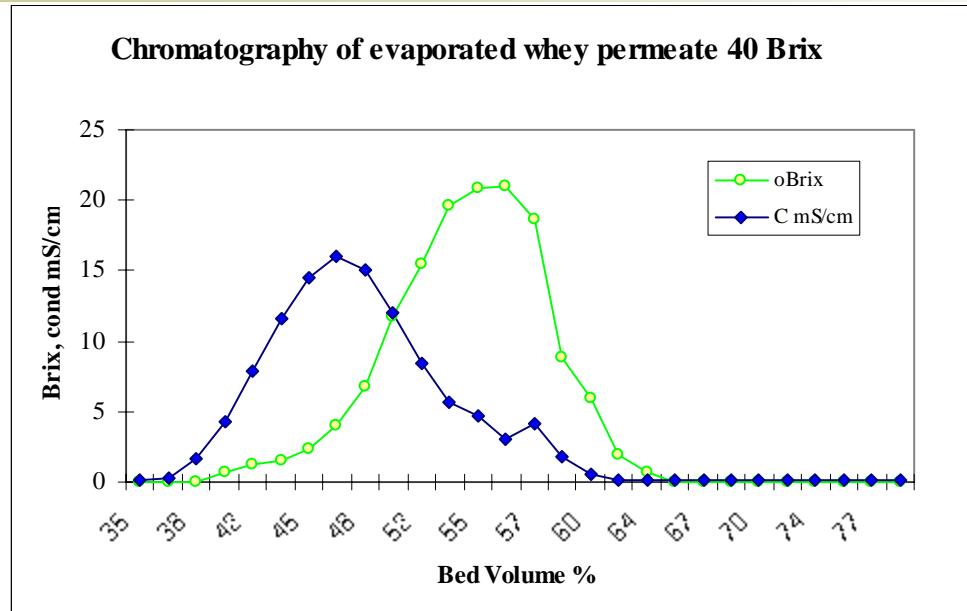


Figure 1. Evaporated whey permeate (40°B) on Mixed cation resin, 1.4m column

## *Problems with evaporated whey permeate:*

- Calcium phosphate (CaP) ppte in evaporated 40°B whey perm led to lactose losses ~ 20% due to occlusion in CaP ppte.
- CaP also precipitated on chromatography column, reacting with calcium counter ions on resin, blocking the column.

# Chromatographic experiments - Nanofiltered whey permeate

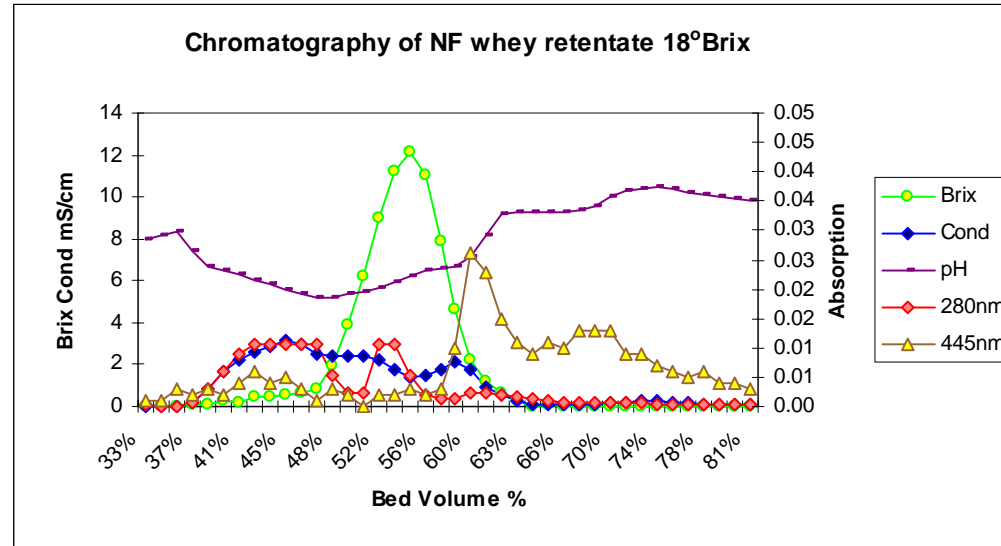


Figure 2. NF whey retentate (18°B) on mixed mono/divalent resin, 5.6m column

## *Problems with NF whey permeate:*

- Nanofiltered whey permeate (18°B) also contains CaP ppte, which when filtered ~ 4% lactose yield lost.
- Poor separation - Triple mineral peak, riboflavin (445nm) and peptides (280nm) contaminated lactose fraction.

# Comparison of chromatographic separations based on 'valency' of whey and resin

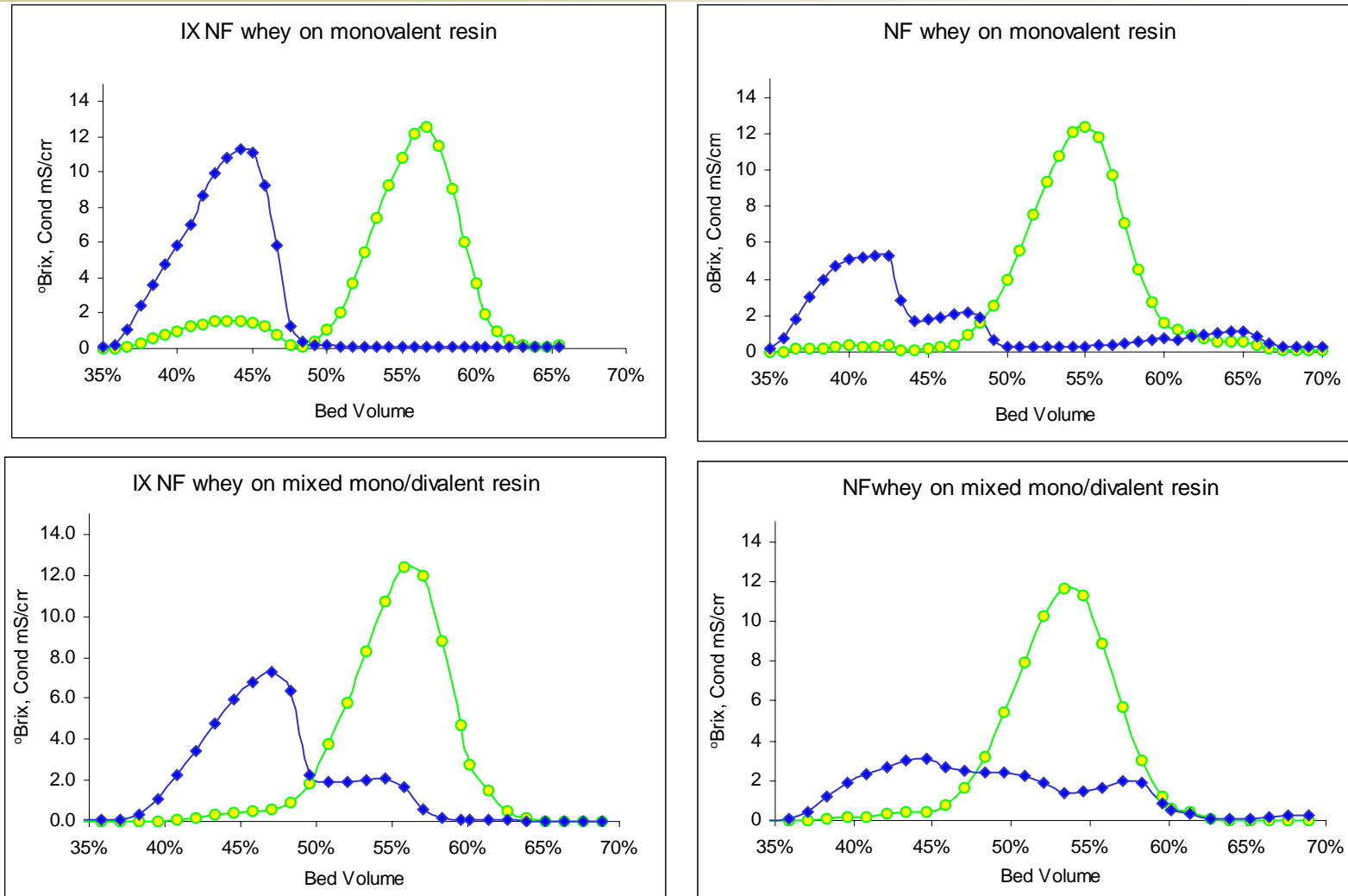


Figure 3. Ion chromatography of NF whey (containing  $K^+$   $Na^+$   $Ca^{2+}$   $Mg^{2+}$ ) and IX NF whey (containing  $K^+$   $Na^+$ ) on mixed "mono/divalent" resin or "monovalent" resins.

# Removal of calcium phosphates

- Whey permeate is supersaturated with insoluble calcium phosphate, interfering with chromatography, causing fouling problems and high lactose losses.
- Minerals can be efficiently removed by **Ion Exchange**:
  - $\text{Ca}^{2+}$  &  $\text{Mg}^{2+}$  by Cation Exchange
  - $\text{PO}_4^{2-}$ , citrate & lactate by Anion Exchange
- Whey permeate is also a rich source of **K, Na, Cl & water** which can be recovered by nanofiltration and used to regenerate the IX resin.

<b>Composition</b>	<b>Whey UF perm</b>
<b>Solids %</b>	<b>5.50</b>
<b>pH</b>	<b>6.10</b>
<b>Lactose %</b>	<b>4.70</b>
<b>Protein %</b>	<b>0.01</b>
<b>Ash %</b>	<b>0.53</b>
<b>Calcium ppm</b>	<b>375</b>
<b>Phosphorus ppm</b>	<b>275</b>
<b>Potassium ppm</b>	<b>1450</b>
<b>Sodium ppm</b>	<b>430</b>
<b>Chloride ppm</b>	<b>940</b>

# Ion exchange of NF retentate vs UF whey permeate

Table 2. Ion exchange of NF retentate & UF permeate on Amberjet 1200 resin.

Ion exchange Feed	°Brix	Cond mS/cm	pH	Minerals ppm			
				Ca	Mg	K	Na
NF retentate	18.81	6.59	6.17	769	233	2438	679
IX NF retentate	18.40	8.9	6.5	544	170	3088	1124
% removal				-29%	-27%	27%	66%
UF whey permeate	5.10	5.15	6.15	284	67	1352	340
IX whey permeate	5.00	5.9	6.3	9	9	1669	529
% removal				-97%	-87%	23%	56%

- NF retentate 18°B has competing regeneration from concentrated K<sup>+</sup>, Na<sup>+</sup>, desorbing Ca<sup>2+</sup>, Mg<sup>2+</sup> during elution.
- UF permeate 5°B not subject to regeneration from intrinsic K<sup>+</sup>, Na<sup>+</sup>
- These results shows that it is better for companies to ion exchange and concentrate prior to transport to central facilities.

# Ion exchange of whey permeate

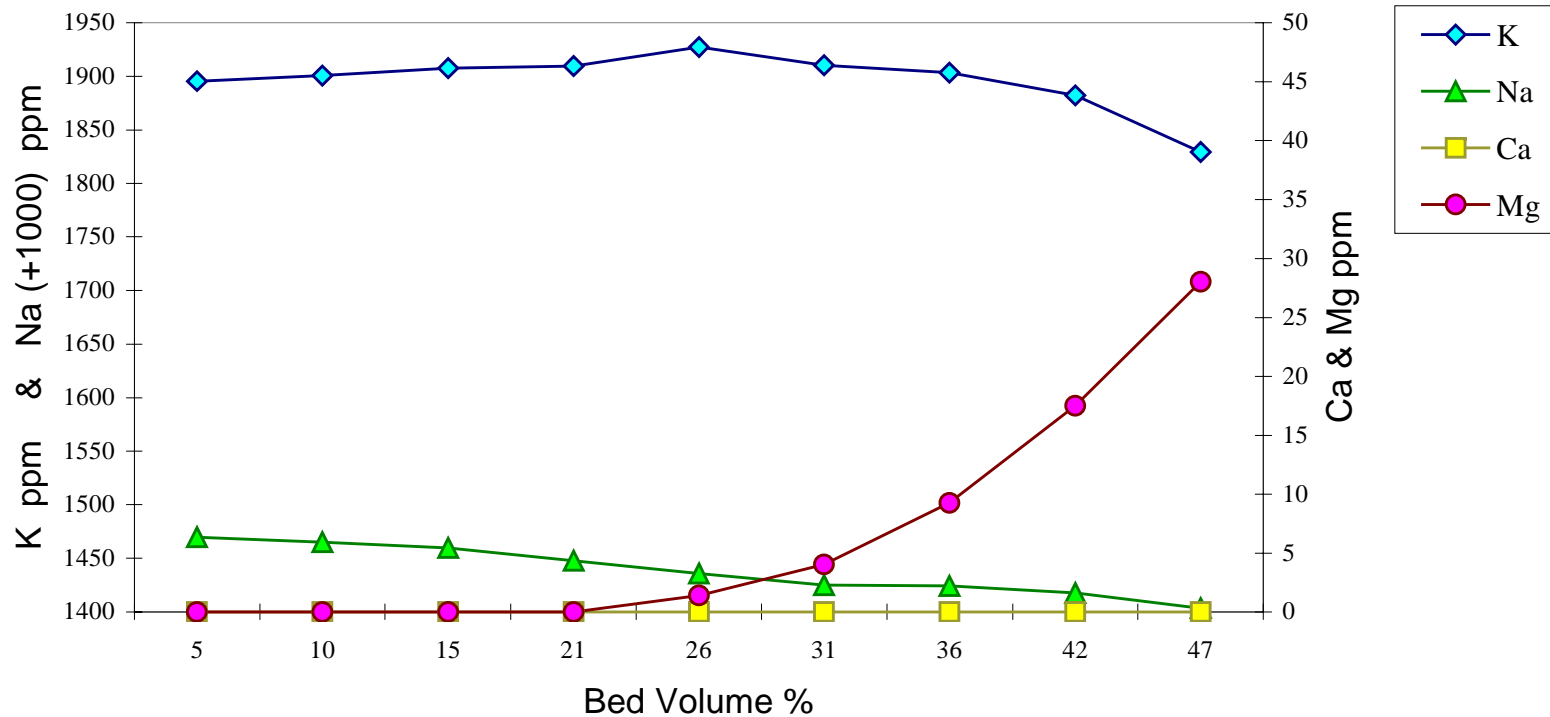


Figure 3. Mineral composition of ion exchanged UF permeate on Amberjet 1200 resin, showing breakthrough capacity for Mg ions.

NB. Sodium conc 400-475ppm; shown as +1000ppm

# Regeneration of IX resin

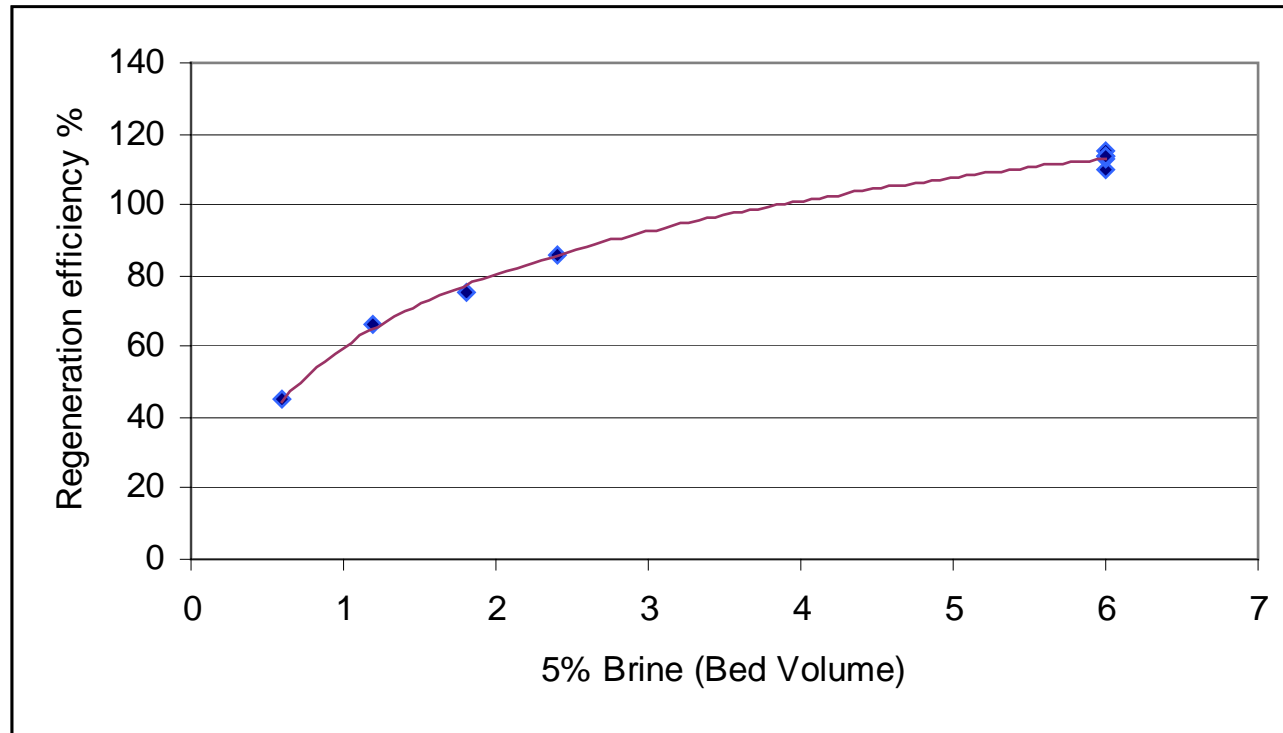


Figure 4. Regeneration of conditioned Amberjet 1200 resin with 5% brine, after treatment of 25 BV UF permeate.

# Whey salt for IX regeneration

- Monovalent salts in NF whey permeate can be recovered by reverse osmosis.

Table 3. Salt brine produced from processing 1000kg whey permeate.

Sample	Weight kg	Cond mS/cm	pH	°Brix	Lactose g/100g	Minerals ppm				
						Ca	K	Mg	Na	P
NF permeate	790	3.5	6.3	0.39	0.0	1.0	402	1.0	175	6
RO brine	47	29.6	6.6	4.11	0.5	1.0	8364	1.0	2244	275
	17 x									
NF permeate	790	0.9	6.5	0.43	0.0	5.0	280	1.0	97	7
RO brine	46	31.2	6.6	4.25	0.8	8.8	11807	2.0	3610	195
	17 x									

- Clean water is also produced, useful for resin washing and elution of the chromatography column.
- Salty cheese whey provides additional sources of salt.
- This avoids need to purchase salt or dispose to the excess salt to the environment.

# Recycling of spent ion exchange brines by nanofiltration

- NF membrane rejection can be manipulated by pH.
- $< \text{pH } 3$  calcium rejection increases from 60% to 95%
- Brine & calcium concentration causes charge shielding & reduce calcium rejection, hence diafiltration is required to optimise brine recovery.

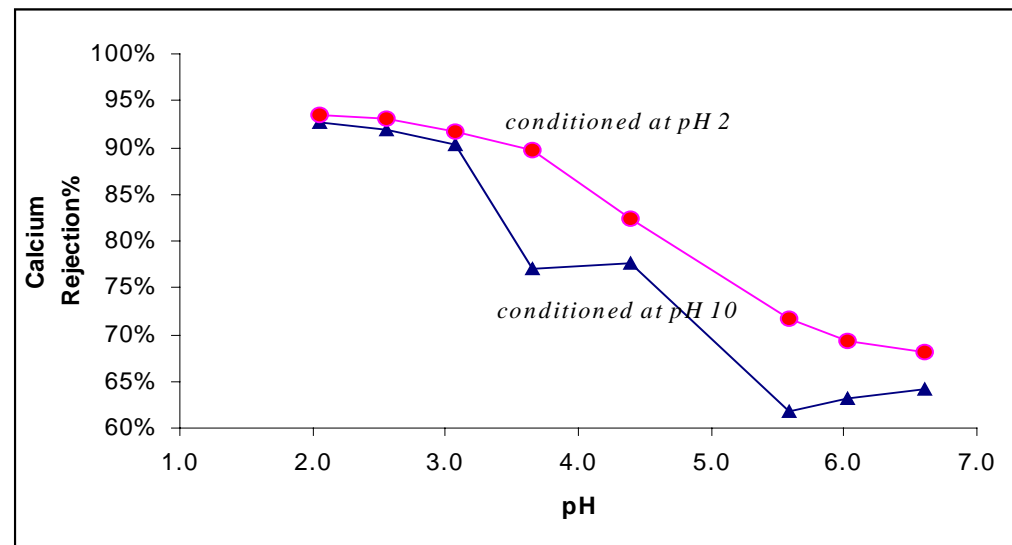
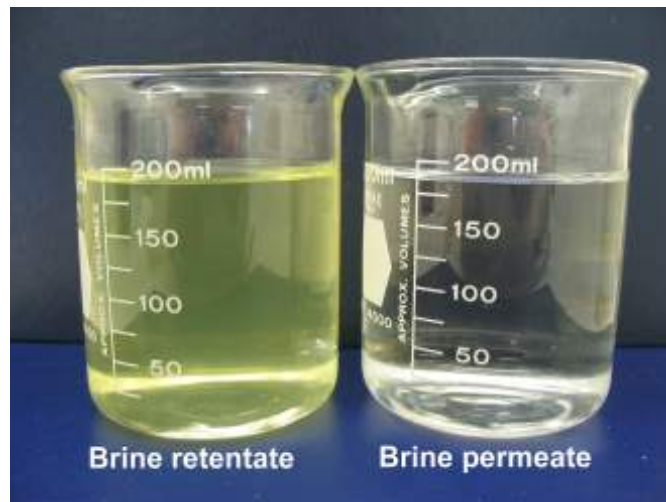
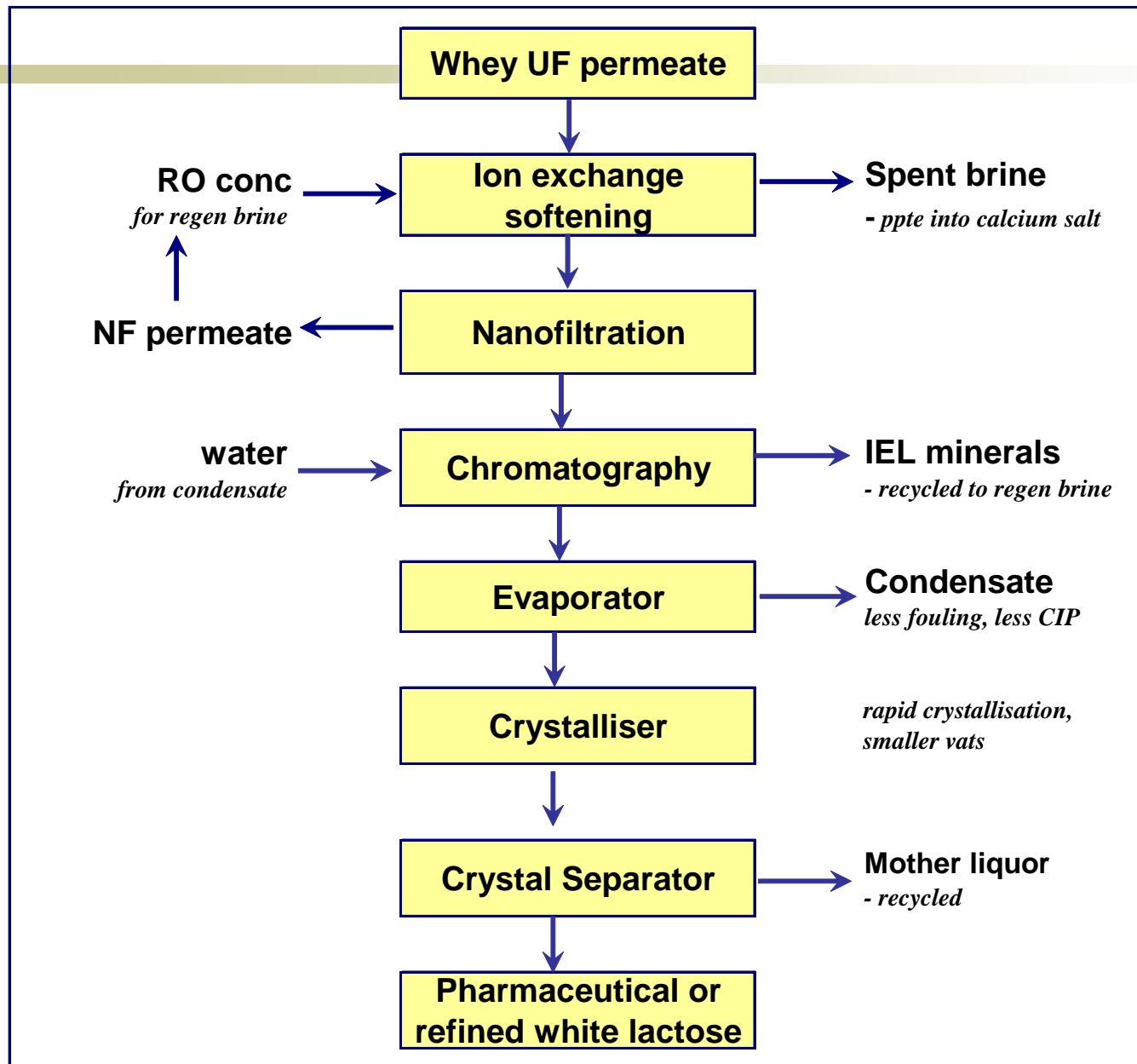


Figure 5. Calcium rejection of Desal 5 Nanofilter vs pH.

# IEL Lactose process



# The IEL pilot plant



- Computer monitored & controlled
- PLC valves, pumps & boiler
- Mass flow meter, pH & conductivity detectors

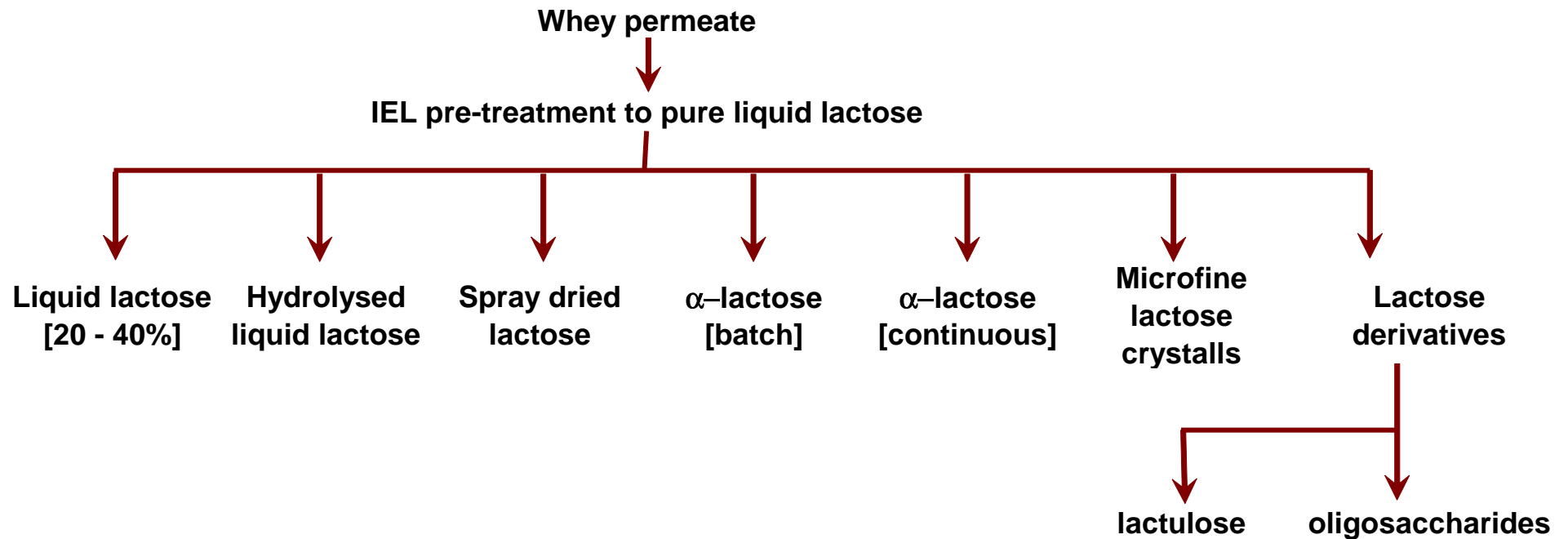
# Purification



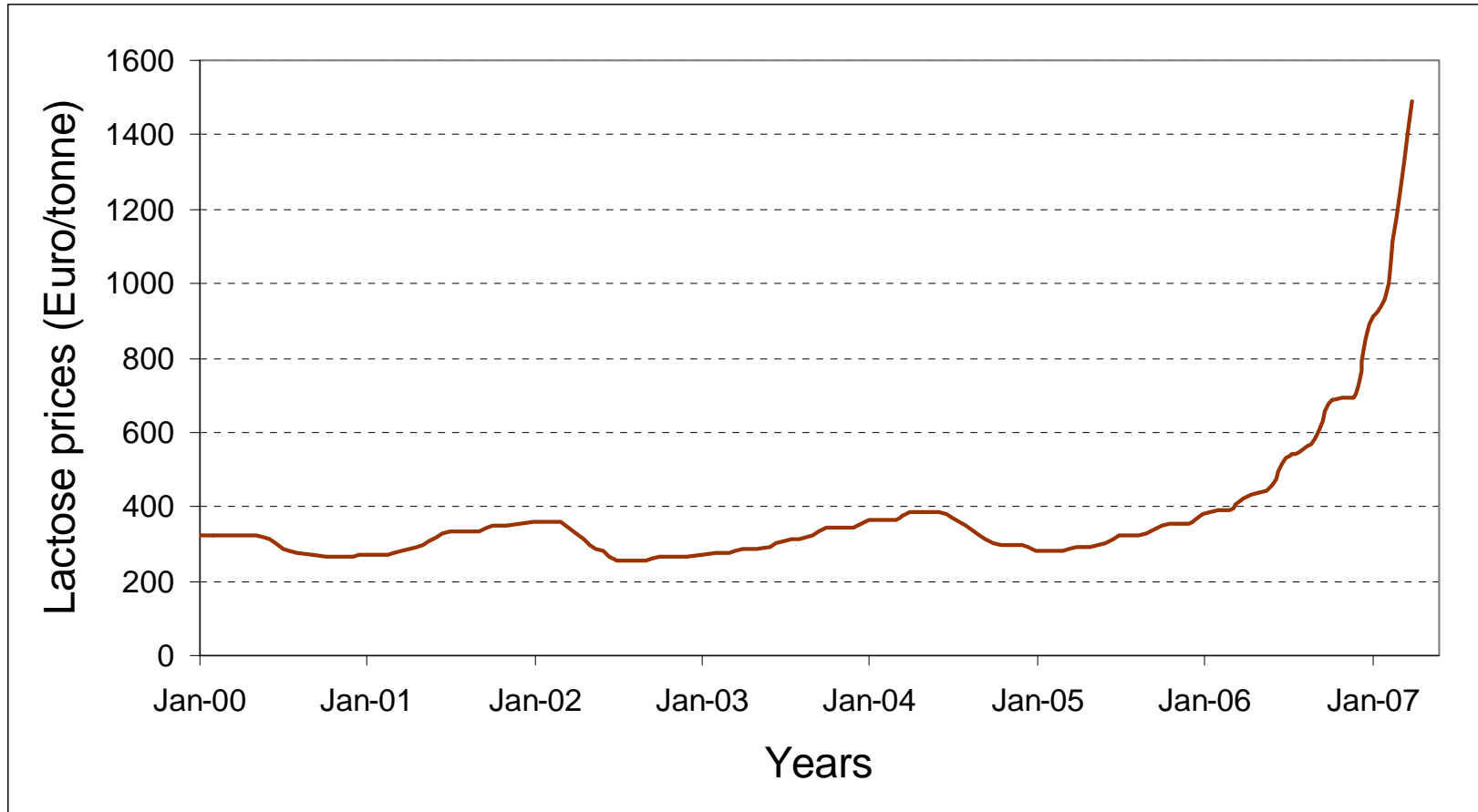
# Quality of IEL lactose compared to commercial lactose

Quality		IEL lactose	Pharma lactose
Total water	%	5.98	5.42
Ash	%	0.003	0.015
Protein at 255 nm	abs	0.005	0.007
Protein at 285 nm	abs	0.000	0.003
Acidity	ml/6g	< 0.1	0.2
Calcium	ppm	3.4	1.1
Potassium	ppm	0.0	34.2
Magnesium	ppm	2.9	1.1
Sodium	ppm	0.0	21.4
Phosphous	ppm	0.0	0.3
Sulphur	ppm	0.0	0.0
Clarity & colour at 400 nm	abs	0.000	0.002
Poured bulk density	g/ml	0.67	0.58
Instant solubility	sec/gram	33.0	77.3
Flowability (10mm funnel)	gram/sec	9.2	nil

# Options for IEL lactose



# Edible Lactose prices - Euro/tonne



Source: [http://future.aae.wisc.edu/data/monthly\\_values/by\\_area/23](http://future.aae.wisc.edu/data/monthly_values/by_area/23)  
(converted from data in US\$/lb from Gould 2007, using Apr07 conversion rates)

# The IEL lactose process

- Unit operations optimised & integrated with internal recycling to minimise waste and maximise efficiency:
  - Recycled whey brine for ion exchange regeneration
  - No need to purchase or dispose excess salt
  - Recovers purified water for elution and resin washing
  - No mother liquor unlike traditional processes
- Value added end-products:
  - Chromatography flexible, enabling optimisation of yield, purity & through-put to produce purified lactose at >95% yield.
  - Pure liquid lactose can be crystallised to refined edible, pharmaceutical, spray dried lactose; or converted into oligosaccharides; lactulose or other derivatives.
  - Soluble calcium fraction - suitable for dietary supplements

# Patenting & commercialisation

- IEL process patented in 1997 and granted in Australia (726559), New Zealand (502015), USA (6,475,390), Europe (1023121), pending in Japan & Canada.
- Licensed by Groupe Novasep in 2005 for worldwide industrial application in dairy industry.



# Acknowledgements

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