Lactose Fouling of Ion Exchange Technology

A thesis presented in partial fulfilment of the requirements for
the Masterate of Technology
In Bioprocess Engineering at
Massey University.

Antony Craig Smith

1999

ABSTRACT

Cheese whey is an ingredient used in infant formulae manufacture. Before addition, the cheese whey is fully demineralised using Ion Exchange (IE) technology. Investigation of the IE process revealed low lactose yields. The objective of this thesis was to provide an understanding of the mechanism causing these low yields. This understanding may be used to improve these yields during IE processing.

Two mechanisms were proposed for the removal of lactose during IE processing namely resin entrapment and lactose mutarotation adsorption. Investigations of the mechanisms were performed with both continuous and batch benchtop methods. Whey, lactose and DMSO/lactose feed solutions were employed with various resins. DMSO/lactose solution experiments were inconclusive in determining the mechanism. Whey and lactose trials revealed lactose adsorption occurred predominantly onto the macroporous anion resin (0.09 g-lactose/g resin) compared with the gel cation resin (0.04 g-lactose/g resin). In comparison the maximum lactose adsorption onto an alternative gel structured anion resin was shown to be 0.05 g-lactose/g resin. Absorption isotherm results were dependent on the supernatant concentration. The majority of lactose adsorbed onto both the macroporous and gel anion resins was recovered with six and three equivalent volumes of water, respectively.

The adsorption dependency on the resin structure and supernatant concentration coupled with the recovery of adsorbed lactose with water proved that the resin entrapment mechanism was causing the low lactose yields. In hindsight the DMSO results were also consistent with the resin entrapment mechanism causing the low lactose yields.

It is recommended that to reduce lactose losses during IE processing by 43%, gel structured anion resin (A847S) should be coupled in series with the existing gel structured cation resin (C100H). The gel anion resin would also halve the anion water requirements during lactose recovery flushing.

ACKNOWLEDGEMENTS

I would like to thank Dr Tony Paterson for his help and guidance throughout this project. Without him this Masterate would never have been completed. Thanks to the senior management at Waitoa for supporting this work. Additional thanks to Mike Howell for helping order laboratory equipment.

Thank you to Rob Boswell and Paul Signal from the Hautapu Protein Development group for their contributions in accessing the suitable laboratory equipment.

A big thanks to Tania Williams and the other Waitoa Laboratory Chemistry staff especially Mayson, Nick, Dick, Mary, and Carmel for helping me with the laboratory work. A special thanks also to Mayson and Nick for providing test methods for the most unusual analyses I could think of.

A collectively thanks to the Waitoa operators of the Ion Exchange plant for they time they provide me to discuss the Ion Exchange process and related issues.

A special thanks to Trevor Bell for helping me prioritise my workload properly and to Trevor and the other Engineering staff for helping me whenever I have general questions.

Special thanks to all those who put up or shared a beer with me during the stressed times.

Finally thanks to my family and my best friend, Shell (but don't tell Colleen), for their support to allow me to get to the position to undertake this project.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF EQUATIONS

GLOSSARY OF TERMS

IE	Ion Exchange – the whey demineralisation process investigated
COD	Chemical Oxygen Demand – a measure of effluent concentration
DVB	Divinyl Benzene – a monomer responsible for the crosslinking of the resin polymer matrix
BOD	Biological Oxygen Demand – a measure of effluent concentration
SNA	Solids Non Ash - Organic components remaining after evaporation of the solvent. Sample SNA calculated from the sample TS less the sample Ash (inorganic)
RO Water	Reverse Osmosis Water – water purified through a reverse osmosis plant
DMSO	Dimethyl Sulphoxide – a polar solvent used to inhibit mutarotation
NF Whey	Nanofiltered Whey – whey processed through a nanofiltration plant
F:R Ratio	Feed:Resin Ratio – the ratio of feed solution processed through a fixed resin volume size
TS	Total Solids – Organic and inorganic components remaining after evaporation of the solvent
BV	Bed Volume – a liquid volume equivalent to the volume occupied by the immersed resin

1.0 GENERAL INTRODUCTION

Anchor Products Waitoa manufactures dairy powders for human infant consumption. These powders utilise demineralised cheddar cheese whey as an ingredient. The cheddar cheese whey is fully demineralised using Ion Exchange (IE) technology. The demineralised whey comprises predominantly lactose solids. Smaller quantities of whey proteins are also contained in the demineralised whey.

During investigation of the IE process it was noted that:

1. Lactose yields were low.
2. Effluent concentrations (>3,000 mg COD/L) were greater than literature recommendations (1700-2200 mg COD/L).
3. The pH of the eluted demineralised whey varies considerably in pH during the process run, ranging from pH 11 to pH 4.
4. Protein yields were also low.
5. A white flocculant was observed towards the end of the processing run, when the pH was low. It was thought this contained precipitated protein.

It was thought that the lactose removal might contribute to the pH process variations and the high effluent concentration. It was also speculated that the pH variations might cause protein precipitation in the columns. Protein precipitation was considered outside the scope of this thesis. The objective of this thesis was to provide an understanding of the mechanism causing lactose removal during IE processing. An understanding of how the lactose was removed may be used to improve the process.

2.0 LITERATURE REVIEW

2.1 INTRODUCTION

Whey is the liquid residue from cheese and casein production. Whey comprises 80-90% of the total milk volume entering the process and contains 50% of the nutrients in the original milk: soluble protein (whey proteins), lactose (the bovine milk sugar), vitamins and minerals (Bylund, 1995).

Historically the whey has been treated as an effluent stream. Disposal of the whey has involved stockfood or land fertiliser applications. The fact that the whey proteins are highly nutritious, coupled with increasing pressure of anti-pollution legislation, has encouraged dairy companies to view whey as a potential product for human consumption (Potgieter *et al.*, 1986).

The major restriction with utilising whey for human consumption products is the salty flavour owing to the high mineral (ash) content (Hoppe & Higgins 1992, Delaney 1976, Potgieter *et al.*, 1986, Ennis 1984). From Table 1 it can be seen that the ash content in cheddar cheese whey is 8.4% of the total solids (TS). The major mineral distribution within the ash component is also shown in Table 1. Removal of these minerals and other ionic species from whey is called demineralisation. Applications for human consumption of demineralised whey powder include its addition to ice cream, confectionery, bakery and infant formulae products (Potgieter *et al.*, 1986).

Table 1: Typical Cheddar Cheese Whey Composition (McElligott, 1990)

	Typical Composition (Liquid whey)	Typical Composition (Dry Basis)
Fat	0.08%	1.3%
Protein	0.90%	14.5%
Lactose	4.70%	75.8%
Ash	0.52%	8.4%
Calcium	530 mg/L	0.052%
Magnesium	80 mg/L	0.008%
Potassium	1500 mg/L	0.147%
Sodium	370 mg/L	0.036%
Phosphorous	550 mg/L	0.054%
Chloride	910 mg/L	0.089%
Total Solids	6.20%	100.0%

Two main technologies are available for achieving high levels of whey demineralisation; namely Ion Exchange (IE) and Electrodialysis (Potgieter *et al.*, 1986). When highly demineralised whey is required, the objective is to maximise mineral removal, while minimising protein and lactose losses. The economics of the two options in other countries, have been reported (Delaney 1976, Houldsworth 1980, Potgieter *et al.*, 1986). It was shown that the economics of Ion Exchange is greatly effected by the cost of regenerant chemicals. New Zealand economic evaluation revealed IE technology the best option for highly demineralised whey production (Berry & Pothecary 1979, Ennis and Higgins 1982, Marshall 1979, NZDB Report 1990).

2.2 IE THEORY

The theory of IE has been detailed by many authors (Applexion *unpublished*, Delaney 1976, Delaney & Donnelly 1976, Delbeke 1972, Downes *et al.* 1985, Ennis 1984, Finer 1993, Grandison 1994, Hermann 1985, Herve 1974, Hoppe *unpublished*, Hoppe & Higgins 1992, Horton *unpublished*, Houldsworth 1980, Jonsson 1987, Kunin 1974, Mason *unpublished*, McElligott 1990, McNulty 1997, Potgieter *et al* 1986, Potgieter *et al.* 1988a, Potgieter *et al.* 1988b, Shelley 1997, Societe Des Prouits Nestle *patent* 1985). A summary of the theory of IE follows.

2.3 IE ORIGINS

The discovery of switching ions in solution was made in 1845 in studies on soil enrichment. The first industrial application of this technology was made in 1905, for the softening of water by zeolites. In 1935, the first synthetic IE polymers were prepared by Adams & Holmes (Hoppe & Higgins, 1992). In 1942, D'Alelio created sulphonated crosslinked polystyrene, followed in 1949 by the corresponding polystyrene anion exchange resin by M^{c}Burney. The polystyrene resins are chemically very stable and are almost ideal ion exchangers from every point of view. Consequently they have now become almost universally used (Arden, 1968).

IE technology is predominantly used for demineralisation and softening of boiler feed water and process water throughout the chemical process industries (M^{c}Nulty, 1997), but during the last 30 years it has also become of interest for the demineralisation of whey (Jonsson, 1992).

2.4 IE RESINS

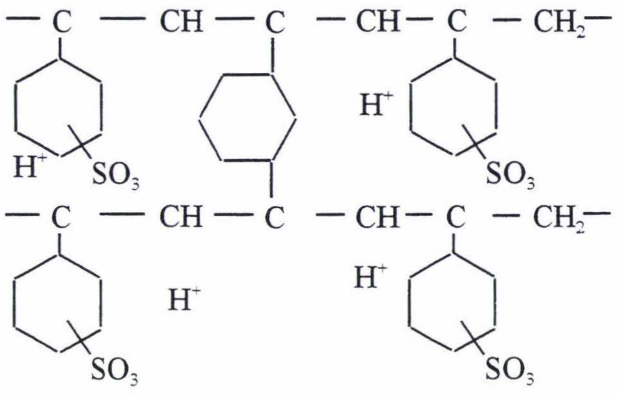
IE is a fixed bed technique involving the use of insoluble resins, (held within a cylindrical column) which have a discrete capacity for adsorption of minerals. The resins are manufactured in the form of small beads (0.4-1.0 mm in diameter). This size is to maximise strength and surface area, and minimise diffusion resistance. The resins are usually made of styrene cross-linked by divinyl benzene (cross-linked polystyrene) with incorporation of charged functional groups. These charged sites may be occupied by anionic or cationic, strong or weak functional groups. For whey demineralisation the following resins are generally used (Arden, 1968):

2.4.1 CATION RESIN

If linear polystyrene is sulphonated, it forms linear polystyrene sulphonic acid, which is water soluble and useless as an ion exchange material. The essential step in the D'Alelio process was to co-polymerise divinyl benzene with the styrene so as to give a crosslinked structure. The hydrocarbon beads are then insoluble in organic solvents, although readily swelled by them, while the sulphonated product is completely insoluble in water although swollen and freely permeable by it.

The sulphonic acid cation exchange resins (Figure 1) made by different manufactures are chemically similar to each other. There are differences in the molecular structure of the skeleton, which affects the physical strength, and the rate of reaction of the different products.

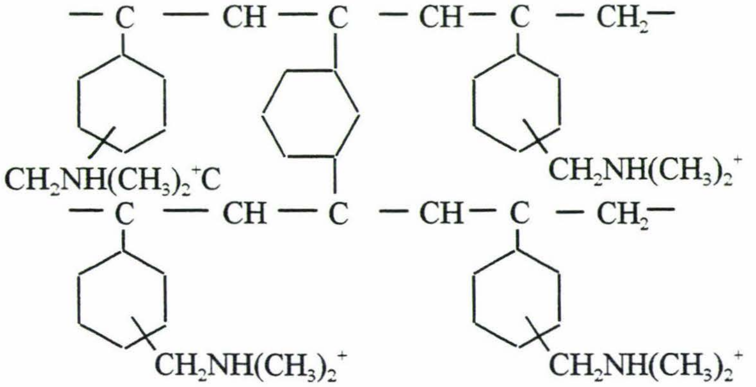
Figure 1: Sulphonic Cation Resin Structure (Arden, 1968)



2.4.2 ANION RESIN

The case of polystyrene anion exchange resins is somewhat different. The resins are prepared by chloromethylating and aminating the hydrocarbon beads. When the amine used in the resin manufacture is a primary or secondary one, the result is a secondary or tertiary amine resin respectively and these products are weakly basic in nature (Figure 2). The number of available amines are very great, and consequently there are commercially available a large number of different weak base resins. While the available resins are broadly equivalent to each other in their functions, they are nevertheless quite different chemically. Therefore the weak base anion resins have widely varying capacities and rates of reaction.

Figure 2: Weakly Basic (tertiary amine) Anion Exchange Resin (Arden, 1968)



Irrespective of the method of manufacture, the ion exchange resins are all crosslinked gels, containing a high proportion of water, which is essential part of their structure. The water can be removed by heat or vacuum, whereupon the molecular chains lie closer together, and the beads shrink. On being re-wetted, the beads swell, the ions contained in them becoming hydrated. If the resin structure were not constrained by crosslinking, the swelling would be infinite, therefore the product would be soluble. The degree of swelling is related precisely to the crosslinking. As the crosslinking increases, the water content decreases, until at 8% crosslinking, which is the optimum for most industrial purposes, the beads contain about 50% water. They are hard and tough, and almost infinitely durable. Materials crosslinked to more than 25% are extremely hard and brittle, while the water content has fallen too low for the satisfactory function of the ion exchange processes.

2.4.3 IE RESIN STRUCTURE

The structure of crosslinked polymers is of greatest importance in connection with the mobility in the resin of large organic ions. The large organic ions can become entangled with the tight regions of the polymer network. During the polymerisation of styrene with divinylbenzene two major forms of inhomogeneity are introduced.

2.4.3.1 Chain Entanglement

The crosslinked structure is a series of three dimensional networks, each of which grows independently of the others from a single starting point, and it is clear that the resulting networks must be intertwined. It is possible that a material could be made with all the characteristics of a normally crosslinked resin, while containing no cross links at all, but purely a high degree of entanglement. Entanglement is controlled by the addition of original monomer to finished beads and repolymerising.

In addition to producing extra entanglement, it is equally possible to arrange for deliberate disentanglement during manufacture. This is achieved by addition of a miscible polymer, which is compatible with the final polymer, but takes no part in the reaction. Thus the polymer chains are forced to grow around the miscible polymer. On completion of the reaction the miscible polymer is then removed.

The process of disentanglement by means of an unreactive polymer can be carried to the extent that the solid polymer chains are stretched, and rigidly locked. On removal of the solvent, the structure does not collapse, but contains genuine holes of up to 1000/ diameter, which forms water channels. These highly disentangled structures are known as "macroporous".

2.4.3.2 Differential Polymerisation

A second effect, completely independent of the entanglement process, is due the to the fact that in divinylbenzene (DVB) and styrene, three different polymerisations are possible:

1. Styrene-Styrene
2. Styrene-Divinylbenzene
3. Divinylbenzene-Divinylbenzene

The reaction rates of these three processes increase in the order given above, therefore polymer chains begin rich with DVB and grow more lightly crosslinked. Therefore the crosslinking of the insoluble beads is not homogeneous, but has regions of high crosslinking and regions of low crosslinking.

As large charged organics pass through the resin structure to exchange sites in the interior of the beads, they must pass regions of excessive crosslinking. The organic ions become entangled (fouled) in the crosslinked regions.

2.4.3.3 Non-Fouling Resins

2.4.3.3.1 Macroporous

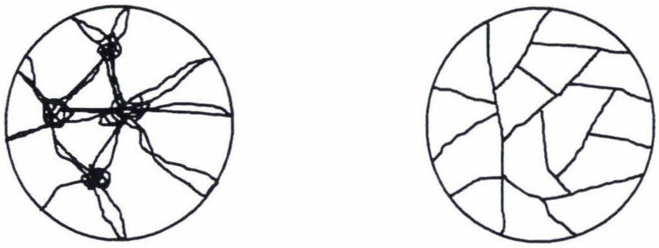
In an attempt to overcome this fouling, macroporous resins were designed and employed. The drawback with these resins was that the capacities are lower than those of the corresponding DVB gel materials. The method of production also led to structures that held the simple inorganics more firmly. This lead to increase regenerant chemical usage for the same level of demineralisation.

2.4.3.3.2 Isoporous

Following the problems with the macroporous resins, the opposite method of overcoming the fouling problem was studied. If fouling was due to irregularities in crosslinking, it could be prevented by making the crosslinking homogeneous (Figure 3). In the preparation of the anion resin the method was changed so that all the crosslinking required occurred in the chloromethylation stage and no divinylbenzene was used at all. This method was a controlled reaction and led to the formation of resin beads with substantially uniform networks, free from tight regions. Resins produced in this way have become known as "isoporous". Resin manufacturers now term these resins "gel" structured.

The isoporous resins have, in British practice at least, superceded the macroporous materials for demineralisation purposes. Macroporous resins are still used for organic traps for which their reduced demineralisation capacity is not disadvantageous.

Figure 3: Comparison of Standard Resin Types (Arden, 1968)



Macroporous Isoporous

The major factors determining the effectiveness of an IE resin are the size, valency and concentration of the ionic species to be exchanged. These factors influence the capacity and selectivity of the resin. The degree of crosslinking of the resins dictates the diffusion rate of the counter ions to the internal sites.

With strong acid/base charged functional groups, ions of high valency diffuse more rapidly in dilute solutions and ions of lower valency diffuse more rapidly in concentrated solutions. By contrast, weak acid/base exchangers have a strong affinity for the hydrogen ion/hydroxyl ion counter ions. This has an effect on the choice of resin. The expensive nature of the regenerant chemical, NaOH, coupled with the strong affinity for the hydroxyl ion ensure it is more economical to use a weak anionic resin for whey demineralisation. It is reported that for the treatment of whey, a strongly acidic cation exchange resin and a weakly basic anion exchange resin are normally used (Delaney (1976), Societe des Produits Nestle (1985), Finer (1993), Hoppe & Higgins (1992)).

2.5 MECHANISMS OF IE

Whey is normally passed through the cation-exchange bed first, where cations in the whey exchange for hydrogen ions on the resin. In the anion exchange bed, anions displace hydroxyl ions, which then react with the hydrogen ions to form water. Both reactions are represented as equilibrium reactions, the position of which depends on ion concentrations in the liquid and on the solid phase of the resin (Finer, 1993). The exchange of ions between resin beads and the surrounding solution is best represented by the following chemical reactions:

Cation exchange:

$\text{Resin-R-H}^+ \quad + \quad M^+ \quad A^- \quad \square \quad \text{Resin-R-M}^+ \quad + \quad H^+ A^-$ **Eq. 2.1**

Regenerated Cation Resin Raw Whey Exhausted Resin Acidified Whey

the acidified whey is then feed to the anion column where the following reaction proceeds:

Anion exchange

$\text{Resin-R-OH}^- \quad + \quad H^+ \quad A^- \quad \square \quad \text{Resin-R-A}^- \quad + \quad OH^- H^+$ **Eq. 2.2**

Regenerated Anion Resin Acidified Whey Exhausted Resin Water

where	R	represents the insoluble resin exchange site
	M^+	represents a mineral cation
	A^-	represents an anion

From the mechanism reactions it can be seen that:

1. after passing through the cation column, the whey is acidified, this causes it to have a low pH (1.7-1.9) and a high conductivity.
2. after passing the decationised whey through the anion column the whey is diluted, as water has been added and no minerals exist. Therefore the pH and conductivity of the fully demineralised whey should be approximately neutral pH and zero conductivity.
3. if the cation resin exhausts first the acidified whey production ceases, increasing the cation pH, and leading to the whey existing the anion column having an increasing pH and conductivity.
4. if the anion resin exhausts first the acidified whey will not be neutralised, leading to the whey existing the anion column having a decreasing pH and increasing conductivity.

As we will see later these observations are used to understand the chemical processes occurring with the pressurised vessels.

2.6 ION SELECTIVITY

The equilibrium constant of the IE mechanism reactions vary depending on the ion species, which determine the selectivity of the IE process. The overall charge on the ion and the molecular or ionic mass are the primary determining factors of selectivity, but selectivity is also related to the degree of hydration, steric effects and environmental factors such as pH or salt content (Grandison, 1994).

For the typical demineralisation of whey the selectivities for strong cations are:

$$Fe^{3+} > Al^{3+} > Ca^{2+} > Mg^{2+} > K^{+} > Na^{+} > H^{+}$$

Similarly the weak anion selectivity is:

$$OH^{-} > SO_4^{2-} > Br^{-} > Cl^{-} > F^{-} > CH_3COO^{-} > HCO_3^{-}$$

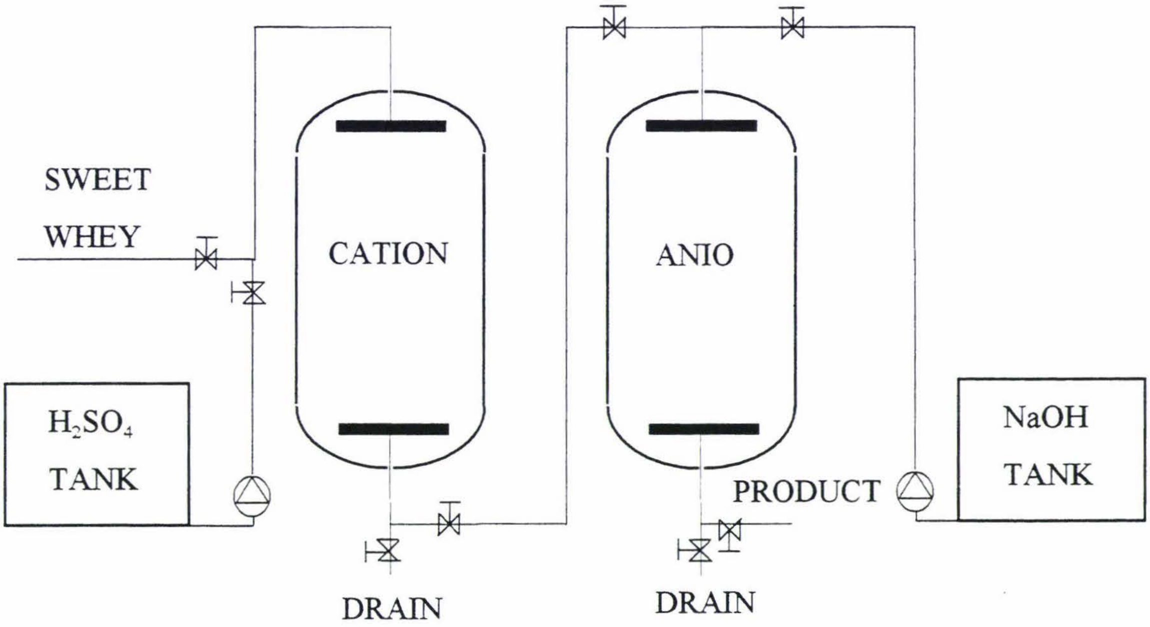
2.7 IE WHEY DEMINERALISATION PROCESS

2.7.1 PROCESS REACTORS

Column systems are the most widely employed (see Figure 4). Columns must be designed for the application to:

1. avoid channeling by the fluid
2. prevention of excessive bed pressure in the resin
3. good distribution of fluid across the bed
4. hygienic construction and operation

Figure 4: IE Process (Herve, 1974).



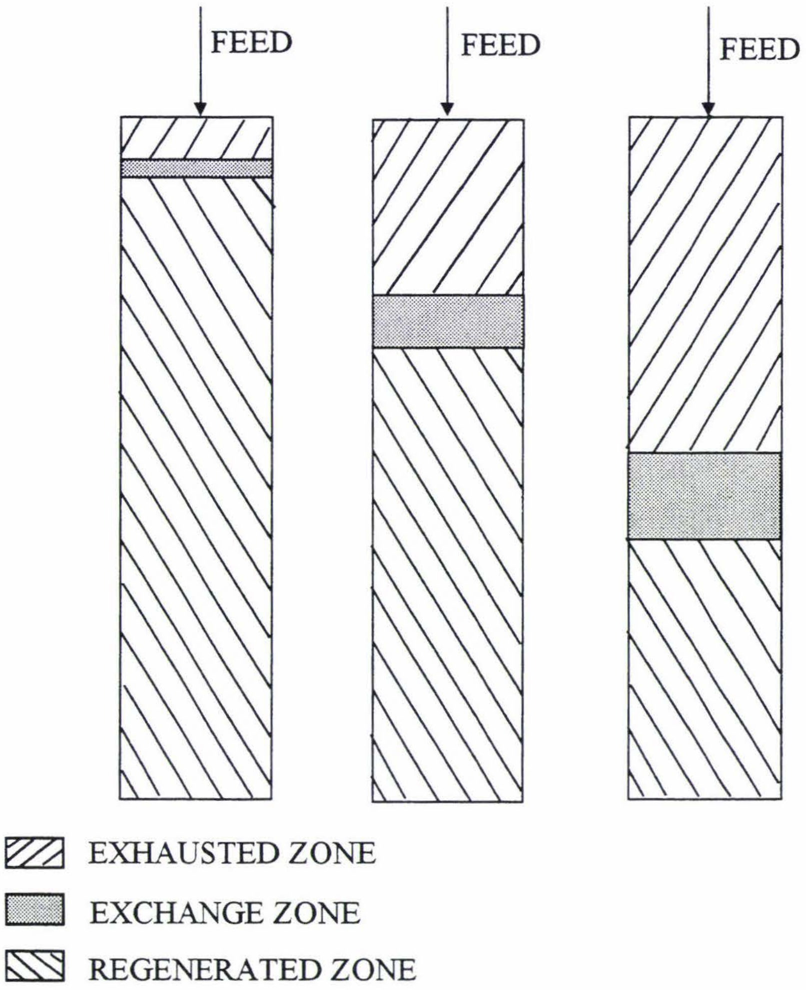
2.7.2 EXCHANGE ZONE

Three zones exist in the IE columns during processing (see Figure 5). These zones are:

1. Exhausted Zone - is the sector from the top of the column that no longer has exchange capacity
2. Exchange Zone - immediately below the exhausted zone, and is where IE is proceeding
3. Regenerated Zone - is the "fresh" part of the column. It is immediately below the exchange zone

The Exchange zone increases in size as it moves through the column. The depth of the zone is determined by mass transfer considerations between the fluid and the IE beads. These mass transfer considerations therefore determine the velocity of the fluid and the height of the column. This limits the practical column height and the whey processing velocity.

Figure 5: Regeneration Zone (Ennis, 1984).



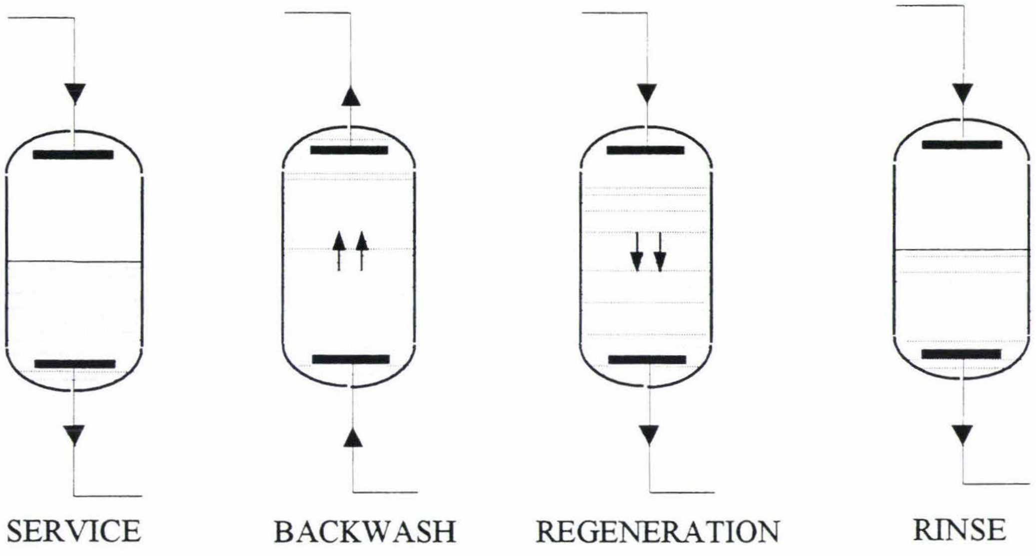
2.7.3 PROCESS OPERATION

When the capacity to exchange ions is exhausted, the adsorbed minerals have to be removed from the resin and the resin regenerated before it can be used again. During regeneration, the high concentration of hydrogen (strong acid) or hydroxyl ions (strong alkali) forces the reactions shown in Equations 2.1 and 2.2 to the left. This displaces the ions that have been removed from the whey (Finer, 1993).

The IE operation therefore involves four processes (see Figure 6):

1. Processing - loading and operation; usually 5-10 bed volumes
2. Backwashing - flushing out residual whey; usually 2 bed volumes
3. Regeneration - with regenerant chemicals; 3-10 bed volumes
4. Rinsing - to remove residual regenerant chemicals; up to 10 bed volumes

Figure 6: Process Steps (Ennis, 1984).



Each process step is critical to the efficient operating of the IE technology. Investigation of each step follows.

2.7.3.1 Service

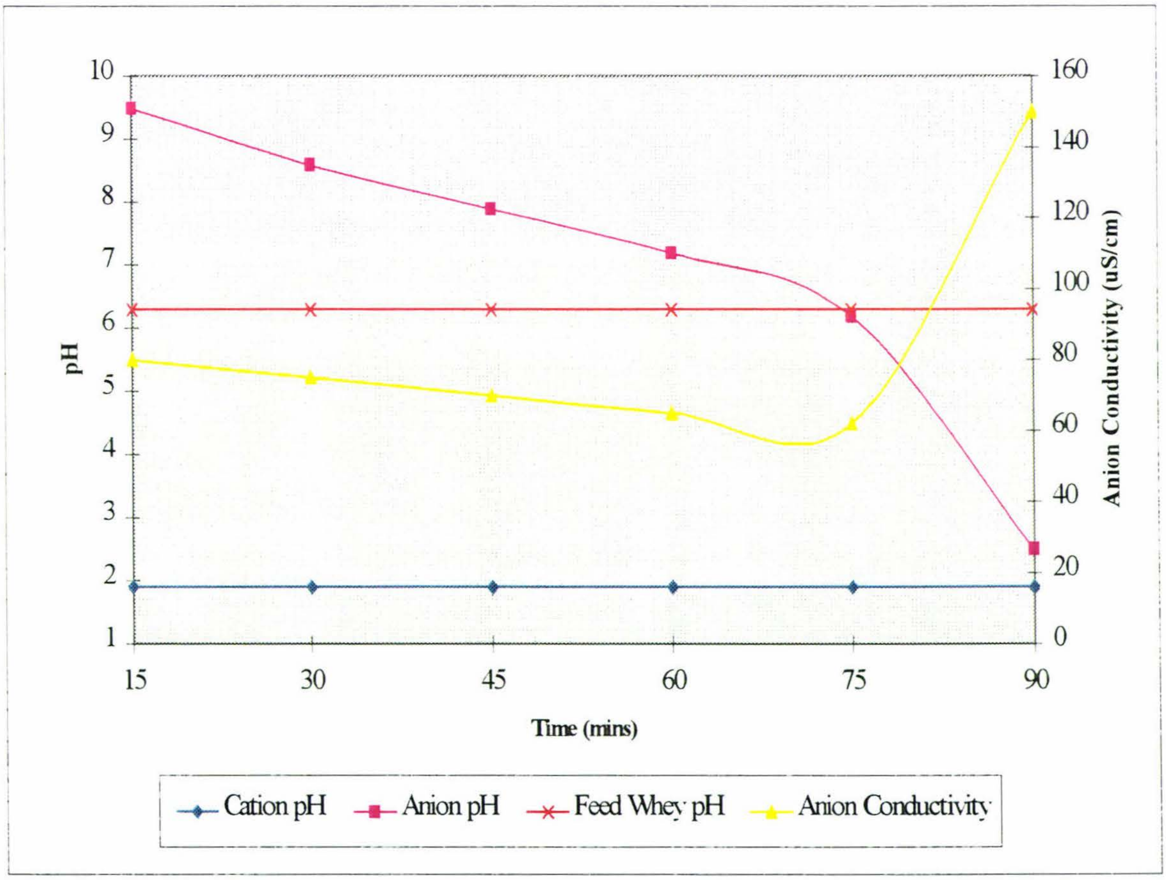
Conventional IE demineralisation of whey is carried out by passing whey over a bed of cation resin and anion resin that have been regenerated (i.e. the functional groups are in the H^+ and OH^- forms).

The primary measurements to follow the characteristics of the processing are pH and conductivity. The former measurement indicates the H^+ and OH^- concentration magnitude.

The conductivity measurement is used to measure the ion concentration in the stream. The H^+ and OH^- ions will effect conductivity measurements. Therefore conductivity measurements can be used to indicate mineral concentration provided the pH adjustment is accounted for.

The typical trends for a processing cycle of cheddar cheese whey is shown below in Figure 7 (Delaney and Donnelly, 1975). Cation ions in the whey feed are exchanged for H^+ ions as shown in Equation 2.1. This continues until the cation column is exhausted. Therefore the cation pH will consistently be strongly acidic. If this column begins to exhaust then the pH will rise.

Figure 7: IE Demineralisation of Cheddar Cheese Whey (Delaney and Donnelly,1975).



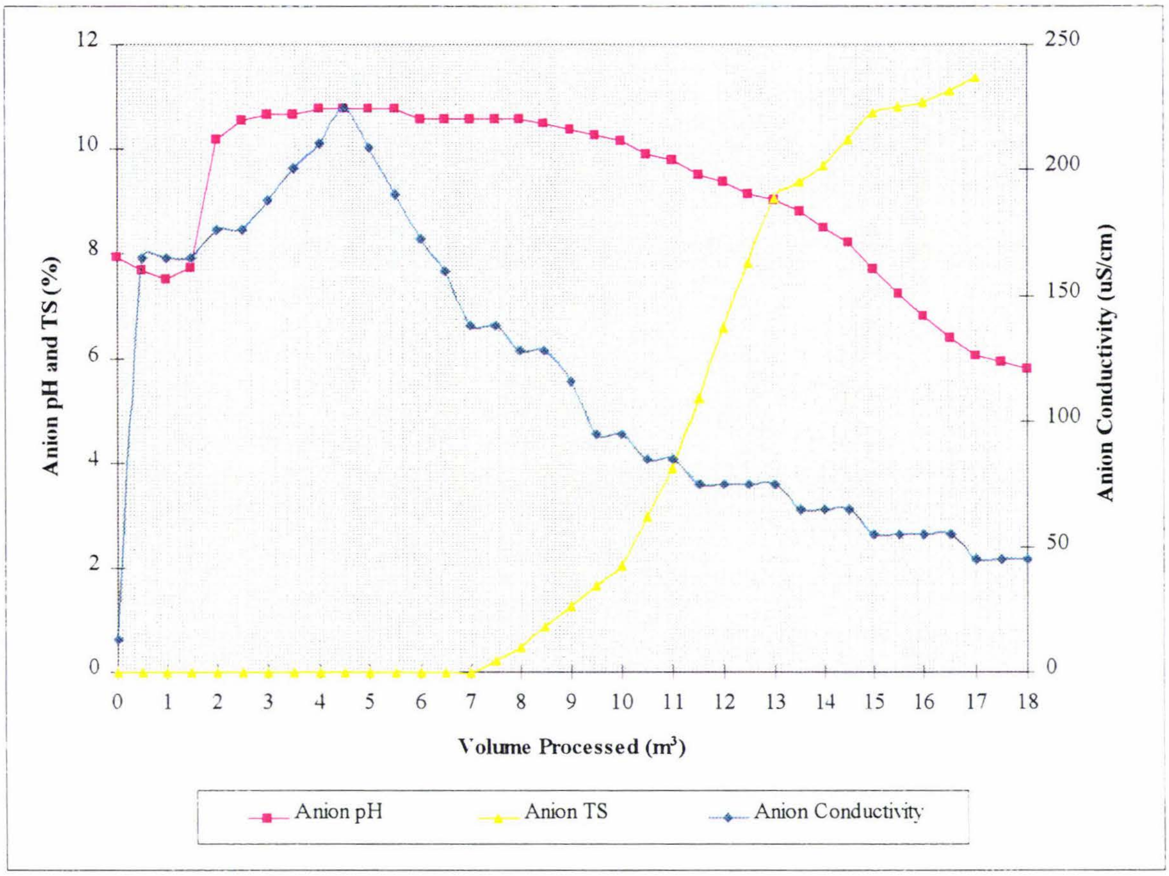
The acidified whey from the cation column is then passed through the anion column. The anion resin removes the anion minerals still in the acidified whey. The H^+ ions and the OH^- ions then react to form water as described in Equation 2.2. If the anion column exhausts the eluting pH will drop to the pH of the feed into the anion column. Therefore the pH of the exhausted anion column will approach the cation whey pH. At the same time the conductivity of the whey eluting from the anion column will rise. These trends are in agreement with Figure 7. Therefore the anion exhausts before the cation column in this process cycle.

In the initial stages of processing the anion elution shows a high pH. The high initial pH is speculated to be due to excess caustic eluting. This occurs as the anion resin beads swell with adsorption of the anion minerals in the feed whey. The swelling of these beads frees caustic trapped between the regenerated beads.

IE processing of whey permeate is shown in Figure 8 (Finer, 1993). These process trends are recorded from fully regenerated IE columns. With the IE columns fully regenerated and flushed with deionised water, the demineralised product would be of low conductivity and neutral pH. As the whey permeate is feed into the columns the anion resin beads will absorb the anion ions. This would cause them to swell and release trapped caustic. Therefore it is expected that the pH and hence conductivity would initially increase. As swelling subsides the trapped caustic eluted would reduce. This would show in a gradual reduction in the pH and conductivity. These trends are in agreement with the whey permeate process results shown in Figure 8 (Finer, 1993).

The only trend not accounted for is the delay before anion TS elutes. This can be due to the adsorption of lactose onto the anion resin. Eventually the sites causing lactose adsorption become full. The eluting lactose gradually begins to increase.

Figure 8: Permeate Demineralisation (Finer, 1993)



2.7.3.2 Back-washing

Water is passed up from the bottom of the column. This back washing removes suspended substances deposited on the top and interior of the resin layer during the exchange step. Back washing also loosens up the compressed resin layer after exhaustion. The resin layer should expand 50-80% during back washing (Mitsubishi, 1986). Due to economical pressures this flush is generally added to the processed product. As a consequence this step is also termed product recovery.

2.7.3.3 Regeneration

Regeneration can be carried out co-currently or counter-currently, with the regenerant flowing down or up the column, respectively. For the cation exchanger, counter-current regeneration is often used. This system lowers the consumption of regeneration chemicals by as much as 30-40%, but requires a more complicated vessel construction to prevent bed expansion (Jonsson, 1987).

Complete regeneration is not used industrially as it is an inefficient method of regenerant use. Rather partial regeneration, whereby the resultant resin capacity is less than the maximum possible, is used. The quantity of regenerant used is termed the regeneration level, and is recorded as the mass of regenerant per unit volume of resin. As the regeneration level is increased, the percentage of regenerated resin also increases, but the ratio of regenerated resin to the actual amount of regenerant utilised (regeneration efficiency) decreases. Hence, operation at a lower regeneration level makes for more efficient use of regenerant chemicals, but if the regeneration level is too low, significant leakage of ions in the product will result (Mitsubishi, 1986).

Strong acid cation columns used in the demineralisation of whey are generally regenerated with 4% concentrated hydrochloric acid. In New Zealand a more economic alternative, 4% concentrated sulphuric acid, a by-product of the fertiliser industry, is employed. Use of concentrated sulphuric acid requires redesign of the IE system. With the exhausted resin loaded with high quantities of calcium, precipitation of calcium sulphate is possible under the conditions. To avoid this precipitation the concentrated sulphuric acid is initially dosed at 1% strength to remove the majority of the calcium. After a predefined period the concentration of the acid is increased to 4% to finish regenerating the columns effectively.

As previously mentioned weak acid/base exchangers have a strong affinity for the hydrogen ion/hydroxyl ion counter ions. This causes the weak base exchangers to require less regenerant chemicals and be more cost effective. Strong acid/bases require large excesses of regenerant chemicals, in the order of 300%. The expensive nature of NaOH for regenerating the anion column prohibits the excessive regeneration required for the strong base anion. Therefore weak base anions are used for whey demineralisation.

It has been recommended that regeneration contact time be kept to a one hour minimum to ensure that sufficient time for removal of polymerised silica and organic matter is allowed (Delaney, 1976).

2.7.3.3.1 SMR Process

Recently an alternative to the conventional IE process was developed in Sweden (Jonsson, 1992, Potgieter *et al.*, 1988a, 1988b, 1988c). In this process the cations in the whey are exchanged for ammonium ions (NH_4^+) and the anions for bicarbonate (HCO_3^-) ions. The regenerant chemical, ammonium bicarbonate, a thermolytic salt is recovered from the whey stream. This is accomplished by evaporating the ammonium bicarbonate and purifying through gas adsorption technology.

This process is only economically feasible in countries where large fines exist for effluent manufacture. In the present environment this system is not economically feasible in New Zealand.

2.7.3.3.2 Thermal Regeneration

Thermally regenerateable IE resins have been developed (Parrish *et al.*, 1979 and Delbeke 1979). Work has been performed on whey permeates. Processing of whey is prohibitive due to the denaturation of the whey protein at the high temperatures required for regeneration.

2.7.3.4 Rinse

Typically two resin volumes of water are used to flush the bulk of the regenerant remaining in the column after regeneration (Mitsubishi, 1986). This volume may be recovered to be used at the start of the next regenerant solution cycle (Shelley, 1997). Rinsing with water is continued until the regenerant is thoroughly washed out. The water is then drained down to the surface of the resin, and demineralisation can recommence, with the feed stream being passed down onto the column. The effluent from the column gradually becomes richer in product.

2.8 PROCESSING VARIABLES

There are a large number of variables that can influence the operation of an ion exchange process. The degree to which these can be controlled depends of the individual plant. The most important of these variables are described below.

2.8.1 COLUMN LENGTH

Column length has a significant effect on plant performance, although it cannot be altered once a plant has been built. When an ion exchange column is in operation, there is an 'exchange zone' where ion exchange is occurring. Behind this zone, the resin is effectively exhausted, and in front of this zone the resin is fully regenerated. The significance of this with respect to column length is that the zone length increases approximately with the square root of time (Horton International, unpublished data), and once this zone reaches the bottom of the column, 'break-through' occurs and regeneration is required.

Although this consideration suggests longer columns give better resin utilisation, longer columns also have a larger exhausted zone during operation which effectively represent wasted resin inventory (Horton International, unpublished data). The minimum resin depth is determined by the need to obtain a good flow distribution without channeling occurring, while the maximum allowable pressure drop normally determines the maximum depth across the bed. If the pressure drop becomes too high, then the resin may be subject to mechanical damage.

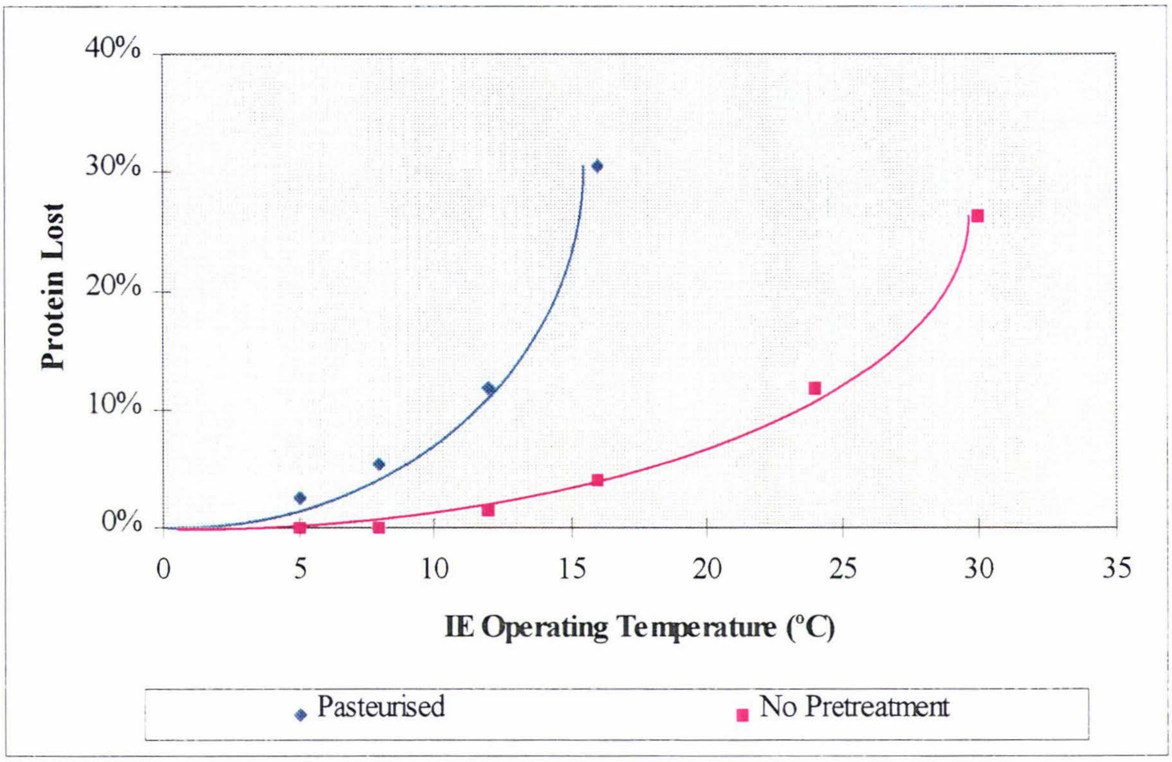
2.8.2 FLUID VELOCITY

The flow rate and the area for available fluid flow determine fluid velocity. High velocities have the benefit of increasing the rate of product throughput and therefore the frequency at which a column can be cycled. However, high velocities also increase the length of the exchange zone, which effectively decreases resin utilisation. Fluid velocity also influences pressure drop across an ion exchange column. This is particularly significant with fluids containing a high concentration of suspended solids (Finer, 1993).

2.8.3 TEMPERATURE

Operating at low temperatures reduces bacterial growth and improves resin life, but the whey stream becomes more viscous, resulting in a larger pressure drop. Temperature also effects the protein loss in the operating column. Operating between 5 and 12°C results in minimum protein loss when processing raw cheese whey (refer to Figure 9). Operating at low temperatures without prior pasteurisation minimises protein loss (Delaney, 1976).

Figure 9: IE Operating Temperature Protein Losses (Delaney, 1976).



2.8.4 CONCENTRATION

Whey is normally processed without pre-concentration, although whey can be processed at up to 18% total solids (Houldsworth, 1980). The only benefit for pre-concentration for IE is the column volumes can be reduced. Partially demineralised pre-concentrated whey at 26% TS is IE processed at a Northland plant. Recently membrane technology (Nanofiltration) was developed which both partially demineralises and concentrates whey in one process step. This process would be beneficial to IE technology.

2.9 PROBLEMS ASSOCIATED WITH IE

IE is quite widely used for whey demineralisation; however the process has a number of disadvantages (Lefevre, 1986). These are:

2.9.1 DISCONTINUOUS NATURE

Because resins require regeneration, the ion exchange process is truly batch in nature, whereas most other operations in the dairy industry are continuous. In order to move toward more continuous operation and interface better with other operations, multiple ion exchange plants can be used in parallel (Finer, 1993). It is possible to operate two identical streams to simulate a continuous operation. This requires completion of the regeneration step in the same time whey processing to mineral breakthrough occurs. The rate limiting step in this balanced plant is the regeneration time, therefore it is important to minimise this step (Horton International, unpublished data).

2.9.2 REGENERANT USAGE

Regenerant costs are the largest factor in the operating cost for ion exchange process. The cost of chemicals per unit volume of whey treated is almost directly proportional to the amount of minerals removed. Logistic problems as well as high costs and safety problems exist with regenerant chemicals (Delaney, 1976).

2.9.3 WATER REQUIREMENTS

The ion exchange process requires large quantities of rinse water to remove excess regenerant from the resins after regeneration. Water volumes of between 1-2 m^3 of water per m^3 of whey processed are stated. The total water requirements for ion exchange depend, for example upon whether the rinsing water from resin regeneration is stored and re-used in subsequent regeneration cycles, or discharged to waste (Delaney, 1976).

2.9.4 EFFLUENT PRODUCTION

The large water requirement of IE gives rise to a considerable quantity of effluent. It follows that effluent volumes correspond to water usage. Typically the effluent volumes are approximately twice the volumes of whey processed. Effluent concentrations of 1700-2200 mg BOD/L are typical. The protein and lactose depositing on the IE resins affect this concentration. Salt disposal and pH balance in the effluent are also problems. Salt arises from both the ash extracted from the whey and the regeneration chemicals (Delaney, 1976).

2.9.5 FOULING

For IE operation the process whey is pumped through the columns. Blocking of the columns can be a major problem. Avoidance of fouling is critical. One major fouling contaminant in the whey is cheese fines. To avoid this, filters are used prior to the IE to remove all suspended solids from the whey.

Fouling coming from the whey stream also has detrimental effects on all the other problems associated with the IE (Hoppe *Unpublished*). The two main components from the whey stream (Table 1) not to be removed in the IE process are lactose and whey protein. In unpublished data (Smith, 1998) industrial losses of approximately 22% lactose and 43% protein were encountered on an IE plant. As fouling can have such a significant effect on the operation of the IE, investigations into these two phenomenons are required.

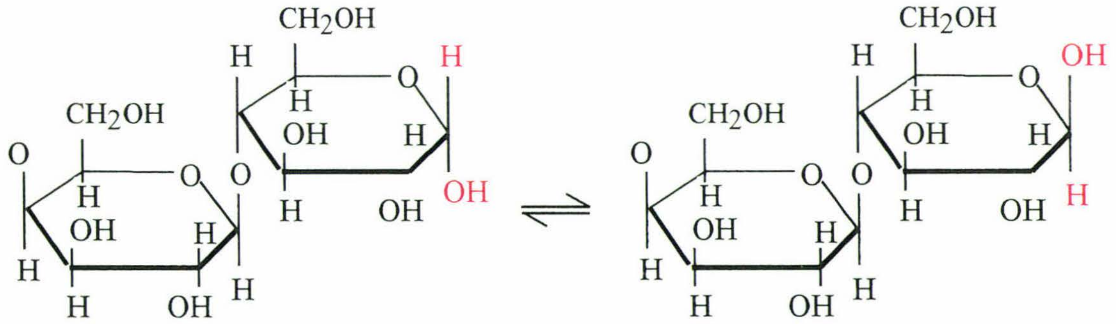
2.9.5.1 Lactose Fouling

Finer (1993) identified considerable lactose adsorption onto anionic resin during processing of whey permeate. Trials showed that these losses were in order of 28% of the total cycle product throughput. In an attempt to understand the mechanism of how lactose, a non-ionic substance, can be adsorbed onto an IE resin, two possible mechanisms have been proposed. The first is entrapment of the lactose within the resin structure. The second involves the mutarotation of lactose. An understanding of lactose chemistry is given to explain this mechanism.

2.9.5.1.1 Lactose Chemistry

Lactose is the naturally occurring reducing sugar in both human and cow's milk (M^{c}Murray, 1988). It is a dissaccharide formed by the linking of glucose and galactose and exists in two stereo-isomers, known as alpha (α) lactose and beta (β) lactose (Figure 10), which differ in steric configuration of the -OH and -H groups (Smart, 1992).

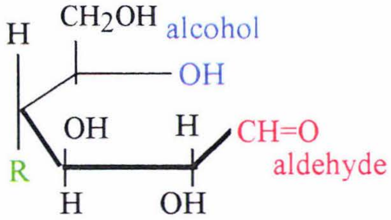
Figure 10: Lactose Isomers (M^{c}Murray ,1988)



α-Lactose β-Lactose

The open chain glucose section of the lactose molecule contains both an aldehyde and alcohol group in close vicinity (Figure 11).

Figure 11: Open Chain Lactose (M^cMurray ,1988)

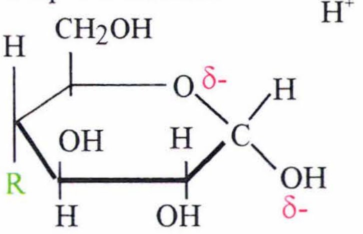


Note:
R = galactose ring

Alcohols undergo a rapid and reversible nucleophilic addition reaction with aldehydes to form hemiacetals. For lactose both the hydroxyl and the carbonyl group are in the same molecule, this leads to the formation of a cyclic hemiacetal (Figure 10). This equilibrium reaction between the two forms of lactose is known as mutarotation. Isbell & Pigman (1969) stated the mutarotation of lactose in an aqueous solution involves the transfer of a proton from an acid catalyst to the lactose, and the transfer of another proton from the lactose to a base catalyst. The reaction starts with attack on the cyclic lactose by either the acid or a base catalyst, followed by a slow ring rupture. Although equilibration is slow at neutral pH, it is catalysed by either acid or base. The detailed reaction mechanisms for both the acid and base catalyst are shown in Figures 12 and 13 respectively.

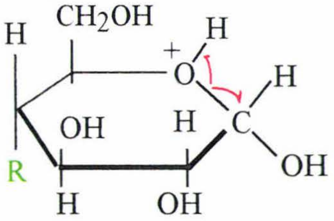
Figure 12: Acid Catalysed Mutarotation

Step 1 **α-Lactose**



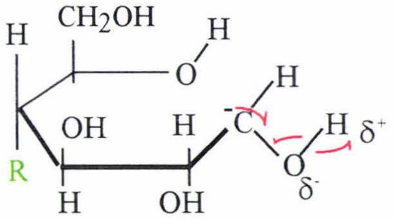
a neutral hemiacetal tetrahedral intermediate and the acid catalyst.

Step 2 **α-Lactose**



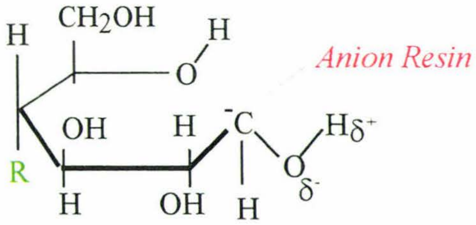
acid catalyst binds to the slightly negatively charged oxygen, leading to formation of a positively charged

Step 3 **Open Chain-Lactose**



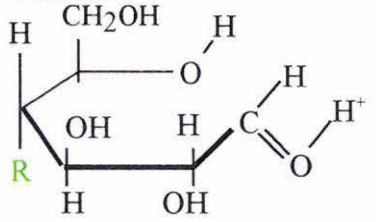
Ring formation bonds breaks, to form another negatively charged intermediary. Terminal carbon free to rotate, therefore negatively charged intermediary can bind to anion resin.

Step 4a **Resin Bound Open Chain-Lactose**



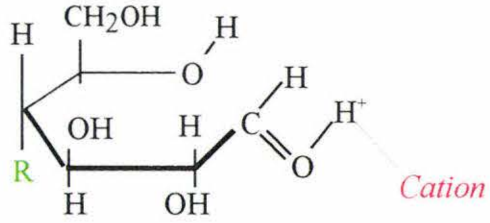
Terminal carbon rotates, exposing negatively charged carbon, which can bind to anion resin.

Step 4b **Open Chain-Lactose**



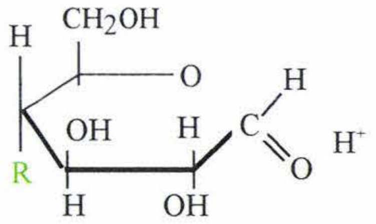
C=O double bond formed, positively charged hydrogen atom formed. Binding to cation resin would remove hydrogen ion.

Step 5a **Resin Bound Open Chain-Lactose**



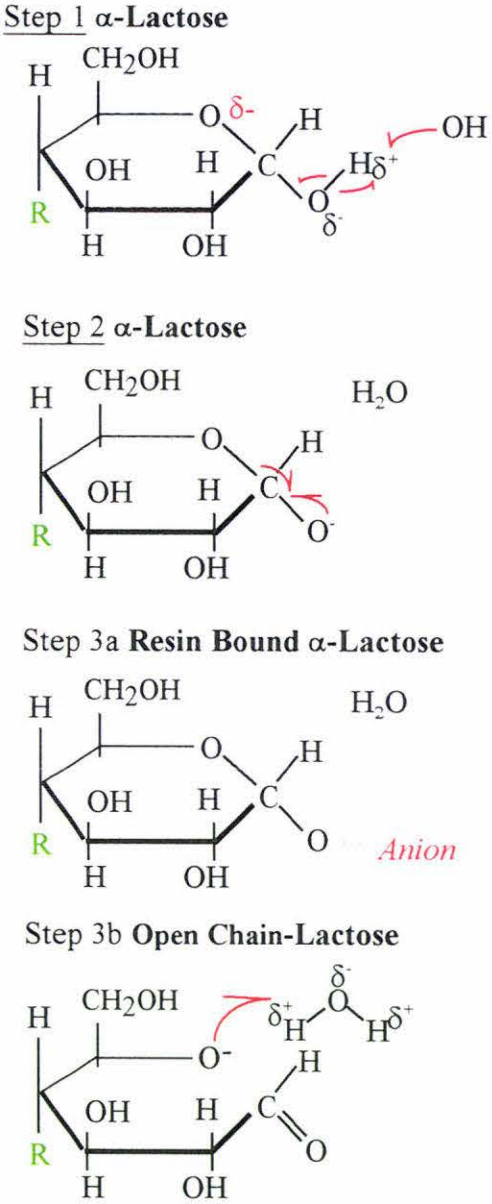
Positively charged hydrogen atom binds to cation resin, hydrogen ion breaks from lactose open chain aldehyde. Resin regenerated ready to demineralise again.

Step 5b **Open Chain-Aldehyde**



acid catalyst expelled and an open chain aldehyde formed, free to rotate.

Figure 13: Alkali Catalysed Mutarotation



Step 1 **α-Lactose**

a neutral hemiacetal tetrahedral intermediate and the base catalyst.

Step 2 **α-Lactose**

base catalyst removes the slightly positively charged hydroxyl proton, leading to formation of a negatively charged intermediary.

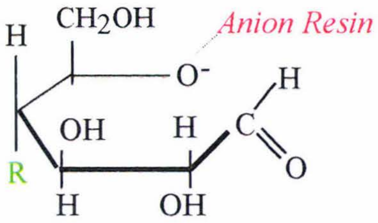
Step 3a **Resin Bound α-Lactose**

base catalyst removes the slightly positively charged hydroxyl proton, leading to formation of a negatively charged intermediary.

Step 3b **Open Chain-Lactose**

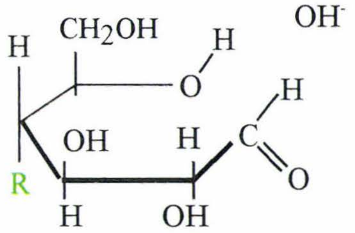
then ring formation bonds breaks, to form another negatively charged intermediary.

Step 4a **Resin Bound Open Chain-Lactose**



the charged intermediary can bind to the anion resin, as the aldehyde group is free to rotate and not sterically hinder the charged O atom.

Step 4b **Open Chain-Aldehyde**



base catalyst regenerated and an open chain aldehyde formed, free to rotate.

It can be seen from both reaction mechanisms that charged intermediary species are formed. For these charged species to interact with the ion exchange resins, the charged atom must be accessible from outside the lactose molecule structure. Positively charged hydrogen ions will cause transfer of the hydrogen to the cation resin. Possible attachments of the charged intermediaries in both reaction mechansims to IE resin are indicated by the introduction of the resin in red lettering.

In the one instance when a positive charge is available for binding to the cation resin, the hydrogen atom adjacent to the bond would bind to the cation resin, regenerating the cation resin and releasing an acyclic lactose molecule. In all three remaining instances when the charged lactose intermediary species were available to bind to the IE resin, the lactose molecule would bind to the anion resin.

From the lactose mutarotation mechanisms it is clear that the lactose could only absorb by ion exchange onto the anion resin, no lactose adsorption should occur on the cation resin. This theory is in agreement with the trial findings uncovered by Finer (1993).

The rate of lactose adsorbed onto the anion resin is dependent on the formation of the charged intermediary species through the mutarotation reaction. The mutarotation rate of reaction is effected markedly by both pH and temperature Lowe (1993). It can be seen from Figure 14 and 15 below that the pH has a strong influence on the mutarotation reaction rate in highly acidic or alkali conditions, whereas the temperature has a positive logarithmic effect on the mutarotation reaction rate. In the IE process pH varies from pH 1.7 to pH 10 (see Figure 7). These pH extremes will cause rapid lactose mutarotation. The rapid lactose mutarotation reaction forms charged intermediary species. It is speculated that these species then absorb onto the anion resin. This releases more hydroxyl ions from the anion resin to further catalyse the process.

Figure 14: The Effect of pH on the Mutarotation Rate of Lactose (Lowe, 1993).

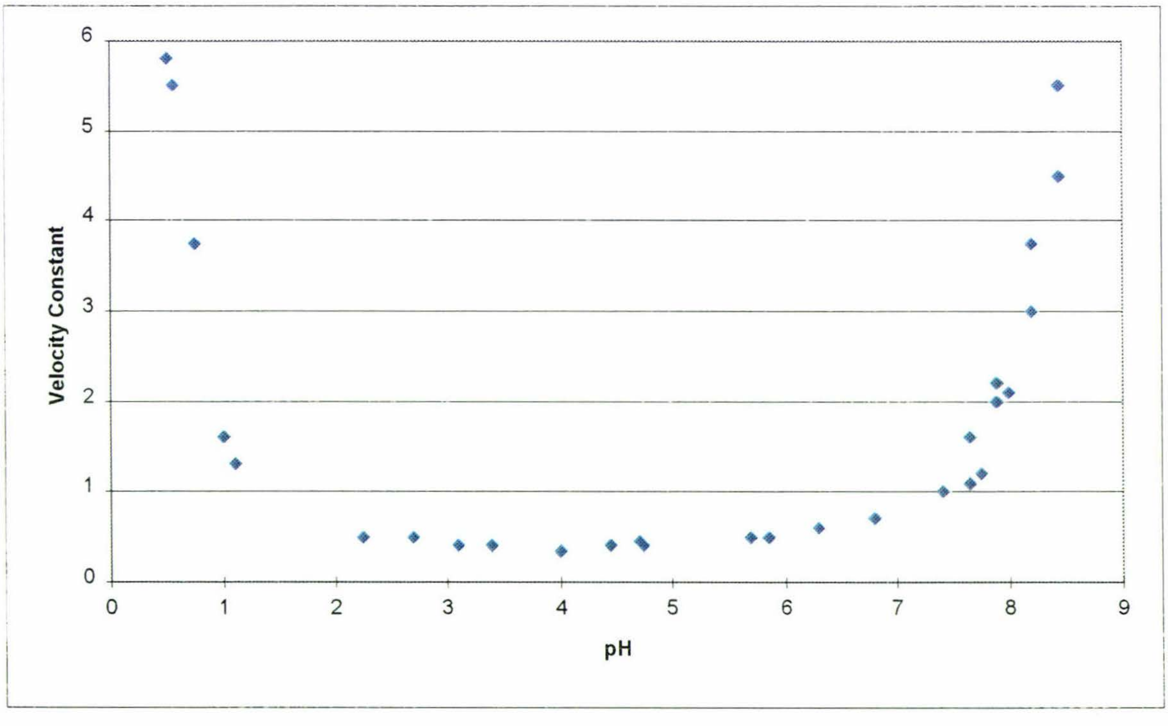
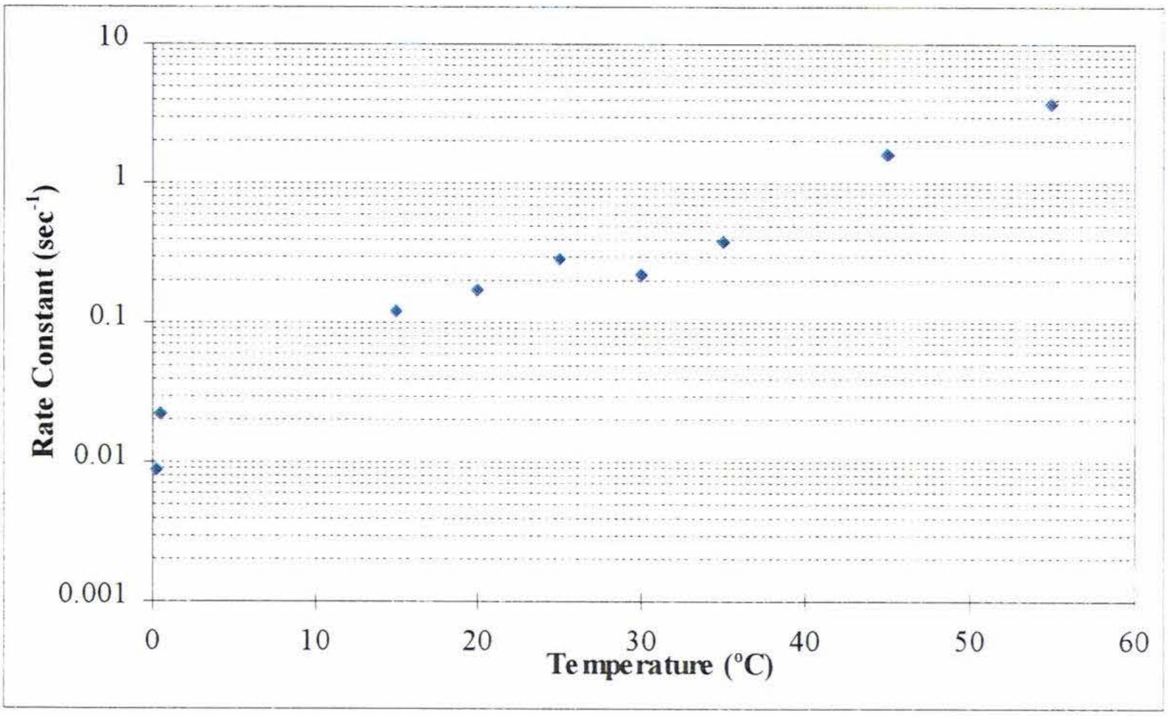


Figure 15: Temperature Effect on the Mutarotation Rate of Lactose (Lowe, 1993).



2.9.5.2 Whey Protein Chemistry

Whey proteins are the non-casein proteins as well as the fraction and fragments of the caseins that remain soluble when the caseins have been precipitated enzymatically by rennet or isoelectrically by acid. They share very few common characteristics except that of being soluble under the conditions that precipitate the casein (Cleland, 1994).

If whey proteins are exposed to acids or bases or heated to temperatures above a certain maximum their structure is altered. They are said to be denatured and lose their original solubility and precipitation occurs. When whey proteins are denatured, their biological activity ceases. After a weak denaturation, proteins can sometimes revert to their original state, with restoration of their biological functions (Bylund, 1995). The pH when the individual proteins precipitate is called the protein isoelectric point (pI). The whey proteins isoelectric points are shown in Table 2 (Hodges, 1992).

Table 2: Individual Whey Protein Data (Hodges, 1992)

Whey Protein Group	Distribution	Molecular Weight	Isoelectric Point pI	Heat Sensitivity
β - Lactoglobulin	45%	18,000	5.2	3
α - Lactoglobulin	20%	16,000	5.1	4
Proteose Peptone	20%	66,000	4.9	Heat Stable 5
Serum Albumin	5%	very large	6.5	Very Sensitive 1
Immunoglobulin	10%	very large	5.1-6.0	2

Very heat sensitive whey proteins, Serum Albumin and Immuglobulins, can denaturate at temperatures encountered during pasteurisation (up to 72°C). This might explain the observation that protein precipitation is effected by the prior heat treatment observed by Delaney (1976). The most heat stable protein, proteose peptone, is stable even at high temperatures (> 120°C). Pasteurisation of the whey is required for cheese whey to inhibit the further conversion of lactose to lactic acid by the bacteria introduced in the cheese making process and also as a precautionary micrbiological control.

From Table 2 the pI of the whey proteins occur between pH 4.9-6.0. The isoelectrical precipitation of the whey proteins is also time dependent. Therefore the precipitation is not instantaneous.

2.9.5.2.1 Whey Protein Fouling

Losses of non protein nitrogen, when processing raw whey through IE, are reported to be typically in the range of 50-70%. Heat treatment also has a significant effect on protein loss. As previously mentioned, when ambient whey is processed through IE technology, protein losses of about 30% occur. At a process temperature of 5-10°C, the whey protein loss is often about 5-10%. (Delaney, 1976).

Delbeke (1972) states trials have shown flocculated protein begin eluting from the anion column when the pH drops to 6. As soon as the effluent reaches a pH of 3.5 no more flocculated protein appears. In a transparent column one can see that a zone of flocculated proteins moves slowly downwards and from the pH elution profile it can be deduced that this zone has a pH of approximately 3.5-6. This zone migrates because after sufficient exhaustion of the upper resin layers, the acidified whey added partially dissolves the flocculated proteins after which they again flocculate lower down the column. A proportion of the flocculated proteins remains adsorbed by the resin, so that the exchange capacity is partially blocked. The effect of protein precipitation at the isoelectric point can explain the phenomenon of protein precipitation observed by Delbeke. Potgieter *et al.* (1986) also stated that resin entrapment of the whey protein increases protein losses during IE processing.

2.9.5.3 Minimising Lactose and Protein Losses

From literature it appears that the factors most effecting the lactose adsorption and protein flocculation are pH, temperature and resin type. Heat pretreatment temperature has also been identified as a factor effecting the protein flocculation, but heat treatment is required to ensure the product is of good quality. Therefore heat pretreatment temperature is not a variable that can be controlled in this process.

Delbeke (1972) found that different resins gave markedly different results when demineralising whey. Delbeke (1972) also stated that experiments have been performed to show that a larger volume of whey can be demineralised per litre of resin before a pH of 6 is reached. These experiments were by the addition of 10% normal whey or addition of 300 mg K per litre to completely decationised (acidified) whey during de-acidification. The total decrease in the ash content of 90% was always obtained. In practice, due to a limited regeneration of the cation resin, the whey is also incompletely decationised. The cation content is however unstable and increases as percolation proceeds (Delbeke, 1972).

2.10 IE FOULING HYPOTHESIS

Solids Non Ash (SNA) or lactose adsorption onto IE resin is a phenomenon that has been identified by previous authors. Two possible mechanisms for the adsorption of the lactose to the IE resin have been proposed, namely the:

1. Resin Entrapment Mechanism
2. Lactose Mutarotation Intermediary Species Binding Mechanism

With knowledge of the controlling mechanism it may be possible to propose a method to minimise the lactose adsorption. The objective of this thesis is to undertake experimental work to identify the mechanism that is controlling the adsorption of lactose to the IE resin.

3.0 MATERIALS AND METHODS

3.1 MATERIALS

3.1.1 RESIN

All resin for experimentation was obtained from resin suppliers. Ion Exchange resins used in these trials are shown below in Table 3.

Table 3: Ion Exchange Resins

Resin Type	Resin Manufacturer	Resin Description	Resin Structure
Strong Cation	Purolite	C100H	Gel
Weak Anion	Purolite	A103S	Macroporous
Weak Anion	Rohms & Haas	IRA95	Macroporous
Weak Anion	Purolite	A847	Gel
Weak Anion	Purolite	A845	Gel

3.1.1.1 Resin Regeneration

Washing and regeneration of the new resins were performed following resin supplier recommendations. Resins were regenerated in 200 mL batches. All resins used for experimentation were initially wetted, excess water drained off and then the resins were regenerated. All resins were regenerated with three equivalent (200 mL) volumes of 4%(m/m) regenerant solutions. For cationic and anionic resins the regenerant solutions were Sulphuric Acid and Sodium Hydroxide respectively. On addition of the solutions to the resins, the slurries were stirred for 30 minutes. Excess regenerant solution was then decantered off.

Regenerated resin slurries were then cleaned with tap water to remove excess regenerant solution. One litre volumes of tap water were added to the resin slurries. The solutions were stirred, left to settle and then excess water decantered off. This process was repeated three times. The washing process was then repeated with reverse osmosis (RO) water. The decantered solution was collected and analysed for conductivity, to indicate how much regenerant chemical remained. Washing with RO water was repeated until the decantered solution conductivity was less than 50 μS/cm. Regenerated resins were stored in sealed containers until used.

3.1.1.2 DMSO Moisture Trial Resin Preparation

For trial 28 (DMSO Moisture analysis trial), the regenerated anionic resin (100 mL) was washed with 200 mL of dimethyl sulfoxide (DMSO), stirred and then decantered off. This process was repeated three times. The objective of this preparation was to minimise water incorporation in the experiment.

3.1.2 FEED SOLUTIONS

Liquid solutions were added to the regenerated resin to investigate the lactose adsorption onto the resin. These liquid solutions were termed "Feed Solutions". Each trial used one of the feed solutions detailed below:

3.1.2.1 NF Whey

Nanofiltered Whey (NF Whey) for the first three experiments was collected from frozen samples of previous NF whey pilot plant samples. The composition of the NF whey is shown below in Table 4. The samples were defrosted, by immersion of the sample and container in cold tap water. NF whey for trial 11 was collected from an industrial plant. The ash and TS composition for NF whey used in trial 11 are shown below in Table 5.

Table 4: NF Whey Composition for Trials 6, 7 and 8

Component	Result
Fat	0.14%
Protein	2.88%
Lactose	13.38%
Ash	0.90%
TS	17.30%

Table 5: NF Whey Composition for Trial 11

Component	Result
Ash	1.3%
TS	20.2%

3.1.2.2 Lactose

Lactose feed solutions were prepared from AnalaR® Lactose (BDH Laboratory Supplies, Poole, England). Lactose was weighed and then a set weight of RO water was added to the lactose. The lactose solution was then stirred to dissolve the lactose. Complete dissolution of the lactose was indicated by no noticeable white powder particles. To aid dissolving of the lactose, the solutions were heated gently in a microwave. Solutions were then allowed to cool to ambient temperatures unless specified in trial data.

3.1.2.2.1 Acidified Lactose

For trials requiring acidified lactose (10 and 12), GPR™ Sulphuric Acid, about 90% concentration (BDH Laboratory Supplies, Poole, England) was added to a lactose solution at ambient temperatures. The sulphuric acid was added dropwise to the continuously stirred lactose solution, until the end point of addition had been reached. The end point of the addition occurred when the stirred solution dropped below pH 1.

3.1.2.2.2 DMSO/Lactose Solutions

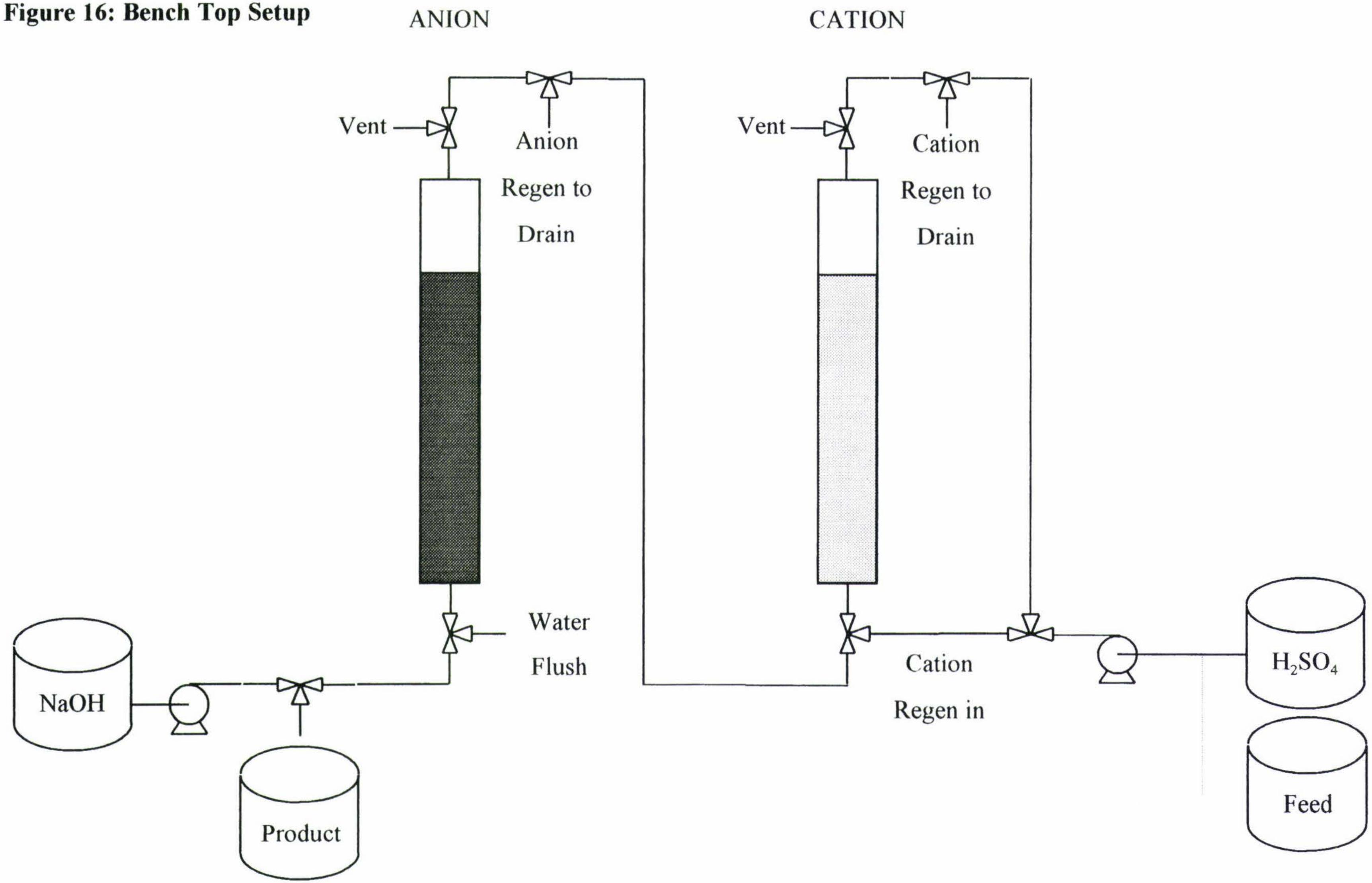
Feed solutions of lactose dissolved in DMSO were prepared for trials 14, 15, 16 and 28. These feed solutions were prepared from AnalaR® Lactose and of AnalaR® DMSO (BDH Laboratory Supplies, Poole, England. Lactose was weighed and then a set weight of DMSO was added to the lactose. The lactose solution was then stirred to dissolve lactose. No heating of the solutions was required to dissolve the lactose/DMSO solutions.

3.2 EXPERIMENTAL METHODS

3.2.1 CONTINUOUS METHODS

Benchtop IE columns (as shown below in Figure 16) were employed for trials 6 to 13. This experimental design was used to create a bench top continuous system similar to the industrial plant. The IE columns used were Pharmacia Biotech XK26 columns. Feed solutions of NF whey, lactose or acidified lactose were pumped through the columns at set flowrates (as indicated in the Appendix trial data). Samples were collected in plastic containers. The cation sample (trial 6) was collected from the "Anion Regen to Drain" three way tap (Figure 16). Yield results for the continuous trials were calculated by dividing the TS collected from the anion column by the TS fed onto the column(s).

Figure 16: Bench Top Setup



3.2.2 BATCH METHODS

Batch methods were used to simplify experiments. Weighed proportions of resin slurry and feed solution were added together in a 100 mL plastic container. Samples were then shaken for approximately one hour, at ambient temperatures with a KS501 Digital Shaker box (Manufactured by IKA LABORATECHNIK). The shaker box was set at 120 revolutions per minute (rpm). Batch samples introduced a new variable of Feed:Resin (F:R) ratio. Feed resin ratio was the ratio of feed solution to resin slurry sample.

3.2.2.1 Batch DMSO Moisture Trials

A batch DMSO moisture trial was performed in trial 28 to investigate if water evolution occurred during the addition of DMSO/lactose solutions to A103S anion resin. Special resin preparation methods were performed as described in Section 3.1.1.3. DMSO/lactose solutions were prepared as described in Section 3.1.2.2.2. Moisture analysis was performed as described in section 3.4.2.

3.2.2.2 Batch Lactose Recovery Trials

Adsorbed lactose was recovered from spent anion resins with RO water. The resins were previously spent in lactose adsorption trials. Excess lactose solution from the adsorption trials was removed from resin slurries. A measured mass of RO water was added to the dried resin sample. The samples were then shaken for approximately one hour, at ambient temperatures with a KS501 Digital Shaker box (Manufactured by IKA LABORATECHNIK). The shaker box was set at 120 revolutions per minute (rpm). Stirring during lactose recovery was only possible during batch methods. A solution sample was then extracted and analysed for TS eluted. Excess solution was removed and discarded. Each lactose recovery cycle was designated an "elution". During lactose recovery trials the number of elutions were as deemed necessary.

3.3 TEST METHODS

3.3.1 CONDUCTIVITY

Conductivity was measured with a CDM83 Conductivity Meter (manufactured by Radiometer Copenhagen). The conductivity probe was immersed in the plastic container holding the sample.

3.3.2 REFRACTIVE INDEX

An Atago N-20 hand-held refractometer (Tokyo, Japan) was used for all Brix measurements. The range of the meter was 0-20% brix. This limited the feed stock to less than 20% TS. The zero reading for the refractometer was performed with deionised water.

3.3.3 pH

Samples were measured for pH with a PHM220 Lab pH Meter (manufactured by Radiometer Copenhagen). The probe was immersed in the plastic container holding the sample.

3.3.4 FLOWRATE

Pumps for the continuous experimental methods were calibrated with a 50 mL measuring cylinder and timer.

3.3.5 WEIGHT

All weight measurements were performed with a Mettler Toledo AB204 set of scales.

3.3.6 LABORATORY METHODS

Standard laboratory analysis methods were as detailed in the following:

Fat - NZTM 3: Chemical Methods Manual. Section 6.3
Protein - NZTM 3: Chemical Methods Manual. Section 15.7
Ash - NZTM 3: Chemical Methods Manual. Section 4.2
Chloride - NZTM 3: Chemical Methods Manual. Section 9.1
Aqueous Total Solids - NZTM 3: Chemical Methods Manual. Section 12.15
Moisture - NZTM 3: Chemical Methods Manual. Section 12.11
Lactose – Calculated by Difference (Total Solids less Fat, Protein and Ash)

Waitoa analytical laboratory staff performed all analyses on the NF whey feed solutions. The author performed all other analyses.

3.3.6.1 DMSO Total Solids Analyses

Trials 14, 15, 16 and 28 used DMSO/lactose as a feed solution. DMSO solvent has a boiling point of 189-193°C. Therefore the aqueous Total Solids method was not appropriate to analyse lactose/DMSO samples for lactose TS. Any new method must account for temperature effects on lactose. From Ling (1963) heating lactose solutions between 110-130°C, lactose hydrate crystals lose their water of crystallisation. Above 150°C they turn yellow, while at 175°C they brown considerably, forming lacto caramel. It was therefore decided to evaporate the samples under vacuum at approximately 130°C until samples appeared caramelised. A Gallenkamp vacuum oven with a transparent door to allow visual monitoring of the samples was employed.

The feed sample was used as the standard, to indicate how much DMSO evaporation had occurred. If evaporation was not complete, samples could be adjusted with a correction factor (shown in Appendix, Pg A13).

3.3.6.2 DMSO Moisture Analyses

DMSO Moisture Analysis was performed as detailed in NZTM 3: Chemical Methods Manual. Section 12.11. The only modification to the above method was the weighed powder sample was replaced with a solution of DMSO/lactose.

4.0 RESULTS AND DISCUSSION

4.1 INTRODUCTION

Herve (1974) and Finer (1993) identified instances of unexpected lactose and/or protein adsorption during the IE processing of whey and whey permeate dairy streams. Finer (1993) indicated the lactose adsorption occurred during the anion demineralisation step. Initial trials were performed on a continuous column system to verify this work. Results from inconclusive trials (trials 1-5, 22) have been omitted.

4.2 CONTINUOUS TRIALS

4.2.1 CATION AND ANION COLUMNS

Initial trials employed the cation (C100H) and anion (A103S) resin experimental set up as described in the Materials and Methods section. Trials 7 and 8 were performed to quantify the SNA adsorption during IE processing of NF whey. Results for trials 7 and 8 are shown in the Appendix, Pg A2-A3. The SNA yields for these trials were 91% and 90% respectively. The SNA yield results for these trials and all the other continuous trials are shown in Table 6.

The objective of trial 6 was to confirm that NF whey SNA adsorption occurred predominantly on the anion column. Results for trial 6 are shown in the Appendix, Pg A1. A cation sample was collected to investigate SNA adsorption onto the cation column. The cation sample (65 mL) was collected after 1.2 bed volumes (BV) had passed through the cation column. Some dilution effects may have occurred at this stage of the process cycle. Comparison of the TS eluting from the cation column (16.2%) and the anion column

(3.8%) at the initial stages of the processing cycle, indicate that SNA losses predominantly occur in the anion column. This was in agreement with literature findings (Finer, 1993). The SNA yield for trial 6 was 90%. From Table 6, it can be seen that the SNA yields for these trials are similar to the results from trials 7 and 8.

Table 6: SNA Yields from Continuous Trials

Continuous Trial Number	SNA Yield
6	90%
7	91%
8	90%
9	92%
10	93%
11	91%
12	91%
13	84%

4.2.2 MODIFIED COLUMN EXPERIMENTAL METHODS

Results from trials 6, 7 and 8 had confirmed that SNA adsorption had occurred during the IE processing of NF whey. To allow further investigation of the adsorption mechanism, modifications to the experimental methods were required. Individual investigations of each resin type were undertaken by separating the cation and anion columns. Results from trial 6 had indicated that adsorption occurred predominantly in the anion column, therefore the anion resin was initially investigated. Anion resin investigations were operated with the feed solution pumping directly to the top of the anion column. Later trials would be required to verify that only limited adsorption occurred in the cation column.

Experimental modifications were also performed on the feed solution. The feed solution was changed to pure lactose to remove any interfering effects of other components in the NF whey. SNA and lactose yields are equivalent with a pure lactose feed stream. The pure lactose feed also allowed specific investigations into the lactose adsorption. With no other organic molecules present in the pure lactose feed solution the TS analysis would also measure the lactose content.

Comparison of the yield results for the modified continuous trials with the earlier trial results would be required to confirm modifications had not significantly effected the SNA yields. With no significant changes in the SNA yields it would be confirmed that earlier trial yield results from NF whey were due to lactose adsorption. Results for trial 9 are shown in the Appendix, Pg A4. The SNA/lactose yield result for trial 9 was 92%, as shown in Table 6. This indicated significant SNA adsorption occurred. This SNA yield was a similar result to those of the previous trials (Table 6). Therefore the experimental modifications had not significantly effected the SNA yield. This confirms that earlier trial SNA yield results were due predominantly to lactose adsorption. The close agreement with SNA yields before and after the experimental modifications indicate the simplified experimental conditions were suitable for further investigations into the lactose adsorption phenomenon.

Cation processing acidifies the dairy solution prior to anion processing. Decreasing the pH significantly increases the rate of the lactose mutarotation reaction. An increase in the lactose adsorbed with acidifying the feed solution would identify the mutarotation mechanism as the controlling mechanism. The objective of trials 10 and 12 was to indicate whether the mutarotation rate at neutral pH was sufficiently fast enough so that it does not control lactose adsorption. Sulphuric acid was used to adjust the lactose feed solution pH to match that typically fed into the anion column from the cation column. The adjusted pH for trials 10 was 0.99 and for trial 12 the feed pH was 0.97. Results for trials 10 and 12 are shown in the Appendix, Pg A5 and A7 respectively. The SNA yields for trials 10 and 12 were 93% and 91% respectively, as shown in Table 6. There was no significant change in the SNA yields compared with trial 9. Acidification of the feed solution did not significantly effect the lactose adsorption. This would indicate that the mutarotation rate at neutral pH was sufficiently fast enough that it does not control lactose adsorption onto the anion resin. Therefore, remaining trials were performed with no pH adjustment.

Investigations were required to ensure that the resin usage was not effecting the SNA yields. NF whey was processed through the anion column to allow comparison with earlier cation and anion processed NF whey trials. No significant change in SNA yields would indicate that the resin usage was not effecting the lactose adsorption. No significant change in the SNA yields would also provide further evidence that lactose adsorption predominated in the anion column. Results for trial 11 are shown in the Appendix, Pg A6. The SNA yield for trial 11 was 91%, as shown above in Table 6. Close agreement with earlier trial results indicated the six cycles of the resin life had not significantly effected the SNA yield. Close agreement with earlier results also provided further evidence that lactose adsorption occurs predominately in the anion column.

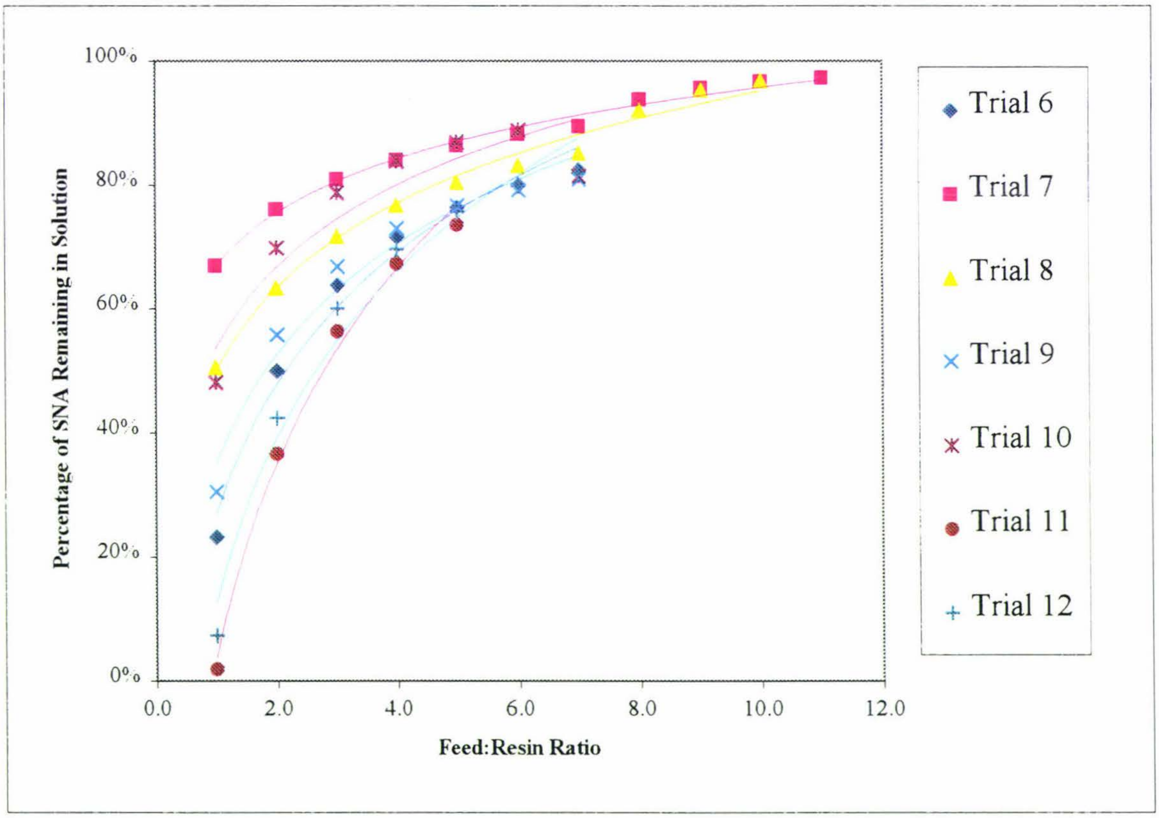
4.2.3 LACTOSE ADSORPTION OCCURRENCE

To identify when SNA adsorption was occurring during the processing cycle investigations of all trial results were required. The percentage of lactose remaining in solution would give an indication of when lactose adsorption was occurring during continuous trials. All trial percentage lactose remaining in solution results are shown in Figure 17.

Variations in the percentage of lactose remaining in solution curves in Figure 17 were due to three factors. The first factor effecting the remaining SNA concentration was channeling effects in the columns. The second effect causing fluctuations was the dilution effects from the water immersing the resin. Resin suppliers recommended that resins contained in the continuous columns were kept immersed in liquid at all times. This was to ensure the resin beads did not dry out and fracture during processing. In the initial stages of sample collection the resin water diluted any lactose in solution. Therefore the lower the F:R ratio the greater the proportion of resin water and dilution effects in the sample.

The third effect causing fluctuations were the point at which samples were collected. Initially in trial 6 the sample collection began when NF whey was first fed onto the cation column. This was to ensure all product was collected. During this trial, it was noticed that the first of the whey eluting from the anion column could visibly be seen. This experience was used in trials 7 and 8 when sample collection began when the first of the whey eluted.

Figure 17: Percentage of SNA Remaining in Solution



When lactose solutions were used, a new problem was encountered. The lactose solutions were opaque, therefore it was impossible to visibly define the lactose front. To ensure all of the lactose flowing from the anion column was recovered, all liquid exiting the anion column was collected. This resulted in collection of the initial resin water. This process effects the percentage of SNA remaining in solution at low F:R ratios as previously discussed. Dilution of the lactose with resin water did not effect the lactose yield for each trial. For trial 10 a pipe movement caused the initial flush water to be lost. Enough experience had been gathered at the time to know the lactose had not reached the collection point. Therefore sample collection began at this point and the trial continued.

Analysis of the results presented in Figure 17 revealed the lowest remaining lactose concentrations occurred at the lower Feed:Resin (F:R) ratios. This would indicate that the greatest adsorption and/or dilution of lactose occurs at the initial F:R ratios. To prove that lactose adsorption occurred at the lower F:R ratios trial 13 was completed. In trial 13 a lower BV throughput was used to focus on the lactose adsorption at the low F:R ratios. Resin water dilution effects would not effect the overall lactose yield provided sufficient flush water was used. A reduction in the yield compared to previous trials would confirm lactose adsorption occurred. Results from trial 13 are shown in the Appendix, Pg A8. From Table 6 it can be seen that the SNA yield for this trial was 84%. This result was substantially lower than yields for the other continuous trials. This confirmed that lactose adsorption occurred. Therefore lactose adsorption occurred at the beginning of the process cycle. The best methodology to continue to investigate the lactose adsorption, while minimising dilution effects at the low F:R ratios was to employ batch methods. An added advantage of the batch method was to remove any effect of channeling through the columns.

Results from continuous trials had confirmed lactose adsorption occurred. Results had also identified when lactose adsorption occurred. Continuous trials had proven the rate of mutarotation reaction at neutral and acidified pHs had not affected lactose adsorption. Continuous trials had not identified the mechanism governing the lactose adsorption. Trials were required to confirm or exclude the lactose mutarotation reaction as the mechanism for any lactose adsorption. Inhibiting lactose mutarotation would allow investigating of the resin entrapment on the lactose adsorption. Therefore a method to completely inhibit the lactose mutarotation reaction was required. Olano *et al.* (1977) stated, no mutarotation could be detected by polarimetry during a period of one hour where α- or β-lactose were dissolved at 25°C in anhydrous dimethyl sulfoxide (DMSO) solvent. In some circumstances addition of acid or base allowed the mutarotation reaction to continue. Therefore DMSO solvent without the presence of acid or base would be used in batch trials to inhibit the mutarotation mechanism.

4.3 TS MEASUREMENT

During trials 11, 12 and 13 a simpler method to measure the TS content of samples was investigated. All TS analysis had been performed with the aqueous oven dried method. Refractive index analyses for all these samples were also performed. Results and graphs for the refractive index comparisons are shown in the Appendix, Pg A9-A11. Regression coefficients for each trial are shown below in Table 7.

Table 7: Refractive Index Regression Coefficients

Trial	Correlation Coefficient	R^2
11	1.017	0.997
12	1.027	0.999
13	1.009	1.000

It can be seen that the correlation coefficients were all close to one. This indicated agreement of the measurement of oven TS and refractive index TS over the range investigated. R^2 values were also close to one, indicating good agreement between the two methods. This supports the use of the refractive index to analyse the lactose TS in the anion elution. It was highlighted that mixing of the samples immediately prior to refractometer analysis, as done in trial 13, was required to give best results. The next trials employ

DMSO solvent to inhibit lactose mutarotation. The appropriateness of the refractive index analysis as a measure of lactose content in DMSO solutions required investigation.

4.4 DMSO BATCH TRIALS

4.4.1 DMSO LACTOSE ADSORPTION TRIALS

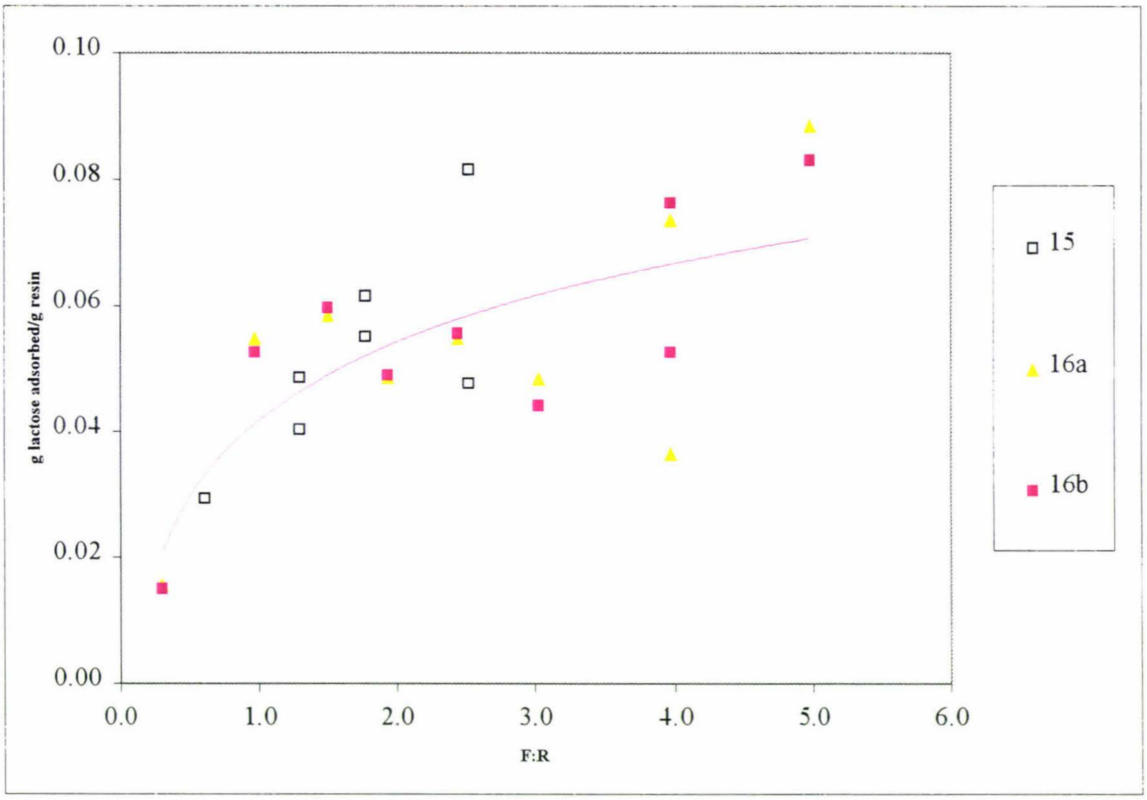
Batch trials with DMSO solvent were performed in trials 14, 15 and 16. The objective of these trials was to use DMSO to inhibit the mutarotation reaction. If any lactose adsorption occurred with the mutarotation theoretically stopped, then the mutarotation mechanism would be eliminated as a pathway for lactose adsorption. The resin slurries were partially dried in an attempt to reduce the resin water dilution effects. The moisture content of the partially dried resin was estimated as 2% for calculation purposes.

Refractive index analysis of the DMSO/lactose solutions did not provide any measurements. Attempts to rescale the refractometer again failed to provide any measurements. Literature investigations (Tennent, 1986) revealed the refractive index of DMSO (1.479) was significantly different from water (1.333). It was therefore decided to use the modified oven TS analysis for DMSO solutions.

During the DMSO oven TS analyses, it was difficult to identify when all the DMSO had evaporated. It was important to limit the oxidising of the lactose component. Samples required continuous monitoring in the oven during the evaporation process. Sample caramelisation was taken as the evaporation end point. Samples were then assessed for TS. In the initial DMSO trial (trial 14), the evaporation process was underestimated. As a result lactose adsorption results for trial 14 were understated. Therefore these results were not used for DMSO batch analyses. In trials 14 and 16 samples were initially dried and weighed, then the drying process was continued and samples reweighed. Results for trial 16 were designated 16a and 16b for the batch DMSO analyses. Corrections to the remaining

DMSO samples were made with a feed correction factor. A feed correction sample calculation is shown in the Appendix, Pg A13. Results for trials 14, 15 and 16 are shown in the Appendix, Pg A12-17. Adjusted lactose adsorption results for the valid DMSO trials (trials 15 and 16) are shown in Figure 18.

Figure 18: Adjusted Lactose adsorption results for DMSO/lactose batch trials



It could be seen from Figure 18 that lactose adsorption occurred in the DMSO/lactose solution samples. If the lactose mutarotation reaction was inhibited, then this reaction was not the mechanism controlling the lactose adsorption phenomenon. It was speculated that water may be catalysing the mutarotation reaction, by acting as an acid or a base. The moisture content of the feed and a feed/resin sample were measured. Results showed the feed solution moisture content was 5.5% and the feed/resin sample contained moisture of 14.9%. The analysis showed a large increase in the moisture content of the sample containing resin. In an attempt to provide a moisture-free environment, oven dried resins were used in the next series of trials.

4.4.2 BATCH OVEN DRIED DMSO TRIALS

To provide DMSO/lactose solution results with no moisture present, oven dried resins were employed. The three resin types used were:

- A103S Anion Resin (trial 18)
- C100H Cation Resin (trial 19)
- IRA95 Anion Resin (trial 20)

Results for the moisture content removed during oven drying are shown in the Appendix, Page A19. This information can be used during design to allow for ion exchange swelling capacity.

Further results for these trials are shown in the Appendix, Page A19-21. Results showed that the formation of TS occurred in the solution. These results were effected by the reactions that occurred. Evidence of reactions occurring during addition of the feed solution to the oven dried resins were noted. Oven dried resin was therefore not a feasible way of eliminating moisture from the trials. Attempts to eliminate water from the resin by washing with successive fractions of DMSO were employed in the next trial.

4.4.3 DMSO MOISTURE EVOLUTION TRIAL

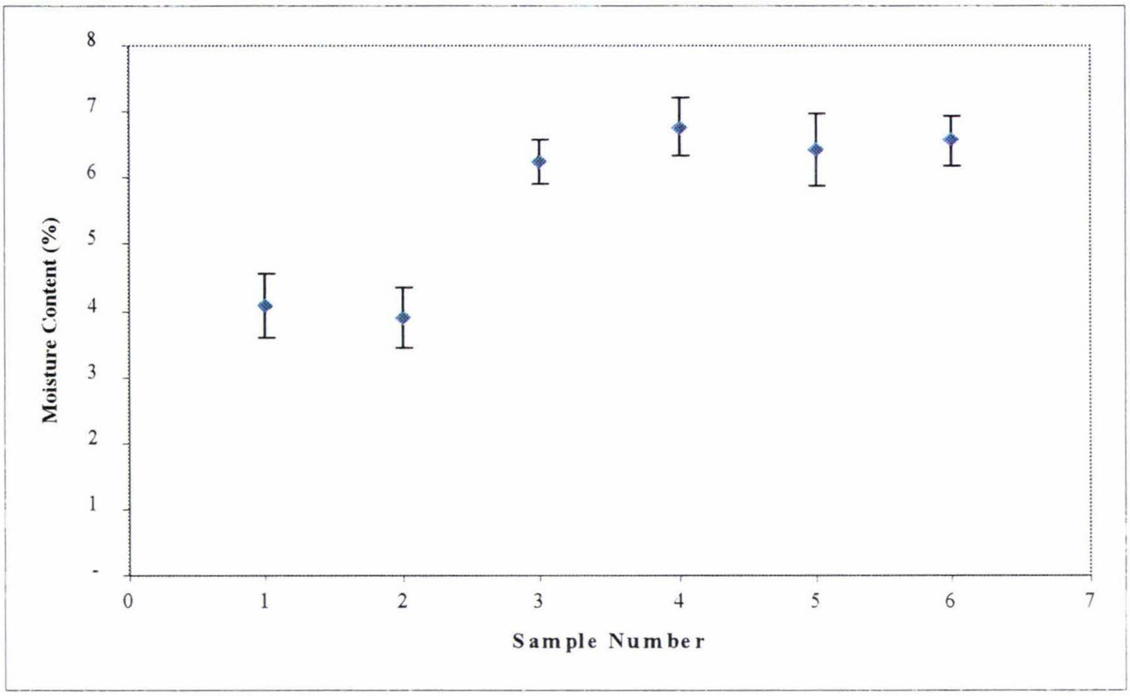
Resin for the moisture evolution trial (trial 28) was washed with successive fractions of DMSO. Washing the resin with DMSO was to eliminate moisture from the resin slurry. Increases in the moisture content could indicate lactose adsorption. If lactose was adsorbed by an ion exchange mechanism, then the charged lactose molecule replaces the OH^- group on the resin. The charged lactose molecule has given up an H^+ molecule to become charged. The free H^+ and OH^- ions can combine to give water. Expected moisture increase calculations are shown in the Appendix, Pg A47. An increase of approximately 0.1% was expected in the moisture content due to the lactose adsorption. If significant moisture exists in the solutions, then the inhibition of the lactose mutarotation with DMSO may not be occurring and hence adsorption by the ion exchange mechanism might explain the observed adsorption.

For trial 28 DMSO/lactose (DMSO/Lac/Resin) and DMSO (DMSO/Resin) solutions were added to A103S anion resin. A DMSO/lactose solution was sampled as a control (DMSO/Lac). Results and statistical analysis for trial 28 are shown in the Appendix, Page A44-A47. Results are graphically presented in Figure 19.

Statistical analysis showed at a 95% confidence level there was no significant difference between the lactose/DMSO/resin samples (samples 3 and 4) and the DMSO/resin samples (samples 5 and 6). This suggests that lactose adsorption by IE did not occur. However from Figure 19 the 95% confidence limits for each sample are greater than the expected increase in moisture content due to lactose adsorption of 0.1%. Measurement of moisture evolution to indicate lactose adsorption has not proved feasible.

Water inclusion occurred from both the DMSO/lactose solution and the washed resin. Therefore it could not be concluded that the lactose mutarotation reaction had been completely inhibited in DMSO/lactose solutions with small quantities of water present. Therefore the DMSO adsorption trials were not conclusive in eliminating the lactose mutarotation mechanism. If no lactose adsorption had occurred from the DMSO solution then it would have positively identified that the DMSO had stopped the mutarotation reaction and that the IE mechanism was the only mechanism of importance. The aim for future trials was moved from eliminating the lactose mutarotation mechanism to proving the resin entrapment adsorption. It was concluded that aqueous solutions were the best environment to prove the resin entrapment mechanism.

Figure 19: 95% Confidence Interval for Moisture Evolution



KEY

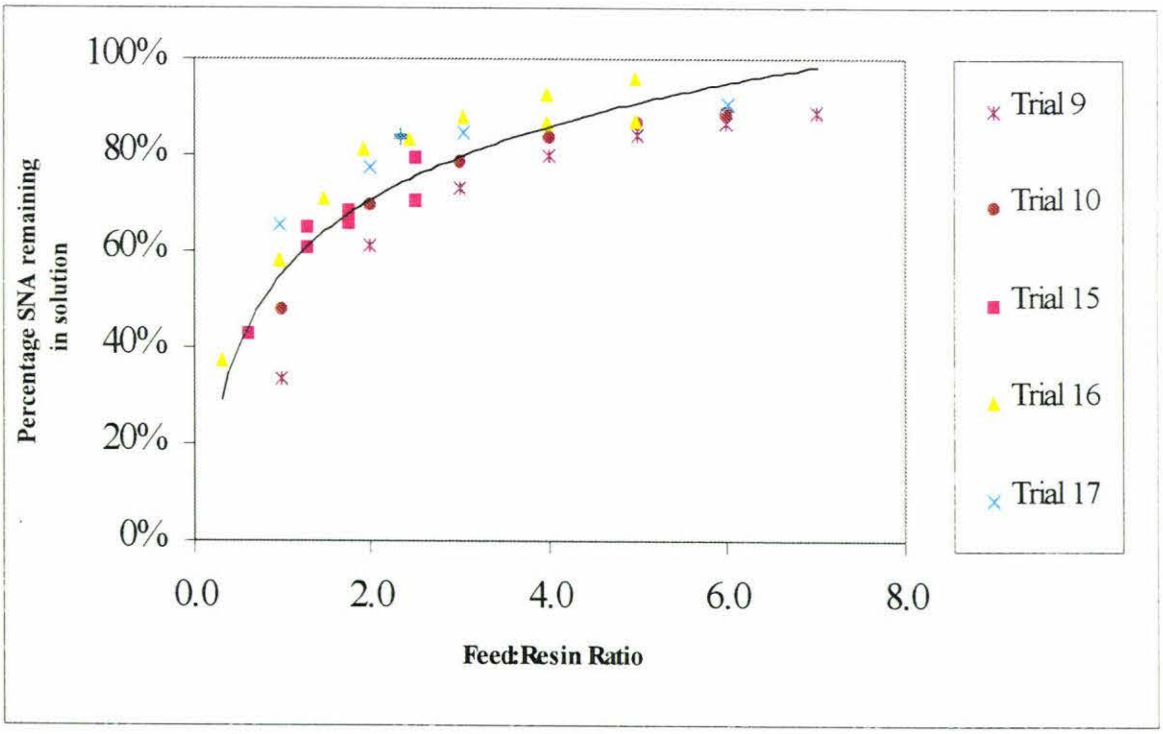
Sample Number	Sample Description
1	DMSO/Lac
2	DMSO/Lac
3	DMSO/Lac/Resin
4	DMSO/Lac/Resin
5	DMSO/Resin
6	DMSO/Resin

4.5 AQUEOUS BATCH TRIALS

4.5.1 CONFIRMING BATCH METHODOLOGY

Before investigation of the entrapment mechanism a trial was undertaken to ensure that the batch method results were comparable with the continuous results. Investigation into batch lactose adsorption in an aqueous environment was performed in trial 17. This was to allow comparison with aqueous batch (trial 17) and aqueous continuous (trials 9 and 10) trials. Results for trial 15 and 16 were included to compare the DMSO batch results. Results for trial 17 are shown in the Appendix, Pg A18. Comparisons of continuous trials (9 and 10) with batch trials (15, 16 and 17) are shown in Figure 20.

Figure 20: Percentage of SNA Remaining in Solution

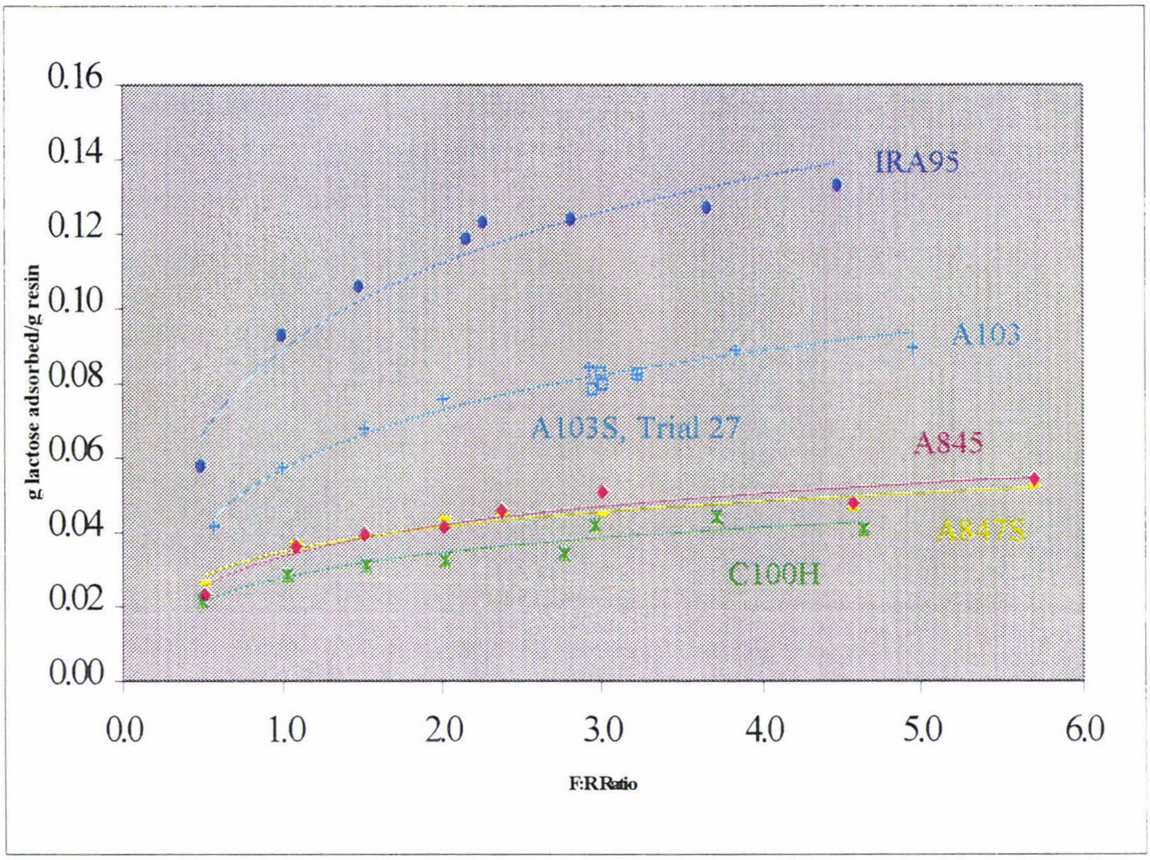


It can be seen from Figure 20 that results from batch trial results are similar to continuous trial results. The batch method was concluded to be a suitable method to approximate the continuous process. DMSO/lactose trials (15 and 16) are also similar to the aqueous trials. This suggests DMSO solvent did not alter the lactose adsorption mechanism.

4.5.2 BATCH AQUEOUS ADSORPTION TRIALS

The objective of the next group of trials was to investigate lactose adsorption with C100H (trial 21), IRA95 (trial 23), A103S (trial 25), A847S (trial 29) and A845 (trial 31) resins. This would allow investigations into the resin structure effect on the lactose adsorption. The C100H, A847S and A845 resins are all gel type resins. The IRA95 and A103S anion resins have macroporous structures. Macroporous structures are more entangled than gel structures. If the resin entrapment mechanism governs the adsorption of lactose, then the more entangled the resin structure, the greater the entrapment of lactose. It would therefore be expected that the lactose adsorption would be significantly greater in the two macroporous resins. Aqueous lactose feed solutions were used for the adsorption trials. The resin water content for resin samples were approximately 7% of the resin slurry weights (Trial 35; Appendix, Pg A60). Dilution effects of this resin water were included in all lactose adsorption calculations. Results of the adsorption trials C100H (Pg A22-23), IRA95 (Pg A24-25), A103S (Pg A31-32), A847S (Pg A48-49) and A845 (Pg A52-53) resins are shown in the Appendix. Lactose adsorption curves for all five resins are shown in Figure 21. Further results for the A103S resin from trial 27 are also shown on Figure 21. Trial 27 results are discussed in more detail in Section 4.5.3.

Figure 21: Lactose Adsorption for all resin types



From Figure 21 it can be seen that the gel structured resins (C100H, A847S and A845) had significantly less lactose adsorption than those of IRA95 and A103S, the macroporous resins. Results from trials 21 and 25 confirmed earlier findings (trial 6), that adsorption of lactose occurred predominantly on the anion resin (A103S) when compared with the cation resin (C100H). The maximum lactose adsorption for the resins investigated are shown below in Table 8.

Table 8: Maximum Lactose Adsorption

Resin	Resin Type	Maximum Lactose Adsorption (g lactose/g resin)
IRA95	Macroporous anion	0.13
A103S	Macroporous anion	0.09
A845	Gel anion	0.05
A847S	Gel anion	0.05
C100H	Gel cation	0.04

Lactose adsorption for all resins were lowest at lower F:R ratios. Earlier continuous trials had shown greatest reduction in the remaining SNA content of supernatant solutions. It was suggested that this was due to maximum lactose adsorption at the low F:R ratios. It was speculated this SNA reduction was also due to the dilution and/or channeling effects. Batch trials had confirmed low remaining SNA content was due primarily to the dilution and/or channeling effects and not lactose adsorption.

Calculations were made to compare the lactose losses on the C100H/A103S initial resin combination and the most promising alternative, C100H/A847S resin combination. Resins have different capacities to absorb the charged species from feed solutions. This adsorption capacity for each resin is termed the total exchange capacity. Total resin exchange capacities and lactose adsorption calculations for C100H, A103S and A847S are shown in Table 9. For calculations only it was assumed that 1000kg of A103S anion resin was required to demineralise a set volume of NF whey. Total exchange capacities for C100H and A847S resins were used to calculate the resin mass required, respectively. Using lactose adsorption capacities from Figure 21, the lactose losses for the demineralisation cycle were calculated. It can be seen that a combination of C100H/A847S (72 kg/cycle) would provide lactose losses 43% lower than a C100H/A103S resin combination (125.6

kg/cycle). These calculations are solely for comparison of the resin combinations. Lactose losses are dependent on the feed solution composition.

Table 9: Lactose Adsorption Calculations onto Selected Resins

Resin	Exchange Capacity meq./kg	Assumed required mass kg/cycle	Lactose Adsorption Capacity g lactose/g resin	Lactose Losses kg/cycle
C100H	4.5	889	0.04	35.6
A103S	4.0	1000	0.09	90.0
A847S	5.5	727	0.05	36.4

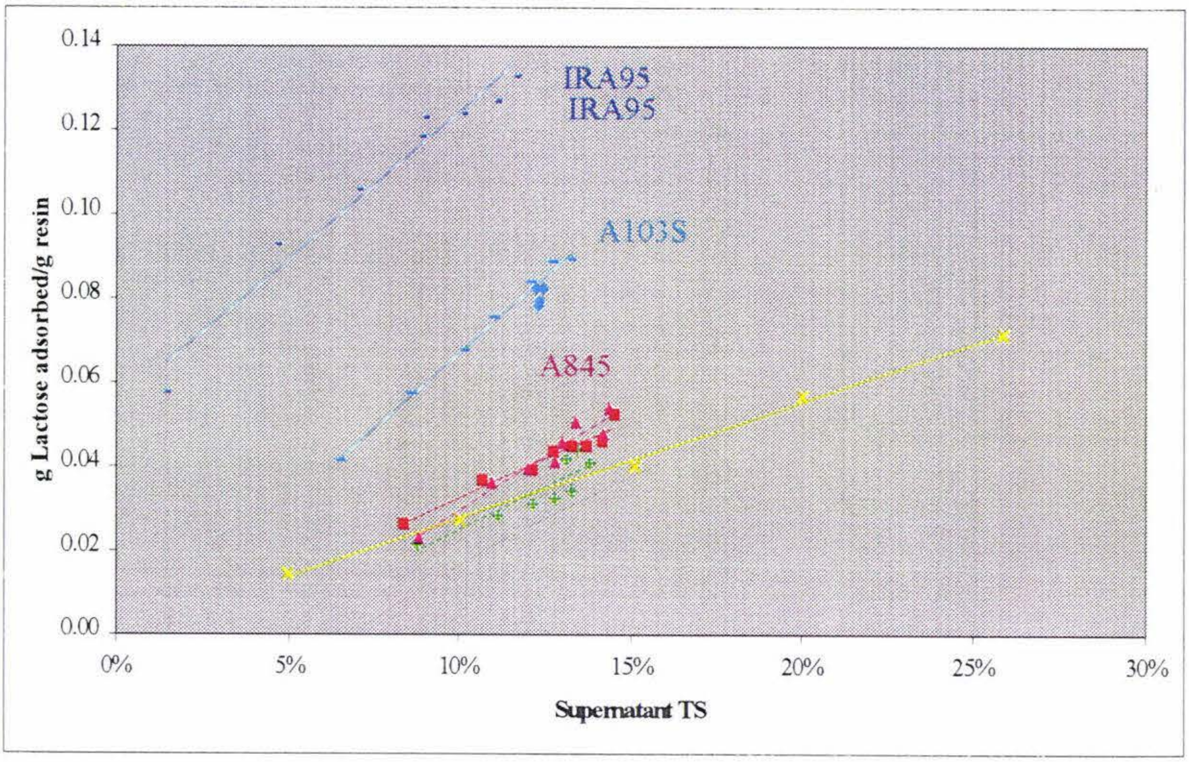
4.5.3 VARIABLE SAMPLE SIZE ADSORPTION TRIAL WITH A103S

To investigate the effect of the resin sample size at a fixed F:R ratio trial 27 was performed. In this trial a fixed F:R ratio of 3.0 was employed. Results for trial 27 are shown in the Appendix, Page A38-A39. Lactose adsorption results are shown graphically in Figure 21. The results are for the A103S adsorption curve. Trial 27 results are shown as boxes. These results are in good agreement with the earlier A103S anion trial. Lactose adsorption was independent of resin sample size.

4.5.4 VARIABLE FEED CONCENTRATION ADSORPTION TRIALS

Investigations into the effect of feed concentration on lactose adsorption on the most promising anion resin, A847S, was performed. Results for trials 33 are shown in the Appendix, Pg A54-A55. The adsorption isotherm for A847S with variable feed solution concentration is shown Figure 22. The aqueous lactose adsorption isotherms for all resins investigated are also shown in Figure 22.

Figure 22: Lactose Adsorption Isotherms for all resins



From Figure 22 it can be seen that the lactose adsorbed for the A847S variable feed concentration trial was directly proportional to the lactose feed concentration. The adsorption isotherms for all other resins are also linear. If the main mechanism was the IE mechanism, then there would be a set number of sites available on a gram of resin. Therefore the amount of lactose adsorbed would be independent of the supernatant concentration. The linearity of all resin isotherms provide evidence that the IE mechanism was not governing the adsorption of the lactose onto the resins. Lactose adsorptions onto gel resins are markedly less the adsorptions onto the macroporous resins. Hence the resin structure strongly influences the lactose adsorption. This provides evidence that the resin entrapment mechanism governs the lactose adsorption mechanism. The recovery of lactose from spent resins with water would provide further evidence that the lactose adsorption is governed by the resin entrapment mechanism.

4.5.5 LACTOSE RECOVERY TRIALS FOR IRA95 AND A103S

Lactose adsorption onto the macroporous anion resins had been established in trial 23 (IRA95) and trial 25 (A103S). Investigations into the recovery of the adsorbed lactose from these resins with water elutions were performed. The trials performed investigated the lactose recovery with the IRA95 anion resin (trial 24) and the A103S anion resin (trial 26). Spent resin samples were recovered for earlier adsorption trials (trials 23 and 25). Excess solution from each resin slurry was discarded. This was to remove any lactose from the slurries, which was not adsorbed to the resin. This small correction was to provide accurate results for lactose recovery. Failure to do this would grossly overstate the recovered lactose efficiency. Adjustment of the lactose adsorbed in both trials 23 and 25 were corrected to account for the supernatant solution discarded. Sample calculations for the correction for trials 24 and 26 are shown in the Appendix, Pg A27 and A34. The amount of lactose recovered in both trial 24 (IRA95) and trial 26 (A103S) was then calculated as a percentage of the lactose adsorbed during the adsorption trials. Full results for the two lactose recovery trials 24 and 26 are shown in the Appendix, Page A26-A30 and A33-A37, respectively. The recovery of the lactose as a function of the wash bed volume for both IRA95 and A103S resins are shown in Figure 23 and 24, respectively.

It can be seen from Figure 23 that after approximately 8 BVs of water only 80% of the adsorbed lactose was recovered. In comparison results for the A103S resin (Figure 24) show all the adsorbed lactose was recovered with approximately 5 BVs of water. Trial 24 samples 5, 6, 7 and 8 are the corresponding samples from trial 23. These samples are the four highest F:R ratio samples from the adsorption trial. For trial 26 all the samples from the adsorption trials are shown in Figure 23. Results for both trials show the F:R ratio during lactose adsorption had no significant effect on the lactose recovery.

As previously stated the demineralised product from IE processing is incorporated into a dried powder. Therefore all recovery water will require evaporative removal. Optimum recovery of adsorbed lactose is dependent on several variables. Each situation requires an independent economic assessment to identify the optimum quantity of recovery water used. For the industrial plant identified in the Introduction (Section 1.0), approximately three BVs of recovery water are used. The industrial plant flushes the exhausted columns with recovery water co-current to operation. Experimental batch lactose recovery trials were performed with the resin and recovery water agitated. Therefore trial recovery results may differ from the industrial plant performance.

Figure 23: IRA95 Anion Lactose Recovery

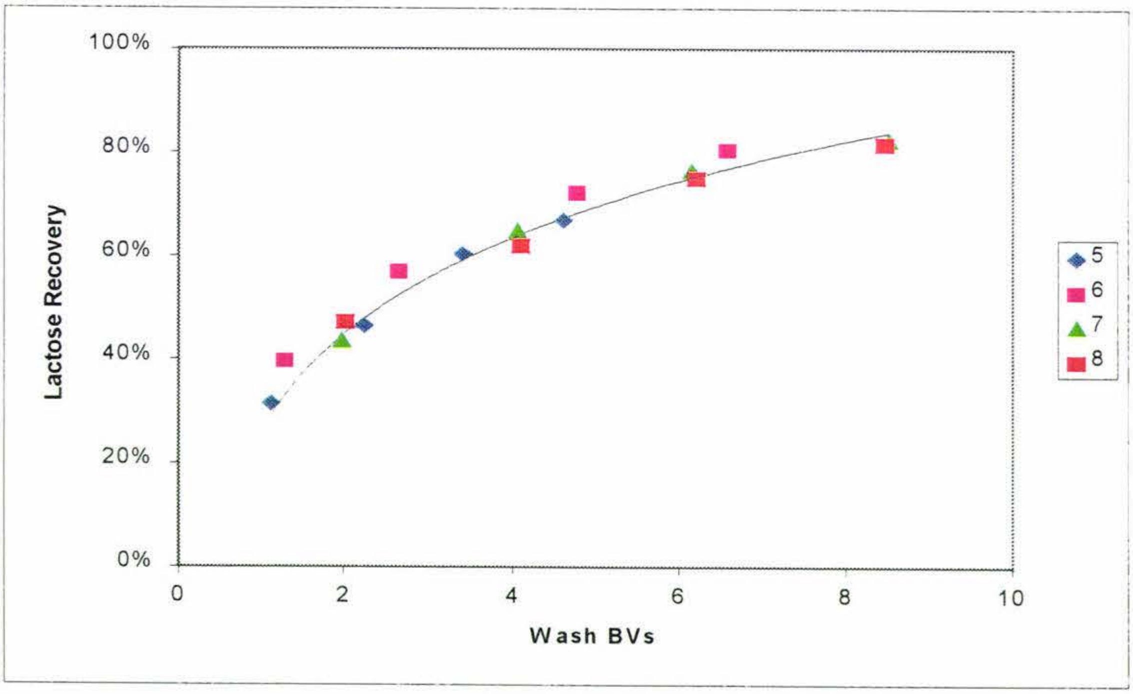
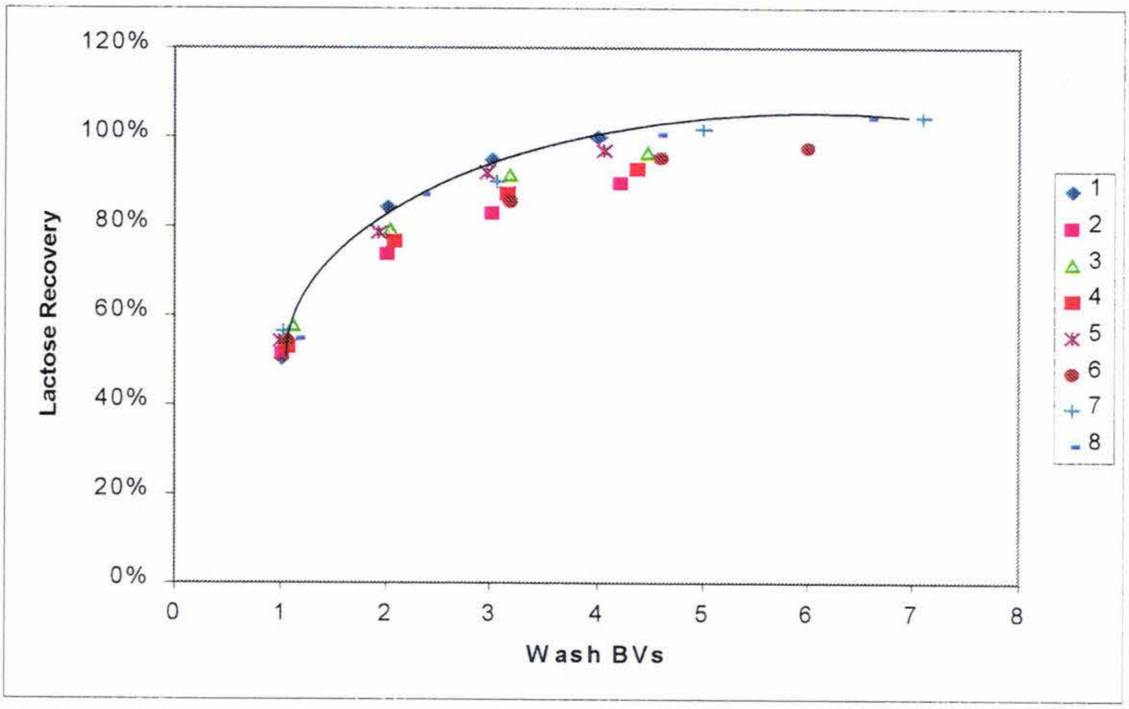


Figure 24: A103S Anion Resin Lactose Recovery

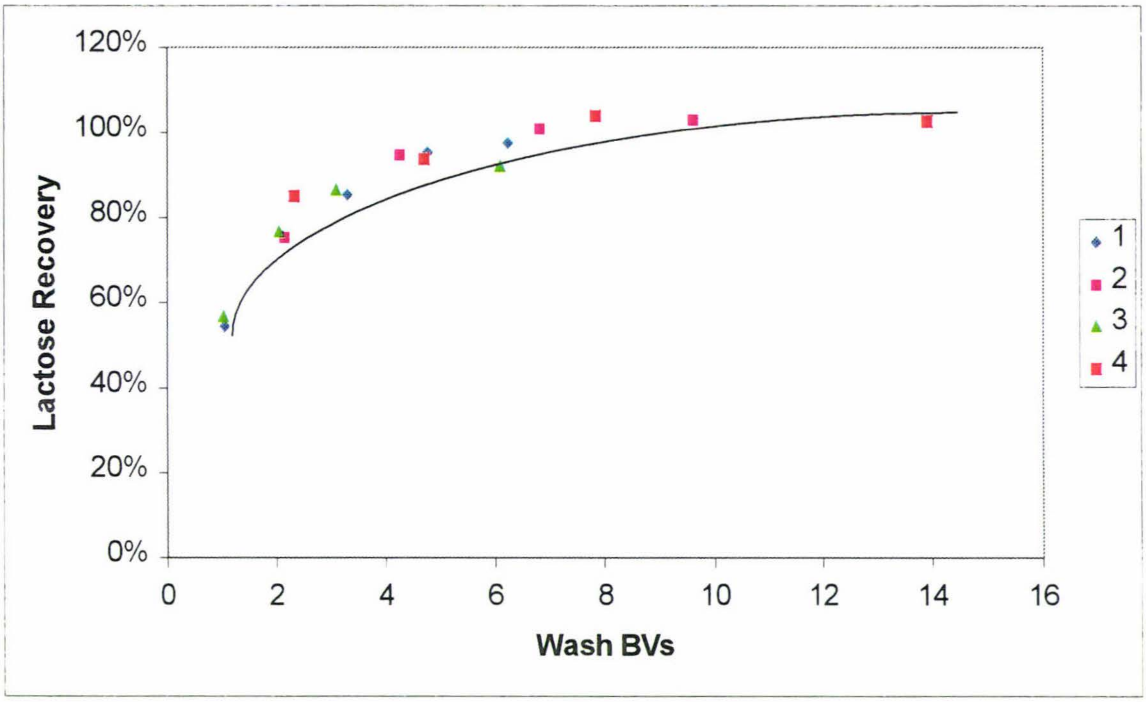


During IE processing of NF whey, regenerant chemicals are used to remove ions chemically bound to the IE resin. Backwashing with water does not remove these chemically bound ionic species. Binding of ionic lactose species to the anion resin was proposed in the lactose mutarotation mechanism. If the lactose was chemically bound to the IE resin, washing with water would not recover the lactose. Clearly all the adsorbed lactose was recovered from the A103S resin with water (trial 26). This proves that the lactose adsorbed to the anion resin was not chemically bound. Therefore lactose adsorption was not due to the lactose mutarotation mechanism.

4.5.6 VARIABLE SAMPLE SIZE LACTOSE RECOVERY TRIAL WITH A103S

The next trial (trial 27) was performed to investigate the effect of the resin sample size during lactose adsorption at a fixed F:R ratio on lactose recovery. In this trial a fixed F:R ratio of 3.0 was employed. Results for trial 27 are shown in the Appendix, Page A40-A43. Lactose recovery results are shown in Figure 25. From Figure 25 all lactose was recovered in approximately 5 BVs. This figure was in good agreement with previous A103S lactose recovery trial (trial 26) result of 5 BVs for full recovery. Lactose recovery was independent of resin sample size.

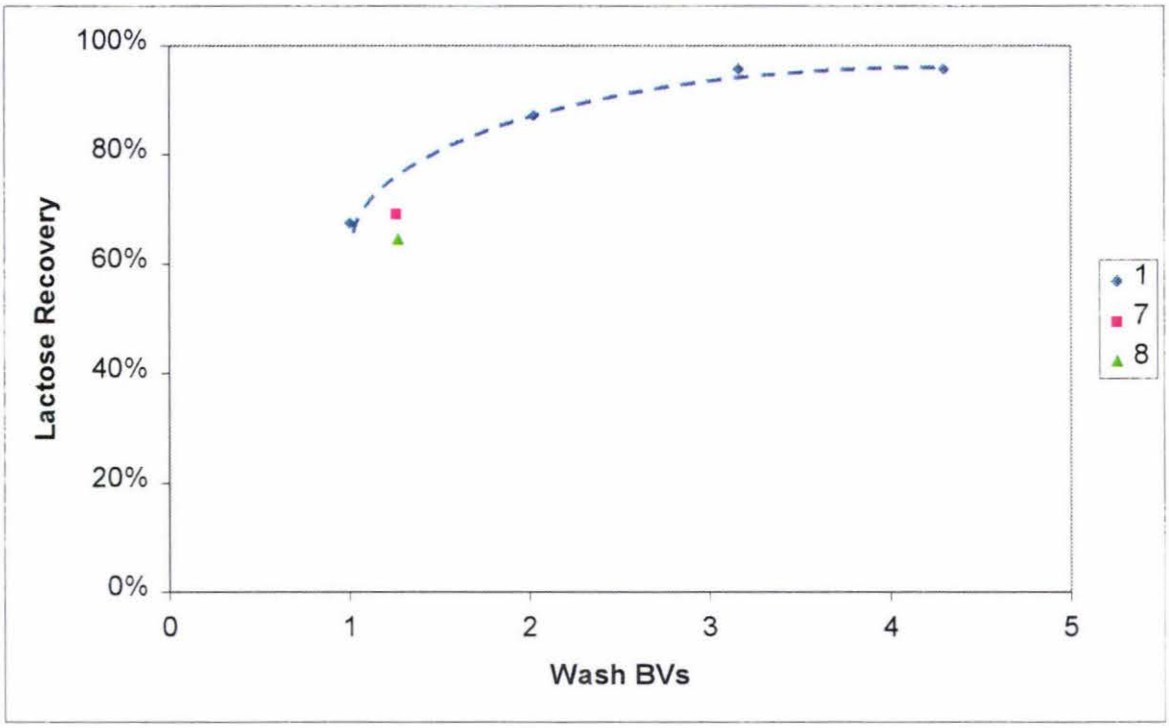
Figure 25: A103S Fixed F:R Ratio Lactose Recovery



4.5.7 A847S AND A845 LACTOSE RECOVERY TRIALS

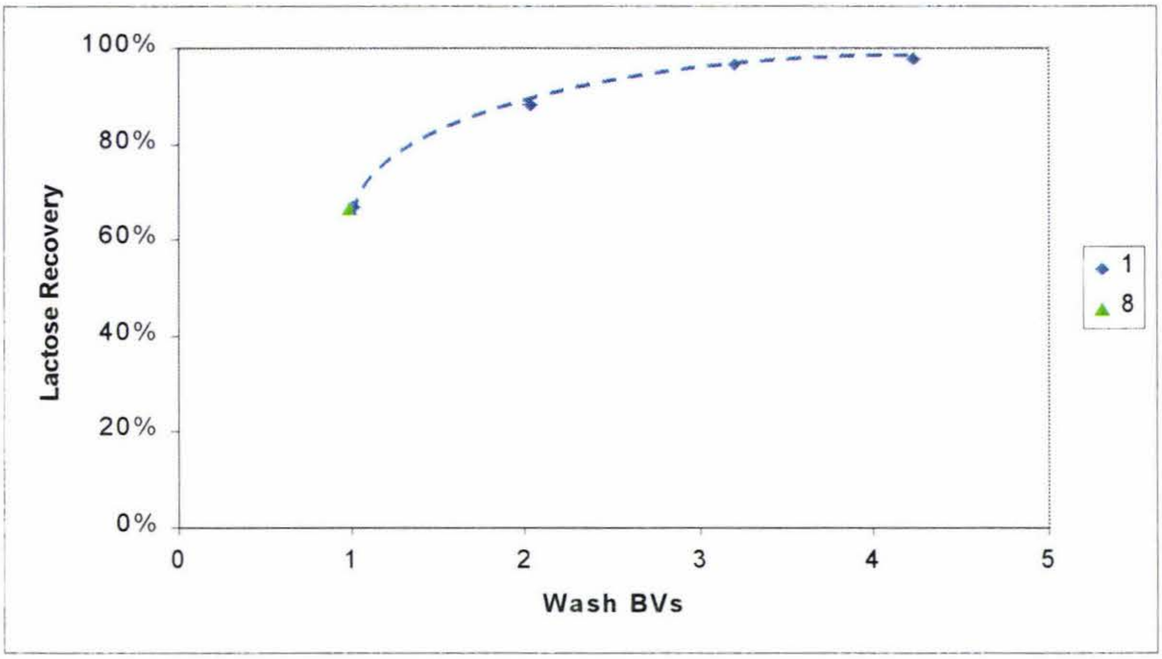
The recovery of the lactose by water from spent A847S and A845 resins was then investigated. Excess elution was discarded from the adsorption trial samples as done in previous recovery trials. Corrections to the yield calculations to allow for the discarded solution were performed as in trials 24 and 26. The results for trials 30 (A847S) and trials 32 (A845) are shown in the Appendix, Page A50-A51 and A56-57. Results for trial 30 and 32 are shown in Figures 26 and 27 respectively.

Figure 26: Lactose Recovery for A847S resin



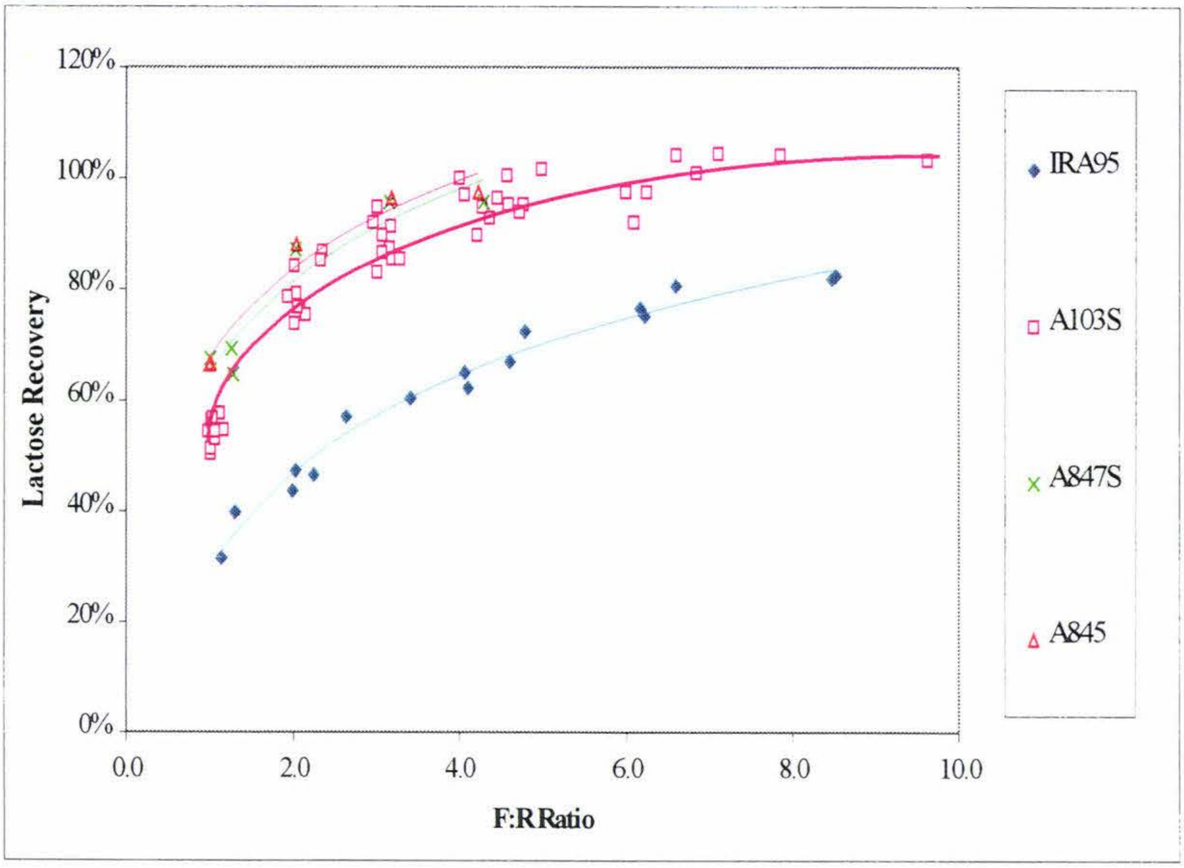
It can be seen from Figures 26 and 27 that the lactose recovery curves for both resins are similar. The majority of adsorbed lactose can be recovered with approximately 3 BVs of RO water. The F:R ratios during lactose adsorption had little effect on the lactose recovery results. Comparison of the lactose recovery curves for all the anion resins investigated are shown in Figure 28.

Figure 27: Lactose Recovery for A845 resin



It can be seen from Figure 28 that the gel type resins (A847S and A845) require less water to recover all the lactose adsorbed to the resin than the two macroporous resins (IRA95 and A103S). The majority of lactose adsorbed to the gel resins was recovered with approximately 3 BVs of water. In comparison the recovery of adsorbed lactose from the A103S resin required approximately 6 BVs of water. For the remaining macroporous resin, IRA95, lactose recovery of 80% was reached with approximately 8 BVs of water. To minimise water requirements for recovery of lactose during sweetening off, A847S or A845 resins should be employed.

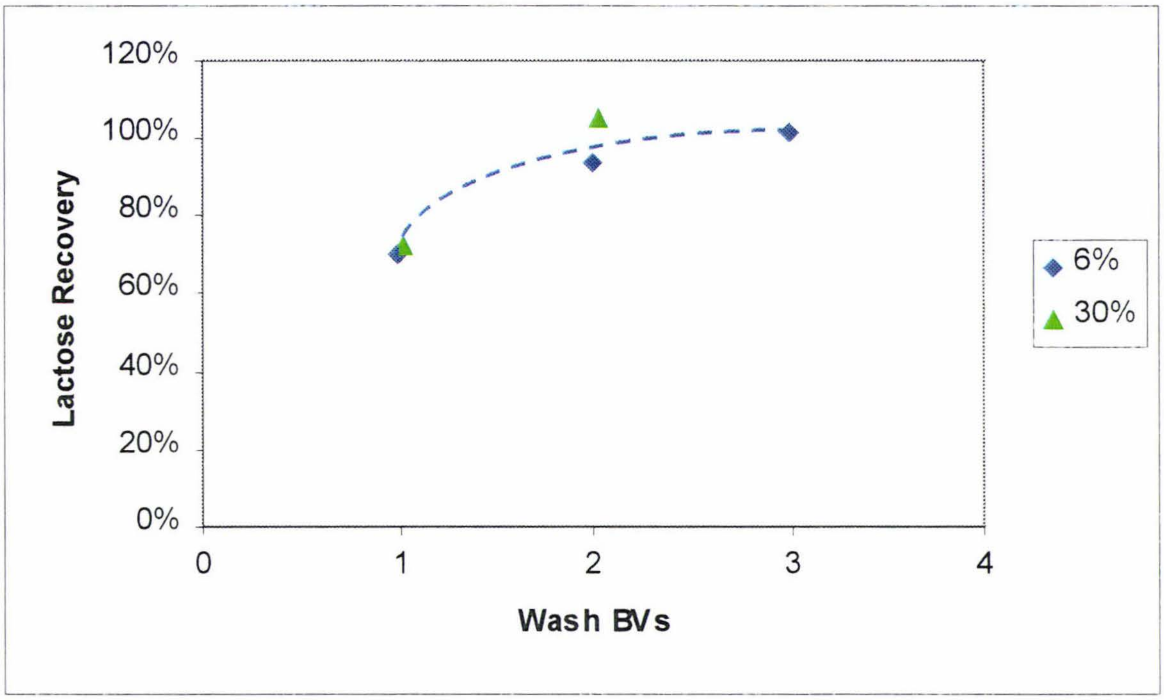
Figure 28: Lactose Recovery for all anion resins



4.5.8 VARIABLE FEED CONCENTRATION RECOVERY TRIALS

The effect of feed solution concentration on the adsorption of lactose onto the A847S resin was investigated in trial 34. Previous work has shown lactose recovery was independent of the initial F:R ratio. Therefore the two extreme concentration samples (6% and 30% TS) were investigated. Results for the recovery of the lactose are shown in Figure 29 and the Appendix, Pg A58-A59. It can be seen that there was no significant evidence to suggest that the recovery of the lactose was dependent on the lactose feed TS during adsorption.

Figure 29: Lactose Recovery for variable feed concentration samples



5.0 CONCLUSIONS

Continuous trials identified that SNA from NF whey adsorbed predominantly onto the macroporous anion resin. SNA adsorbed onto the anion resin was identified as lactose. SNA yields of 84-93% of the feed solution SNA, occurred during IE processing of NF whey or lactose solutions. Continuous trials identified lactose was adsorbing predominantly at the beginning of the IE process cycle. An alternative lactose analysis method, refractive index, was investigated for aqueous lactose determination. The refractive index was a suitable method to determine aqueous lactose content in demineralised lactose solutions. Acidification of the feed solution was performed to investigate if the mutarotation rate effected the lactose adsorption. Increases in the quantity of lactose adsorbed would indicate the mutarotation mechanism effected the lactose adsorption. Trials revealed the acidification of the feed solution did not markedly alter the quantity of lactose adsorbed. Therefore the mutarotation mechanism was not confirmed as the governing mechanism. Continuous trials had not identified the mechanism governing the lactose adsorption. In an attempt to inhibit the mutarotation mechanism during batch trials, DMSO was used. This was to allow investigation of lactose adsorption with the lactose mutarotation mechanism blocked.

A DMSO trial showing no adsorption would have conclusively proved that the mutarotation mechanism was governing lactose adsorption. The actual results showed the lactose adsorption still occurring. This could be interpreted as either that resin entrapment was the governing mechanism or that the DMSO had not stopped the mutarotation reaction. Subsequent experiments investigating the water content were not accurate enough to distinguish between the mechanisms. It was concluded aqueous solutions were the best environment to prove the resin entrapment mechanism.

Aqueous batch benchtop trials with lactose feed solutions were performed with various resins. Results revealed the gel structured resins (C100H, A847S and A845) had lactose adsorption significantly less than those of IRA95 and A103S macroporous resins (0.04, 0.05, 0.05, 0.13, 0.09 g lactose/g resin respectively). These results confirmed earlier findings that lactose adsorption occurred predominantly on the macroporous anion resin (A103S) when coupled with the gel cation resin (C100H). Calculations revealed that a combination of C100H/A847S resins would provide lactose losses 43% lower than a C100H/A103S resin combination. Lactose adsorption was independent of resin sample size at fixed F:R ratios. Lactose adsorption was directly proportional to the lactose feed concentration.

The adsorption isotherms for all resins were linearly proportional to the supernatant concentration. If the main mechanism was the IE mechanism, then there would be a set number of sites available on a gram of resin. Therefore the amount of lactose adsorbed would be independent of the supernatant concentration. The linearity of all resin adsorption isotherms provided evidence that the IE mechanism was not governing the adsorption of the lactose onto the resins. Lactose adsorption onto gel resins were markedly less the adsorption onto the macroporous resins. Hence the resin structure strongly influences the lactose adsorption. This provides evidence that the resin entrapment mechanism governs the lactose adsorption mechanism.

Recovery of lactose from spent resin with water and agitation was performed. The industrial processing plant does not agitate the recovery water and spent resin during processing. Recovery of the adsorbed lactose with water revealed the gel type resins (A847S and A845) require less water to recovery all adsorbed lactose than the two macroporous resins (IRA95 and A103S). The majority of adsorbed lactose can be recovered from A847S resin with approximately half the RO water compared with the A103S resin (approximately 3 BVs and 6 BVs of RO water, respectively). Recovery of bound ionic species requires regenerant chemicals. All adsorbed lactose from the gel anion resins was recovered with water. This reveals that the resin entrapment mechanism was governing the adsorption of lactose onto the IE resins. The recovery of lactose entrapped in the resin during washing was independent of how the lactose was adsorbed. Lactose recovery was independent of resin sample size.

Adsorption of lactose was dependent on the structure of the resins. Adsorption isotherms were linear. All adsorbed lactose onto the gel anion resins was recovered with water. These results reveal that the resin entrapment mechanism was governing the adsorption of lactose onto the IE resins.

In hindsight the DMSO results were also consistent with the resin entrapment mechanism governing the lactose adsorption. This suggests that the DMSO was stopping the mutarotation reaction without effecting the lactose adsorption.

6.0 RECOMMENDATIONS

It is recommended that to reduce lactose adsorption during IE processing by 43%, gel structured anion resin (A847S) should be coupled in series with the existing gel structured cation resin (C100H). The gel anion resin would also halve the anion water requirements during lactose recovery flushing.

7.0 REFERENCES

ANONYMOUS
"APPLEXION CORPORATION INSTRUCTION BOOK"
Unpublished Data

ARDEN, T.V. (1968)
"Water Purification by Ion Exchange"
Butterworth & Co. (New Zealand) Ltd.

BERRY, S. and POTHECARY, M.J. (1979)
"Use of Electrodialysis and Ion Exchange in combination to produce low mineral wheys and permeates"
DIGTP Whey Products Technology Section Assignment.

BYLUND, G (1995)
"Dairy Processing Handbook",
Tetra Pak Processing Systems AB, Sweden

CLELAND, H.R. (1994)
"Whey Protein Products: A Summary",
DIGTP Course Notes (Confidential).

DELANEY, R.A.M (1976)
"Demineralisation of Whey",
The Australian Journal of Dairy Technology, 31, Pg 12-17.

DELANEY, R.A.M and DONNELLY, J.K. (1976)
"Ion Exchange/Ultrafiltration Studies on Whey Complimentary Aspects",
Separation Processes by Membranes, Ion Exchange and Freeze Concentration in the Food Industry International Symposium, 13-14 March, 1975.
Paris.

DELBEKE, R. (1972)
"Assessment of certain ion-exchange resins for the demineralise of cheese whey",
Netherlands Milk Dairy Journal, 26, Pg 155-167.

DELBEKE, R. (1979)
"Purification of an ultrafiltration permeate with adsorbent and ion exchange resins",
Netherlands Milk Dairy Journal, 33, Pg 181-192.

DOWNES, T.E.H. , VENTER, B.G. and PELSTER, J.C. (1985)
"Research Progress Report on Whey Processing and Utilization",
South African Journal of Dairy Technology, 17 (2), Pg 77-81.

ENNIS, B.M. (1984)
"Introduction to whey demineralisation",
Unpublished Data. NZDRI 18, 19 April 1984.

ENNIS, B.M. and HIGGINS, J.J. (1982)
"Demineralization of Deproteinated sweet whey - an economic study",
New Zealand Journal of Dairy Science and Technology, 17, Pg 27-34.

FINER, S. I. (1993)
"Performance Improvements in Ion Exchange used for Sweet Whey Permeate Demineralisation"
Diploma of Dairy Science and Technology Dissertation, Massey University, Palmerston North.

GRANDISON, A.S. (1994)
"Applications of ion-exchange in food processing",
Food Technology International - Europe, Pages 53-57.

HERMANN, M. (1985)
"Ion Exchanges: a way to desalinate milk and dairy products"
Deutsche Milchceirtschaft, 43, Pg 1435-1439.

HERVE, D. (1974)
"Whey Treatment by Ion-exchange resins",
Journal of Process Biochemistry, February/March 1974 Pg 16-17.

HODGES, A.H. (1992)
"Overview of Milk Protein Products and Processing"
Project No: 110.01.01 NZ Dairy Group R & D Report

HOPPE, G.K.
"Organic Uptake and Fouling during demineralisation by Ion Exchange",
NZDRI Unpublished Literature

HOPPE, G.K. and HIGGINS, J.J. (1992)
"Demineralization",
Pg 91-131. In Whey and Lactose Processing. Edited by Zadow, J.G. London:
Elsevier Applied Science, London and New York.

HORTON, B.S.
Horton International, Inc. Cambridge, Massachusetts, U.S.A.
Unpublished Data

HOULDSWORTH, D.W. (1980)
"Demineralization of whey by means of ion exchange and electrodialysis",
Journal of the Society of Dairy Technology, 33 (2) Pg 45-51.

ISBELL, H.S. and PIGMAN, W. (1969)
"Mutarotation of Sugars in Solution: Part II",
Advances in Carbohydrate Chemistry and Biochemistry, 24 Pg 13-65.

JONSSON, H. (1987)
"Ion Exchange for the demineralisation of cheese whey",
Bulletin of the International Dairy Federation, 212, Pg 91-98.

KUNIN, R. (1974)
"Helpful hints in ion exchange technology",
Amber-hi-lites Report No. 138. Rohm and Haas Company, Philadelphia, Pennsylvania.

LEFEVRE, L.J. (1986)
"Ion Exchange: Problems and troubleshooting"
Journal of Chemical Engineering, July 1986, Pg 73-75.

LING E.R. (1963)
"A Text-Book of Dairy Chemistry – Volume One"
Third Edition Publishers Chapman & Hall Limited, London

LOWE, E. (1993)
"Ion Exchange: Problems and troubleshooting"
Ph.D. Thesis, Massey University, Palmerston North, New Zealand.

MASON, G. (1991)
"Whey Ion Exchange Workshop"
Seminar Notes, 29 May 1991.

MARSHALL, S.C. (1979)
"Advances in Whey Processing - Demineralisation and other Fractionation Processes",
New Zealand Journal of Dairy Science and Technology, 14, Pg 103-112.

M^{c}ELLIGOTT, D.M. (1990)
"Industrial Evaluation of Loose Reverse Osmosis",
Diploma of Dairy Science and Technology Dissertation, Massey University, Palmerston North.

M^{c}MURRAY, J.M. (1988)
"Organic Chemistry",
Second Edition. Publisher Brooks/Cole Publishing Company Belmont, California.

M^{c}NULTY, J.T. (1997)
"The many faces of Ion Exchange Resins"
Journal of Chemical Engineering, June 1997, Pg 94-100.

MITSUBISHI (1986)
Diaion. Manual of Ion Exchange Resins (I) (revised Edition). Pg 16-20; 94-99.
Mitsubishi Chemical Industries Limited, Tokyo, Japan.

NZDB (1990)
"Production of DWP from Acid Casein Wheys: Desk Study of the Meggle Casein Process", NZDB CATEGORY II FUNDED PROJECT (1990), (Confidential)

NEW ZEALAND DAIRY INDUSTRY (1993)
"Chemical Methods Manual" New Zealand Test Methods 3; June1993
New Zealand Dairy Industry, Wellington New Zealand.

OLANO, A., BERNHARD, R.A., and NICKERSON, T.A. (1977)
"Alteration in the Ratio of α- to β-Lactose Co-crystallized from Organic Solvents"
Journal of Food Science, 42(4) Pg 1066-1083.

PARRISH, F.W. , SHARPLES, P.M. , HOAGLAND, P.D. and WOYCHIK, J.H. (1979)
"Demineralisation of Cheddar Whey Ultrafiltrate with Thermally Regenerable Ion Exchange Resins: Improved Yield of α-lactose Monohydrate",
Journal of Food Science, 44(2) Pg 555-557.

POTGIETER, A.P. , DE HAAST, J., MOSTERT, J.F. and DOWNES, T.E.H. (1986)
"The Removal of Minerals from Whey",
The South African Journal of Dairy Science, 18 (3) Pg 79-89.

POTGIETER, A.P., DE HAAST, J., MOSTERT, J.F. and DOWNES, T.E.H. (1988a)
"Demineralization of Cheese Whey Using an Ammonium Bicarbonate Process II. Optimum Processing Conditions for the Cation Exchanger",
New Zealand Journal of Dairy Science and Technology, 23 Pg 195-202.

POTGIETER, A.P., DE HAAST, J., MOSTERT, J.F. and DOWNES, T.E.H. (1988b)
"Demineralization of Cheese Whey Using an Ammonium Bicarbonate Process III. Optimum Processing Conditions for the Anion Exchanger",
New Zealand Journal of Dairy Science and Technology, 23 Pg 195-202.

POTGIETER, A.P. , DE HAAST, J. , DOWNES, T.E.H. , and MOSTERT, J.F. (1988c)
"Demineralization of Cheese Whey Using an Ammonium Bicarbonate Process IV. Operating Conditions Affecting Regeneration of Cation- and Anion- Exchange Resins",
New Zealand Journal of Dairy Science and Technology, 23 Pg 203-210.

SHELLEY, S. (1997)
"Ion Exchange: Curb Water and Chemical Use"
Journal of Chemical Engineering, December 1997, Pg 117-118.

SMART, J.B. (1992)
"Proceedings: Lactose R&D Review",
10 March 1992, NZDRI Report No. AB92R02, Pg 5.

SMITH, A.C. (1998)
"Ion Exchange Process Characteristics",
Unpublished Data.

SOCIETE DES PRODUITS NESTLE S. A. (1985)
"Process for treating dairy by-products"
New Zealand Patent 211112.

TENNENT R. M. (1986)
"Science Data Book"
Tenth Edition. Publisher Oliver & Boyd, Edinburgh.

APPENDIX

Trial 6

Description: Continuous, Cation/Anion, NF Whey and Ambient temperature

Objective: Investigate Solids Non Ash (SNA) absorption.

Trial Notes: Cation sample (65 mL) collected after 1.2 BV had passed through the cation column.

Appendix Table 6a: Results

Sample Description	Sample Number	Time Collected (minutes)	Conductivity (μS/cm)	pH	TS (%)	Volume Collected (mL)	TS Collected (g)
Feed			4750	6.2	17.3%		
Cation			4550	2.04	16.2%		
Anion	1	8	7	4.85	3.8%	65.6	2.49
	2	8	69	3.82	12.6%	65.6	8.27
	3	8	219	3.21	15.0%	65.6	9.84
	4	8	309	3.02	15.6%	65.6	10.23
	5	8	387	3.17	15.7%	65.6	10.30
	6	8	486	2.83	16.0%	65.6	10.50
	7	8	523	2.87	16.0%	65.6	10.50
	8	8	606	2.99	14.0%	65.6	9.18
	9	8	873	2.76	5.2%	65.6	3.41
	10	8	732	2.78	1.6%	65.6	1.05
	11	8	610	2.88	0.6%	65.6	0.39
Total						721.6	76.16

Appendix Table 6b: Product Calculations

Time taken to pass through cation column	5	minutes
Time taken to pass through anion column	7	minutes
Changed feed to DI water at end of sample	6	
TS Yield	85%	
Solids Non-Ash Yield	90%	
Average Conductivity	438	μS/cm
Average TS	10.6%	

Trial 7

Description: Continuous, Cation/Anion, NF Whey and Ambient temperature

Objective: Further investigate Solids Non Ash (SNA) absorption on uninterrupted anion results.

Appendix Table 7a: Results

Sample Description	Sample Number	Time Collected (minutes)	Conductivity (μS/cm)	pH	TS (%)	Volume Collected (mL)	TS Collected (g)
Feed			4750	6.23	17.3%		
Anion	1	8	9	4.90	11.0%	65.6	7.22
	2	8	14	4.55	14.0%	65.6	9.18
	3	8	21	4.29	14.9%	65.6	9.77
	4	8	49	3.96	15.3%	65.6	10.04
	5	9	92	3.88	15.7%	73.8	11.59
	6	8	118	3.83	16.0%	65.6	10.50
	7	8	150	3.51	15.9%	65.6	10.43
	8	8	188	3.43	16.0%	65.6	10.50
	9	8	224	3.54	16.1%	65.6	10.56
	10	8	267	3.32	16.1%	65.6	10.56
	11	8	322	3.41	16.0%	65.6	10.50
	12	8	320	3.52	7.2%	65.6	4.72
	13	8	191	3.56	3.0%	65.6	1.97
	14	8	137	3.60	1.7%	65.6	1.12
	15	8	117	3.64	1.0%	65.6	0.66
	16	7	112	3.56	0.5%	57.4	0.29
Total						1,049.6	119.6

Appendix Table 7b: Product Calculations

Time taken to pass through cation column	9	minutes
Time taken to pass through anion column	16	minutes
Changed feed to DI water after	760	ML of feed solution
Yield	87%	
Solids Non-Ash Yield	91%	
Average Conductivity	146	μS/cm
Average TS	11.4%	

Trial 8

Description: Continuous, Cation/Anion, NF Whey and Ambient temperature

Objective: Repeat trial 7 to verify and quantify lactose absorption.

Appendix Table 8a: Results

Sample Description	Sample Number	Time Collected (minutes)	Conductivity (μS/cm)	pH	TS (%)	Volume Collected (mL)	TS Collected (g)
Feed			4750	6.23	17.3%		
Anion	1	8	14	4.90	8.3%	65.6	5.44
	2	8	19	4.44	12.5%	65.6	8.20
	3	8	38	3.78	14.5%	65.6	9.51
	4	8	93	3.62	15.1%	65.6	9.91
	5	9	137	3.51	15.6%	73.8	11.51
	6	8	161	3.45	15.9%	65.6	10.43
	7	8	189	3.39	16.0%	65.6	10.50
	8	8	231	3.34	16.2%	65.6	10.63
	9	8	243	3.30	16.3%	65.6	10.69
	10	8	274	3.25	16.1%	65.6	10.56
	11	8	325	3.10	13.1%	65.6	8.59
	12	8	313	3.03	5.0%	65.6	3.28
	13	8	279	3.10	2.2%	65.6	1.44
	14	8	213	3.29	1.2%	65.6	0.79
	15	7	191	3.31	0.5%	57.4	0.29
Total						984.0	111.77

Appendix Table 8b: Product Calculations

Time taken to pass through cation column	8.5	minutes
Time taken to pass through anion column	15.5	minutes
Changed feed to DI water at end of sample	8	
Yield	85%	
Solids Non-Ash Yield	90%	
Average Conductivity	181	μS/cm
Average TS	11.4%	

Trial 9

Description: Continuous, Anion, Lactose and Ambient temperature

Objective: Prove pure lactose feed solution will cause lactose absorption in the anion column. The anion column used separately.

Trial Notes: Collected samples in 100 mL containers.

Appendix Table 9a: Results

Sample Description	Sample Number	Time Collected (minutes)	Conductivity (μS/cm)	PH	TS (%)	Volume Collected (mL)	TS Collected (g)
Feed			180	6.57	15.0%		
Anion	1	8	180	10.03	5.0%	100.0	5.00
	2	8	140	9.86	13.3%	100.0	13.30
	3	8	162	9.82	14.6%	100.0	14.60
	4	8	136	9.79	15.0%	100.0	15.00
	5	9	136	9.74	15.0%	100.0	15.00
	6	8	137	9.75	15.0%	100.0	15.00
	7	8	137	9.81	15.0%	100.0	15.00
	8	8	139	9.80	11.2%	100.0	11.20
	9	8	171	10.37	2.5%	100.0	2.45
Total						900.0	106.55

Appendix Table 9b: Product Calculations

Changed feed to DI water at end of sample	7.35	
SNA Yield	92%	
Average Conductivity	149	μS/cm
Average TS	11.8%	

Trial 10

Description: Continuous, Anion, Acidified Lactose and Ambient temperature

Objective: Investigate effect of feed lactose acidification on lactose absorption.

Appendix Table 10a: Results

Sample Description	Sample Number	Time Collected (minutes)	Conductivity (μS/cm)	pH	TS (%)	Volume Collected (mL)	TS Collected (g)
Feed			37000	0.99	16.6%		
Anion	1	8	241	10.4	8.0%	100.0	8.00
	2	8	188	10	15.2%	100.0	15.20
	3	8	267	10	16.1%	100.0	16.10
	4	8	175	9.92	16.4%	100.0	16.40
	5	9	167	9.85	16.5%	100.0	16.50
	6	8	163	9.86	16.4%	100.0	16.40
	7	8	188	9.82	6.2%	100.0	6.20
	8	8	263	10.7	1.4%	100.0	1.40
	9	8	277	10.7	0.6%	100.0	0.60
Total						900.0	96.80

Appendix Table 10b: Product Calculations

Changed feed to DI water at end of sample	5.91	
SNA Yield	93%	
Average Conductivity	214	μS/cm
Average TS	10.8%	

Trial 11

Description: Continuous, Anion, NF Whey and Ambient temperature

Objective: Repeat continuous NF whey trials to ensure cation/anion SNA yield results are similar to anion SNA results.

Appendix Table 11a: Results

Sample Description	Sample Number	Time Collected (minutes)	Conductivity (μS/cm)	pH	TS (%)	Volume Collected (mL)	TS Collected (g)
Feed			6310	6.09	20.2%		
Anion	1	8	1510	11.70	0.4%	100.0	0.40
	2	8	4500	11.37	14.4%	100.0	14.40
	3	8	4910	10.67	19.4%	100.0	19.40
	4	8	5420	9.74	20.2%	100.0	20.20
	5	9	5710	8.57	20.0%	100.0	20.00
	6	8	4310	7.87	9.0%	100.0	9.00
	7	8	1680	7.60	1.7%	100.0	1.70
Total						700.0	85.10

Appendix Table 11b: Product Calculations

Changed feed to DI water at end of sample	4.5	
Yield	87%	
Solids Non-Ash Yield	93%	
Average Conductivity	4006	μS/cm
Average TS	12.2%	

Trial 12

Description: Continuous, Anion, Acidified Lactose and Ambient temperature

Objective: Investigate effect of feed lactose acidification on lactose absorption.

Appendix Table 12a: Results

Sample Description	Sample Number	Time Collected (minutes)	Conductivity (μS/cm)	pH	TS (%)	Volume Collected (mL)	TS Collected (g)
Feed			10300	0.97	15.0%		
Anion	1	8	1840	11.68	1.1%	100.0	1.10
	2	8	303	10.27	11.6%	100.0	11.60
	3	8	250	10.06	14.3%	100.0	14.30
	4	8	248	10.08	14.8%	100.0	14.80
	5	9	247	10.07	15.0%	100.0	15.00
	6	8	250	10.08	15.0%	100.0	15.00
	7	8	255	10.09	14.3%	100.0	14.30
	8	8	260	10.43	3.8%	100.0	3.80
	9	8	313	10.89	0.5%	100.0	0.50
Total						900.0	90.40

Appendix Table 12b: Product Calculations

Changed feed to DI water after	630	ML of feed solution
SNA Yield	91%	
Average Conductivity	485	μS/cm
Average TS	10.0%	

Trial 13

Description: Continuous, Anion, Lactose, Ambient temperature and Reduced throughput

Objective: Focus on when the lactose absorption occurs in the cycle by only processing three bed volumes.

Appendix Table 13a: Results

Sample Description	Sample Number	Conductivity (μS/cm)	pH	TS (%)	Volume Collected (mL)	TS Collected (g)
Feed				15.1%		
	0	0	0	0.0%	-	-
Anion	1	248	10.16	1.5%	100.0	1.50
	2	218	9.84	11.7%	100.0	11.70
	3	187	9.71	14.4%	100.0	14.40
	4	172	9.74	14.8%	100.0	14.80
	5	170	9.74	15.3%	100.0	15.30
	6	147	9.83	7.9%	100.0	7.90
	7	151	10.22	1.3%	100.0	1.30
Total					700.0	66.90

Appendix Table 13b: Product Calculations

Changed feed to DI water at end of sample	5	
SNA Yield	84%	
Average Conductivity	185	μS/cm
Average TS	9.6%	

Trial 11 – Refraction Investigation

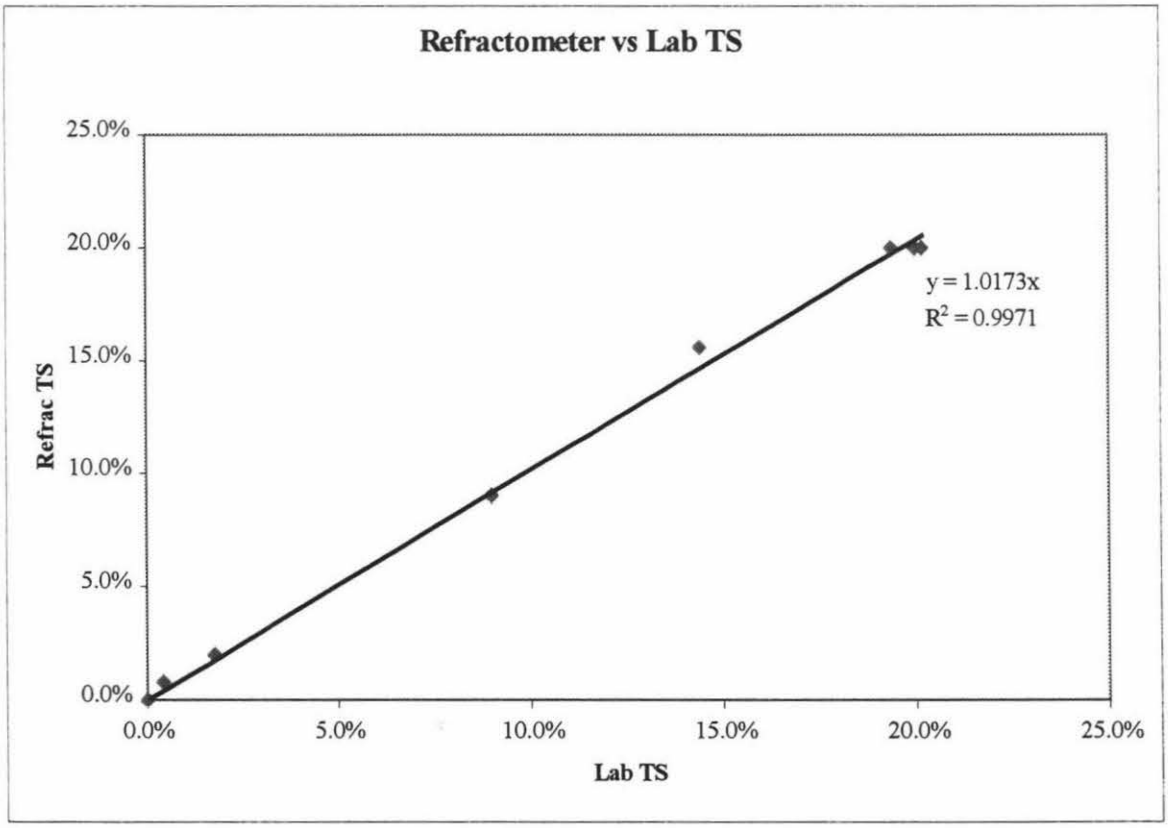
Description: Continuous, Anion, NF Whey and Ambient temperature

Objective: Compare Oven TS measurement with Refractive TS.

Appendix Table 11c: TS Results

Sample Number	Oven TS	Refractive Measurement
1	0.4%	0.8%
2	14.4%	15.6%
3	19.4%	20.0%
4	20.2%	20.0%
5	20.0%	20.0%
6	9.0%	9.0%
7	1.7%	2.0%

Appendix Figure 1



Trial 12 – Refraction Investigation

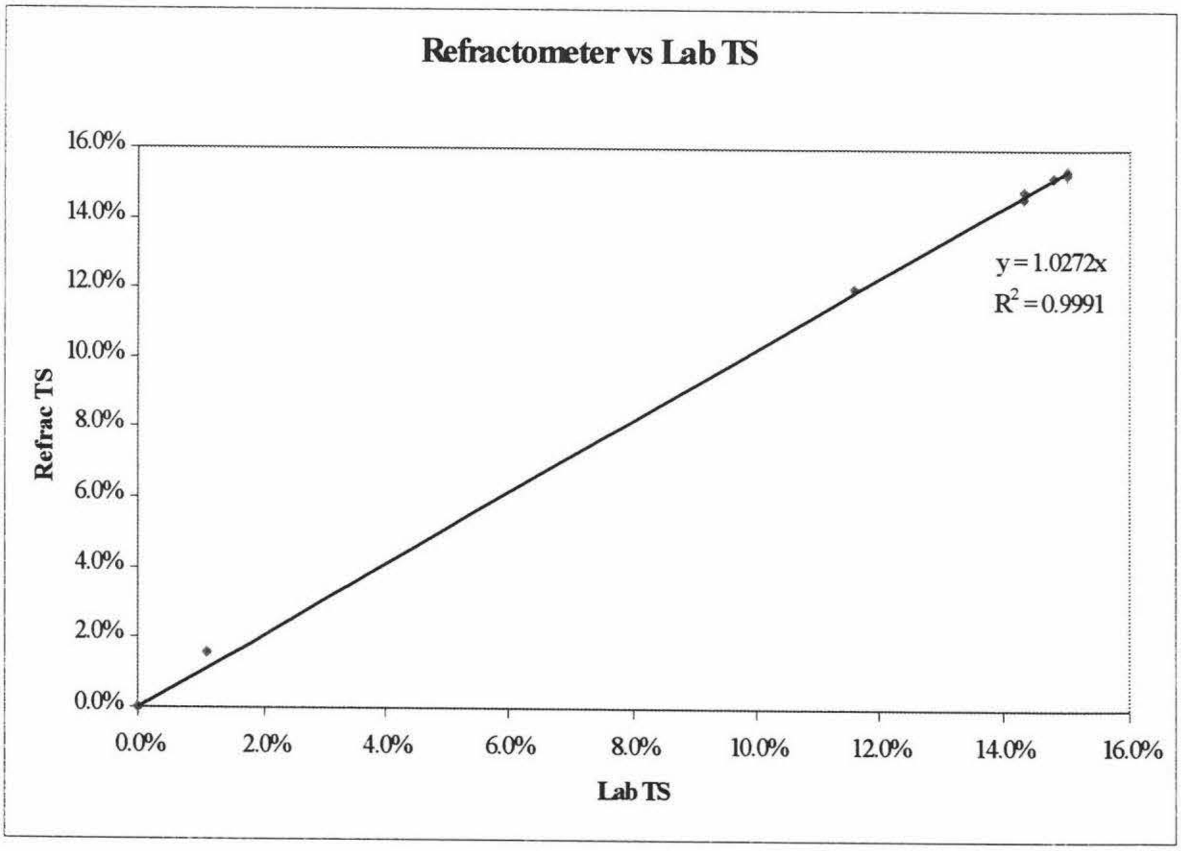
Description: Continuous, Anion, Acidified Lactose and Ambient temperature

Objective: Compare Oven TS measurement with Refractive TS.

Appendix Table 12c: TS Results

Sample Number	Oven TS	Refractive Measurement
1	1.1%	1.6%
2	11.6%	12.0%
3	14.3%	14.8%
4	14.8%	15.2%
5	15.0%	15.3%
6	15.0%	15.4%
7	14.3%	14.6%
8	3.8%	4.2%
9	0.5%	0.9%

Appendix Figure 2



Trial 13 – Refraction Investigation

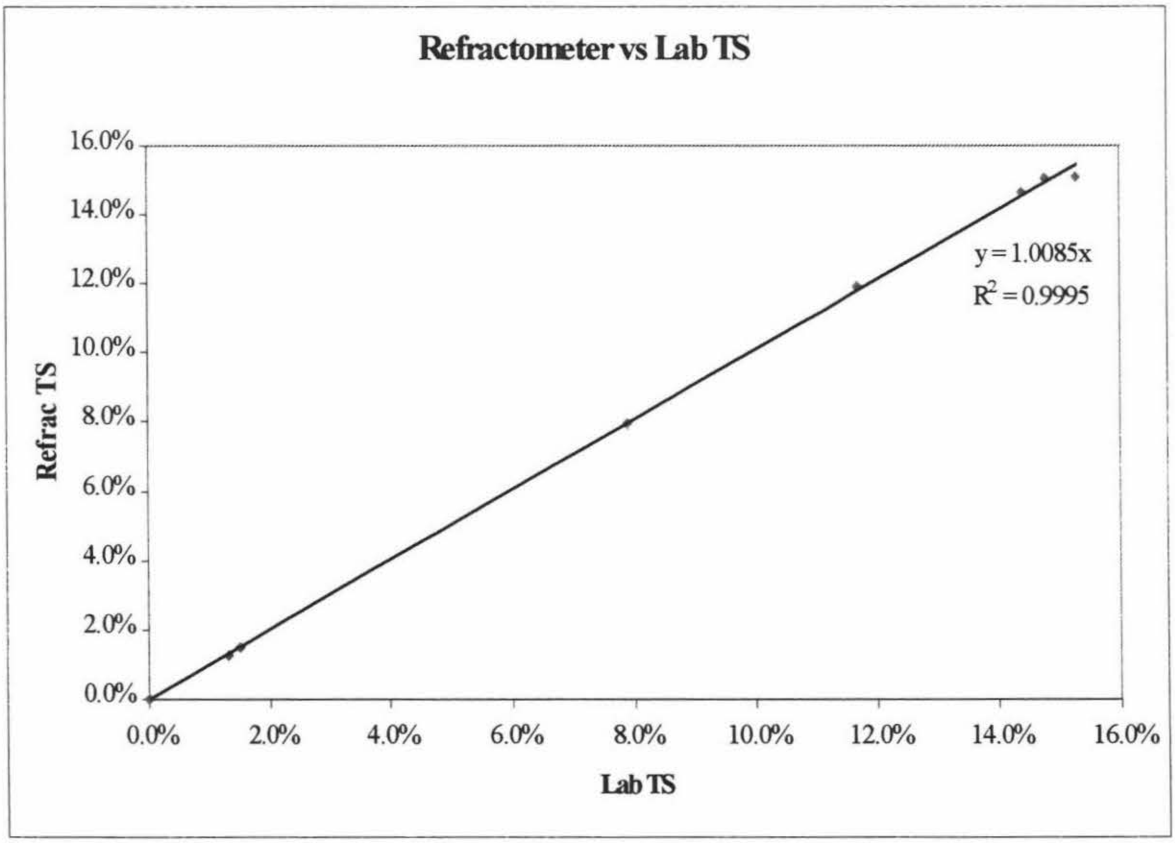
Description: Continuous, Anion, Lactose, Ambient temperature and Reduced throughput

Objective: Compare Oven TS measurement with **mixed** Refractive TS.

Appendix Table 13c: TS Results

Sample Number	Oven TS	Refractive Measurement
1	1.5%	1.5%
2	11.7%	11.9%
3	14.4%	14.7%
4	14.8%	15.1%
5	15.3%	15.1%
6	7.9%	8.0%
7	1.3%	1.3%

Appendix Figure 3



Trial 14

Description: Batch, Anion, DMSO/Lactose, Ambient temperature

Objective: Investigate trials with DMSO/lactose solution methods

Trial Notes: Samples were heated for 1 hour weighed, then heated for a further 25 minutes and reweighed.
Lactose concentration in lactose/DMSO solution = 12.19%
Oven Method modified for DMSO, evaporate at 130°C and under 60 kPa vacuum until solution has caramelised and does not flow.
Standard samples were produced to investigate refractometer readings.
No reading occurred on samples.

Appendix Table 14a: First Weighing Results

Sample	Moist Resin	DMSO/lactose Solution	Feed:Resin	Dish Weight	Sample + Dish	Sample	Ashed Sample	TS
Feed		7.00		11.33	14.79	3.46	11.80	13.57%
1	9.9709	21.82	2.2	9.12	14.05	4.94	9.69	11.61%
2	9.9709	21.82	2.2	10.10	13.64	3.54	10.48	10.71%
3	9.9709	21.82	2.2	11.41	14.65	3.24	11.75	10.67%

Appendix Table 14b: First Weighing Product Calculations

Sample	Lactose Remaining In Solution	Lactose Absorbed to resin	g Lactose added to resin	g Lactose remaining in solution	g Lactose absorbed to resin	g Lactose Absorbed/ g resin
Feed	100.0%					
1	85.6%	14.4%	2.96	2.53	0.43	0.043
2	78.9%	21.1%	2.96	2.34	0.62	0.063
3	78.6%	21.4%	2.96	2.33	0.63	0.063

Appendix Table 14c: Second Weighing Results

Sample	Moist Resin	DMSO/lactose Solution	Feed:Resin	Dish Weight	Sample + Dish	Sample	Ashed Sample	TS
Feed				11.33	14.79	3.46	11.75	12.1%
1	9.9709	21.82	2.2	9.12	14.05	4.94	9.61	9.9%
2	9.9709	21.82	2.2	10.10	13.64	3.54	10.44	9.6%
3	9.9709	21.82	2.2	11.41	14.65	3.24	11.72	9.7%

Appendix Table 14d: Second Weighing Product Calculations

Sample	Lactose Remaining In Solution	Lactose Absorbed to resin	g Lactose added to resin	g Lactose remaining in solution	g Lactose absorbed to resin	g Lactose Absorbed/ g resin
Feed	100.0%					
1	81.9%	18.1%	2.63	2.16	0.48	0.048
2	79.3%	20.7%	2.63	2.09	0.54	0.055
3	80.6%	19.4%	2.63	2.12	0.51	0.051

Sample Calculations to predict results for second weighing from first weighing:

$$\frac{\text{First Weighing Sample 2 TS}}{\text{First Weighing Feed TS}} * \text{Actual Feed TS} = \text{Sample 2 TS}$$

$$\frac{10.7\%}{13.6\%} * 12.2\% = 9.6\%$$

This approximation can be used when the Feed TS does not match the oven TS method.

Trial 15

Description: Batch, Anion, DMSO/Lactose, Ambient temperature

Objective: Investigate trials with DMSO/lactose solution over greater F:R Ratio range

Trial Notes: Lactose concentration in lactose/DMSO solution = 14.63%

Appendix Table 15a: Results

Sample	Moist Resin	DMSO/lactose Solution	Feed:Resin	Dish Weight	Sample + Dish	Sample	Ashed Sample	TS
Feed 1		7.37		11.24	13.92	2.67	11.73	18.14%
Feed 2		7.37		10.91	14.42	3.51	11.55	18.44%
1	9.94	25.06	2.5	9.12	12.01	2.90	9.54	14.6%
2	9.94	25.06	2.5	11.10	14.33	3.22	11.52	12.9%
3	10.09	17.87	1.8	10.75	13.40	2.64	11.07	12.1%
4	10.09	17.87	1.8	11.33	14.64	3.31	11.74	12.5%
5	10.03	12.92	1.3	9.04	11.92	2.88	9.36	11.1%
6	10.03	12.92	1.3	10.52	16.21	5.70	11.19	11.9%
7	10.12	6.26	0.6	10.11	13.16	3.05	10.35	7.8%

Appendix Table 15b: Calculations

Sample	Lactose Remaining in Solution	Lactose Absorbed to resin	g Lactose added to resin	g Lactose Remaining in solution	g Lactose absorbed to resin	g Lactose Absorbed/ g resin
Feed 1	100.0%					
Feed 2	100.0%					
1	79.7%	20.3%	4.58	3.65	0.93	0.093
2	70.5%	29.5%	4.58	3.23	1.35	0.136
3	66.0%	34.0%	3.27	2.16	1.11	0.110
4	68.5%	31.5%	3.27	2.24	1.03	0.102
5	60.7%	39.3%	2.36	1.43	0.93	0.093
6	65.1%	34.9%	2.36	1.54	0.82	0.082
7	42.9%	57.1%	1.14	0.49	0.65	0.065

Trial 16

Description: Batch, Anion, DMSO/Lactose, Ambient temperature

Objective: Investigate trials with DMSO/lactose solution methods over a greater F:R range.
Investigate moisture evolution also.

Trial Notes: Preheated oven before entering samples.
Lactose concentration in lactose/DMSO solution = 15.00%

Appendix Table 16a: Results

Sample	Moist Resin	DMSO/lactose Solution	Feed:Resin	Dish Weight	Sample	Ashed Sample	TS
Feed		1.00		10.11	3.04	10.63	17.11%
1	2.00	1.50	0.7	All added liquid absorbed by the resin.			
2	2.18	2.13	1.0	11.10	0.54	11.16	10.0%
3	2.12	3.17	1.5	10.96	2.13	11.22	12.2%
4	2.06	3.99	1.9	9.15	2.59	9.51	13.9%
5	2.07	5.05	2.4	11.35	2.89	11.76	14.3%
6	2.05	6.22	3.0	9.04	1.81	9.31	15.1%
7	2.04	8.12	4.0	10.76	2.31	11.10	14.8%
8	2.04	8.12	4.0	10.74	2.37	11.11	15.9%
9	2.19	10.88	5.0	10.52	3.12	10.98	14.9%
10	6.52	2.04	0.3	11.31	0.87	11.37	6.4%

Appendix Table 16b: Product Calculations

Sample	Lactose Remaining in Solution	Lactose Absorbed to resin	g Lactose added to resin	g Lactose remaining in solution	g Lactose Absorbed To resin	g Lactose Absorbed/ g resin
Feed 1	100.0%					
1						
2	58.4%	41.6%	0.37	0.21	0.15	0.070
3	71.2%	28.8%	0.54	0.39	0.16	0.074
4	81.2%	18.8%	0.68	0.55	0.13	0.062
5	83.4%	16.6%	0.86	0.72	0.14	0.070
6	88.0%	12.0%	1.06	0.94	0.13	0.062
7	86.6%	13.4%	1.39	1.20	0.19	0.091
8	92.9%	7.1%	1.39	1.29	0.10	0.049
9	87.3%	12.7%	1.86	1.63	0.24	0.108
10	37.3%	62.7%	0.35	0.13	0.22	0.034

Appendix Table 16c: Moisture Results

Sample	Karl Fisher Moisture
Feed 1	5.5%
7	14.9%

Trial 16 Continued – Second Reweighing

Description: Batch, Anion, DMSO/Lactose, Ambient temperature

Objective: Investigate trials with DMSO/lactose solution methods over a greater F:R range.
Investigate moisture evolution also.

Trial Notes: Lactose concentration in lactose/DMSO solution = 15.00%

Appendix Table 16d: Results

Sample	Moist Resin	DMSO/lactose Solution	Feed:Resin	Dish Weight	Sample	Ashed Sample	TS
Feed		1.00		10.11	3.04	10.61	16.65%
1	2.00	1.50	0.7				
2	2.18	2.13	1.0	11.10	0.54	11.16	9.9%
3	2.12	3.17	1.5	10.96	2.13	11.21	11.8%
4	2.06	3.99	1.9	9.15	2.59	9.50	13.5%
5	2.07	5.05	2.4	11.35	2.89	11.75	13.8%
6	2.05	6.22	3.0	9.04	1.81	9.31	14.8%
7	2.04	8.12	4.0	10.76	2.31	11.09	14.3%
8	2.04	8.12	4.0	10.74	2.37	11.09	15.0%
9	2.19	10.88	5.0	10.52	3.12	10.98	14.6%
10	6.52	2.04	0.3	11.31	0.87	11.37	6.4%

Appendix Table 16e: Product Calculations

Sample	Lactose Remaining in Solution	Lactose Absorbed to resin	g Lactose added to resin	g Lactose remaining in solution	g Lactose absorbed to resin	g Lactose Absorbed/ g resin
Feed	100.0%					
1						
2	59.8%	40.2%	0.36	0.21	0.14	0.065
3	70.6%	29.4%	0.53	0.37	0.15	0.073
4	81.0%	19.0%	0.66	0.54	0.13	0.061
5	83.1%	16.9%	0.84	0.70	0.14	0.069
6	88.9%	11.1%	1.04	0.92	0.11	0.056
7	86.2%	13.8%	1.35	1.17	0.19	0.092
8	90.1%	9.9%	1.35	1.22	0.13	0.065
9	88.0%	12.0%	1.81	1.59	0.22	0.099
10	38.5%	61.5%	0.34	0.13	0.21	0.032

Trial 17

Description: Batch, Anion, Aqueous Lactose, Ambient temperature

Objective: To obtain batch aqueous lactose results. Then allow comparison with batch DMSO/lactose results and continuous results.

Trial Notes: Lactose concentration in lactose/DMSO solution = 14.15%

Appendix Table 17a: Results

Sample	Moist Resin	DMSO/lactose Solution	Feed:Resin	Dish Weight	Sample	Ashed Sample	TS
Feed		1.00		24.36	12.88	26.22	14.4%
1	4.29	4.16	1.0	27.09	2.02	27.28	9.4%
2	2.66	5.29	2.0	24.34	3.01	24.68	11.2%
3	2.00	6.07	3.0	27.99	4.07	28.49	12.2%
4	2.55	15.39	6.0	23.98	12.41	25.60	13.0%

Appendix Table 17b: Product Calculations

Sample	Lactose Remaining in Solution	Lactose Absorbed to resin	g Lactose added to resin	g Lactose remaining in solution	G Lactose Absorbed To resin	g Lactose Absorbed/ g resin
Feed	100.0%					
1	65.4%	34.6%	0.60	0.39	0.21	0.048
2	77.6%	22.4%	0.76	0.59	0.17	0.064
3	84.5%	15.5%	0.87	0.74	0.14	0.068
4	90.5%	9.5%	2.22	2.01	0.21	0.082

Trial 18

Description: Batch, Oven Dried Anion, DMSO/Lactose, Ambient temperature

Objective: To investigate lactose absorption using oven dried anion resin to remove any water dilution effect from the resin.

Trial Notes: Oven dried resin overnight.
Lactose concentration in lactose/DMSO solution = 15.02%

Appendix Table 18a: Results

Weighing after 1 hr							
Sample	Moist Resin	DMSO/lactose Solution	Feed:Resin	Dish Weight	Sample	Ashed Sample	TS
Feed 1		1.00		11.34	2.74	11.85	18.6%
Feed 2		1.00		10.76	2.69	11.27	19.2%
Feed 3		1.00		10.90	2.10	11.28	18.1%
1	2.82	6.59	2.3	9.04	1.93	9.34	15.6%

Reweighing after 1 hr 15 mins							
Sample	Moist Resin	DMSO/lactose Solution	Feed:Resin	Dish Weight	Sample	Ashed Sample	TS
Feed 1		1.00		11.34	2.74	11.85	18.3%
Feed 2		1.00		10.76	2.69	11.26	18.7%
Feed 3		1.00		10.90	2.10	11.27	17.8%
1	2.82	6.59	2.3	9.04	1.93	9.33	15.3%

Reweighing after 1 hr 30 mins							
Sample	Moist Resin	DMSO/lactose Solution	Feed:Resin	Dish Weight	Sample	Ashed Sample	TS
Feed 1		1.00		11.34	2.74	11.84	18.1%
Feed 2		1.00		10.76	2.69	11.25	18.4%
Feed 3		1.00		10.90	2.10	11.27	17.4%
1	2.82	6.59	2.3	9.04	1.93	9.33	15.2%

Appendix Table 18b: Dilution Calculations

Water Removal Method	Water removed from original resin mass
Free drain from column	37%
Oven dried for 1.5 hours	71%
Oven dried for 20 hours	77%

Trial 19

Description: Batch, Oven Dried Cation, Aqueous Lactose, Ambient temperature

Objective: To investigate lactose absorption using oven dried cation resin to remove any water dilution effect from the resin.

Trial Notes: Oven dried resin overnight.
On addition of solution to resin, noticeable cracking heard, heat evolved.
Lactose concentration in lactose/DMSO solution = 15.00%

Appendix Table 19a: Results

Sample	Dry Resin	Lactose Solution	RO Water	Feed:Resin	Dish Weight	Sample	Ashed Sample	TS
Feed 1		5.00			24.79	2.18	25.11	14.7%
Feed 2		5.00			24.33	2.49	24.71	15.3%
1	10.00	5.02	19.57	0.5	28.97	2.50	29.09	4.8%
2	4.03	4.16	12.09	1.0	30.78	2.37	30.95	7.2%
3	3.01	9.92		3.3	25.05	2.04	25.47	20.5%
4	2.01	6.61		3.3	24.15	1.60	24.48	20.7%
5	2.00	5.06		2.5	28.20	0.54	28.33	23.0%
6	1.51	4.56		3.0	27.01	0.79	27.18	21.3%
7	1.01	4.04		4.0	25.58	1.33	25.84	19.3%
8	1.00	5.05		5.0	26.81	2.05	27.18	18.1%

Trial 20

Description: Batch, Oven Dried R & H Anion, Aqueous Lactose, Ambient temperature

Objective: To investigate lactose absorption using oven dried anion resin to remove any water dilution effect from the resin.

Trial Notes: Oven dried resin overnight.
On addition of solution to resin smell evolved.
Lactose concentration in lactose/DMSO solution = 15.00%

Appendix Table 20a: Results

Sample	Moist Resin	Lactose Solution	Feed:Resin	Dish Weight	Sample	Ashed Sample	TS
Feed 1		5.00		24.51	2.11	24.83	14.8%
1	5.44	11.14	2.0	24.13	1.75	24.48	20.1%
2	2.02	4.15	2.1	25.75	0.54	25.86	20.6%
3	3.01	6.70	2.2	30.14	1.67	30.47	20.0%
4	2.01	4.03	2.0	24.11	0.41	24.19	20.3%
5	2.02	5.03	2.5	23.62	1.42	23.90	19.6%
6	1.50	4.55	3.0	28.19	1.67	28.50	18.6%
7	1.00	4.03	4.0	25.16	1.35	25.40	17.9%
8	1.00	5.01	5.0	29.68	2.16	30.05	17.1%

Trial 21

Description: Batch, Cation, Aqueous Lactose, Ambient temperature

Objective: To show moisture in the resin did not have a significant effect on TS result.

Trial Notes: Lactose concentration in lactose solution = 14.87%

Appendix Table 21a: Results

Sample	Resin Slurry	Lactose Solution	Feed:Resin	Dish Weight	Sample	Ashed Sample	TS
Feed 1		5.00		28.15	2.59	28.54	15.0%
Feed 2		5.00		28.64	2.36	28.99	14.8%
1	10.03	5.05	0.5	25.51	2.43	25.73	8.8%
2	8.33	8.57	1.0	25.00	2.22	25.25	11.2%
3	5.86	8.93	1.5	25.13	2.34	25.41	12.2%
4	3.86	7.79	2.0	24.75	3.07	25.14	12.8%
5	3.79	10.51	2.8	26.93	2.35	27.24	13.3%
6	2.85	8.46	3.0	30.71	2.33	31.02	13.2%
7	2.47	9.17	3.7	24.07	3.49	24.54	13.5%
8	1.09	5.08	4.6	30.12	2.38	30.45	13.8%

Appendix Table 21b: Product Calculations

Sample	Lactose Remaining in Solution	Lactose Absorbed to resin	g Lactose added to resin	g Lactose remaining in solution	g Lactose absorbed to resin	g Lactose Absorbed/ g resin
Feed 1	100.0%					
Feed 2	100.0%					
1	58.9%	41.1%	0.75	0.44	0.31	0.031
2	75.0%	25.0%	1.28	0.96	0.32	0.038
3	81.8%	18.2%	1.33	1.09	0.24	0.041
4	85.9%	14.1%	1.16	1.00	0.16	0.043
5	89.2%	10.8%	1.57	1.40	0.17	0.045
6	88.2%	11.8%	1.26	1.11	0.15	0.052
7	90.2%	9.8%	1.37	1.23	0.13	0.054
8	92.6%	7.4%	0.76	0.70	0.06	0.051

Appendix Table 21c: Resin Water Adjustment Calculations

7.0% Water fraction of resin slurry.					
Sample Description	Free Water	g Lactose added	Total Solution	Lactose Solution Concentration	TS
1	0.70	0.75	5.76	13.1%	8.8%
2	0.58	1.28	9.15	14.0%	11.2%
3	0.41	1.33	9.34	14.3%	12.2%
4	0.27	1.16	8.06	14.4%	12.8%
5	0.27	1.57	10.78	14.5%	13.3%
6	0.20	1.26	8.66	14.6%	13.2%
7	0.17	1.37	9.34	14.6%	13.5%
8	0.08	0.76	5.16	14.7%	13.8%

Appendix Table 21d: Resin Water Adjusted Lactose Absorption Calculations

Sample Description	Adjusted Lactose Absorbed to resin	g Lactose added to resin	g Lactose absorbed to resin	g Lactose Absorbed/ g resin
1	29%	0.75	0.22	0.022
2	19%	1.28	0.24	0.029
3	14%	1.33	0.18	0.031
4	11%	1.16	0.13	0.033
5	8%	1.57	0.13	0.034
6	9%	1.26	0.12	0.042
7	8%	1.37	0.11	0.044
8	6%	0.76	0.04	0.041

Trial 23

Description: Batch, IRA95 Anion, Aqueous, Ambient temperature

Objective: To investigate lactose absorption with a different resin.

Trial Notes: Lactose concentration in aqueous lactose solution = 14.93%
It is noticeable that IRA95 resin are smaller than A103S resin.

Appendix Table 23a: Results

Sample Description	Resin Slurry	Lactose Solution	Feed:Resin	Dish Weight	Sample	Ashed Sample	TS
Feed		5.00		28.64	2.20	28.97	14.9%
1	10.04	5.00	0.5	30.10	2.48	30.14	1.5%
2	4.03	4.03	1.0	26.75	2.26	26.85	4.6%
3	3.04	4.51	1.5	25.89	2.70	26.08	7.0%
4	1.99	4.28	2.2	24.02	2.55	24.25	8.9%
5	2.23	5.06	2.3	23.59	2.41	23.80	9.0%
6	1.60	4.51	2.8	28.62	2.46	28.87	10.1%
7	1.10	4.03	3.7	25.67	2.46	25.94	11.1%
8	1.12	5.01	4.5	30.71	2.72	31.03	11.7%
RO Water	2.10	4.45	2.1	24.36	2.12	24.36	0.0%

Appendix Table 23b: Product Calculations

Sample Description	Lactose Remaining in Solution	Lactose Absorbed To resin	g Lactose added to resin	g Lactose Remaining in solution	g Lactose absorbed to resin	g Lactose Absorbed/ g resin
Feed	100.0%					
1	9.8%	90.2%	0.74	0.07	0.67	0.067
2	31.0%	69.0%	0.60	0.19	0.41	0.103
3	47.4%	52.6%	0.67	0.32	0.35	0.116
4	59.8%	40.2%	0.64	0.38	0.26	0.129
5	60.5%	39.5%	0.75	0.46	0.30	0.133
6	68.0%	32.0%	0.67	0.46	0.21	0.134
7	74.8%	25.2%	0.60	0.45	0.15	0.137
8	78.5%	21.5%	0.75	0.59	0.16	0.143

Appendix Table 23c: Resin Water Adjustment Calculations

7.0% Water fraction of resin slurry.					
Sample Description	Free Water	g Lactose added	Total Solution	Lactose Solution Concentration	TS
1	0.70	0.74	5.70	13.0%	1.5%
2	0.28	0.60	4.31	13.9%	4.6%
3	0.21	0.67	4.72	14.2%	7.0%
4	0.14	0.64	4.42	14.4%	8.9%
5	0.16	0.75	5.22	14.4%	9.0%
6	0.11	0.67	4.62	14.5%	10.1%
7	0.08	0.60	4.11	14.6%	11.1%
8	0.08	0.75	5.09	14.6%	11.7%

Appendix Table 23d: Resin Water Adjusted Lactose Absorption Calculations

Sample Description	Adjusted Lactose Absorbed to resin	g Lactose added to resin	g Lactose absorbed to resin	g Lactose Absorbed/ g resin
1	78%	0.74	0.58	0.058
2	62%	0.60	0.37	0.093
3	48%	0.67	0.32	0.106
4	37%	0.64	0.24	0.119
5	37%	0.75	0.28	0.123
6	30%	0.67	0.20	0.124
7	23%	0.60	0.14	0.127
8	20%	0.75	0.15	0.133

Trial 24

Description: Batch, Spent Anion, Aqueous Lactose, Ambient temperature

Objective: To investigate the recovery of lactose with wash water from spent anion resin.

Trial Notes: Used spent resin from trial 23.
Total Elution yield is the cumulative results for the trial

Appendix Table 24a: First Elution Results

Sample Description	Resin Slurry	Lactose Solution	Initial F:R	Resin Slurry + container	Resin Slurry	RO Water	Wash BV	Total Wash BV	Dish Weight	Sample	Ashed Sample	TS
1	10.04	5.00	0.5	17.6	10.04	10.05	1.0	1.0	29.82	5.64	29.87	0.9%
2	4.03	4.03	1.0	11.2	4.03	4.08	1.0	1.0	27.34	3.02	27.41	2.5%
3	3.04	4.51	1.5	10.2	3.04	3.46	1.1	1.1	25.52	2.58	25.61	3.4%
4	1.99	4.28	2.2	9.0	1.99	2.27	1.1	1.1	28.53	2.15	28.62	4.3%
5	2.23	5.06	2.3	9.4	2.23	2.53	1.1	1.1	28.28	2.45	28.39	4.4%
6	1.60	4.51	2.8	8.6	1.60	2.08	1.3	1.3	28.82	2.18	28.92	4.7%
7	1.10	4.03	3.7	8.1	1.10	2.19	2.0	2.0	26.91	2.07	27.00	4.3%
8	1.12	5.01	4.5	8.0	1.12	2.26	2.0	2.0	25.90	2.25	25.99	4.2%
Control	2.10	4.45	2.1	9.0	2.10	2.32	1.1	1.1	28.13	2.18	28.13	0.0%

Appendix Table 24b: First Elution Calculations

Sample Description	Lactose Eluted g	g Lactose /g resin	g Lactose absorbed[1]	Total Elution Yield	Absolute Errors
1	0.095	0.009	0.686	14%	2%
2	0.102	0.025	0.444	23%	2%
3	0.118	0.039	0.397	30%	2%
4	0.097	0.049	0.298	33%	2%
5	0.113	0.051	0.358	32%	2%
6	0.106	0.066	0.266	40%	2%
7	0.092	0.083	0.210	44%	3%
8	0.098	0.087	0.206	47%	2%

Table Note:

1) Lactose absorbed (g) was calculated in trial 23, Appendix 23b. Solution in excess of the resin slurry solution was discarded. This discarded lactose was deducted from the absorbed lactose in Appendix 23b to produce the g Lactose absorbed in Appendix Table 24b above. Discarded lactose was calculated as the sample container (6.50g) plus the Resin Slurry (Appendix Table 24a) plus the Lactose Solution (Appendix Table 24a) less the Resin Slurry and container weight after discarding the solution (Appendix Table 24a).

Trial 24 continued – Second Elution

Appendix Table 24c: Results

Sample Description	Resin Slurry	Lactose Solution	Initial F:R	Resin Slurry + container	Resin Slurry	RO Water	Wash BV	Total Wash BV	Dish Weight	Sample	Ashed Sample	TS
5	2.23	5.06	2.3	9.4	2.23	2.48	1.1	2.2	24.02	2.45	24.07	2.1%
6	1.60	4.51	2.8	8.4	1.60	2.16	1.4	2.7	30.11	1.95	30.15	2.2%
7	1.10	4.03	3.7	8.2	1.10	2.30	2.1	4.1	28.65	2.46	28.69	1.8%
8	1.12	5.01	4.5	7.9	1.12	2.34	2.1	4.1	28.69	2.33	28.72	1.3%

Appendix Table 24d: Calculations

Sample Description	Lactose Eluted g	Total g Lactose eluted	Total Elution Yield	Absolute Errors
5	0.054	0.167	47%	2%
6	0.046	0.152	57%	3%
7	0.045	0.137	65%	3%
8	0.031	0.128	62%	3%

Trial 24 continued – Third Elution

Appendix Table 24e: Results

Sample Description	Resin Slurry	Lactose Solution	Initial F:R	Resin Slurry + container	Resin Slurry	RO Water	Wash BV	Total Wash BV	Dish Weight	Sample	Ashed Sample	TS
5	2.23	5.06	2.3	9.3	2.23	2.61	1.2	3.4	24.02	2.71	24.07	1.8%
6	1.60	4.51	2.8	8.5	1.60	3.42	2.1	4.8	28.64	3.01	28.68	1.3%
7	1.10	4.03	3.7	7.9	1.10	2.30	2.1	6.2	30.11	2.14	30.13	1.1%
8	1.12	5.01	4.5	7.9	1.12	2.36	2.1	6.2	29.82	2.19	29.85	1.2%

Appendix Table 24f: Calculations

Sample Description	Lactose Eluted g	Total g Lactose eluted	Total Elution Yield	Absolute Errors
5	0.050	0.216	60%	3%
6	0.041	0.192	72%	3%
7	0.024	0.161	77%	4%
8	0.027	0.155	75%	4%

Trial 24 continued – Fourth Elution

Appendix Table 24g: Results

Sample Description	Resin Slurry	Lactose Solution	Initial F:R	Resin Slurry + container	Resin Slurry	RO Water	Wash BV	Total Wash BV	Dish Weight	Sample	Ashed Sample	TS
5	2.23	5.06	2.3	9.1	2.23	2.69	1.2	4.6	26.92	2.35	26.94	1.0%
6	1.60	4.51	2.8	8.9	1.60	2.88	1.8	6.6	30.71	2.93	30.74	0.8%
7	1.10	4.03	3.7	8.0	1.10	2.61	2.4	8.5	28.82	1.79	28.83	0.7%
8	1.12	5.01	4.5	7.9	1.12	2.53	2.3	8.5	28.28	2.36	28.29	0.6%

Appendix Table 24h: Calculations

Sample Description	Lactose Eluted g	Total g Lactose eluted	Total Elution Yield	Absolute Errors
5	0.023	0.240	67%	3%
6	0.022	0.214	81%	4%
7	0.012	0.174	83%	4%
8	0.014	0.169	82%	5%

Trial 25

Description: Batch, Anion, Aqueous, Ambient temperature

Objective: To provide further data of lactose absorption with A103S resin.

Trial Notes: Lactose concentration in aqueous lactose solution = 15.00%

Appendix Table 25a: Results

Sample Description	Resin Slurry	Lactose Solution	Feed:Resin	Dish Weight	Sample	Ashed Sample	TS
Feed		3.00		24.02	2.67	24.43	15.4%
1	9.77	5.68	0.6	25.32	2.21	25.46	6.5%
2	3.98	3.99	1.0	24.08	2.74	24.31	8.6%
3	3.05	4.62	1.5	28.81	2.20	29.03	10.2%
4	2.02	4.07	2.0	24.36	2.71	24.66	11.1%
5	2.05	6.01	2.9	28.28	5.57	28.95	12.1%
6	1.52	4.91	3.2	25.52	4.42	26.07	12.5%
7	1.06	4.05	3.8	28.62	3.64	29.09	12.8%
8	1.05	5.19	5.0	29.82	4.76	30.46	13.3%

Appendix Table 25b: Product Calculations

Sample Description	Lactose Remaining In Solution	Lactose Absorbed to resin	g Lactose added to resin	g Lactose remaining in solution	g Lactose Absorbed to resin	g Lactose Absorbed/ g resin
Feed	100.0%					
1	42.6%	57.4%	0.87	0.37	0.50	0.051
2	56.1%	43.9%	0.61	0.34	0.27	0.068
3	66.4%	33.6%	0.71	0.47	0.24	0.078
4	72.1%	27.9%	0.62	0.45	0.17	0.086
5	79.0%	21.0%	0.92	0.73	0.19	0.095
6	81.3%	18.7%	0.75	0.61	0.14	0.093
7	83.1%	16.9%	0.62	0.52	0.10	0.099
8	86.9%	13.1%	0.80	0.69	0.10	0.100

Appendix Table 25c: Resin Water Adjustment Calculations

7.0% Water fraction of resin slurry.					
Sample Description	Free Water	g Lactose added	Total Solution	Lactose Solution Concentration	TS
1	0.68	0.87	6.36	13.7%	6.5%
2	0.28	0.61	4.27	14.4%	8.6%
3	0.21	0.71	4.84	14.7%	10.2%
4	0.14	0.62	4.21	14.8%	11.1%
5	0.14	0.92	6.16	15.0%	12.1%
6	0.11	0.75	5.02	15.0%	12.5%
7	0.07	0.62	4.13	15.1%	12.8%
8	0.07	0.80	5.26	15.1%	13.3%

Appendix Table 25d: Resin Water Adjusted Lactose Absorption Calculations

Sample Description	Adjusted Lactose Absorbed to resin	g Lactose added to resin	g Lactose absorbed to resin	g Lactose Absorbed/ g resin
1	47%	0.87	0.41	0.042
2	37%	0.61	0.23	0.058
3	29%	0.71	0.21	0.068
4	25%	0.62	0.15	0.076
5	19%	0.92	0.17	0.084
6	17%	0.75	0.13	0.082
7	15%	0.62	0.09	0.089
8	12%	0.80	0.09	0.089

Trial 26 – First Elution

Description: Batch, Spent Anion, Aqueous Lactose, Ambient temperature

Objective: To investigate the recovery of lactose with wash water from spent anion resin.

Trial Notes: Used spent resin from trial 25.
Total Elution yield is the cumulative results for the trial

Appendix Table 26a: First Elution Results

Sample Description	Resin Slurry	Lactose Solution	Initial F:R	Resin Slurry + container	Resin Slurry	RO Water	Wash BV	Total Wash BV	Dish Weight	Sample	Ashed Sample	TS
1	9.77	5.68	0.6	16.7	9.77	9.86	1.0	1.0	28.54	9.59	28.80	2.7%
2	3.98	3.99	1.0	10.8	3.98	4.03	1.0	1.0	26.91	3.81	27.06	3.8%
3	3.05	4.62	1.5	9.8	3.05	3.41	1.1	1.1	25.89	3.37	26.04	4.3%
4	2.02	4.07	2.0	8.9	2.02	2.15	1.1	1.1	28.64	2.05	28.75	5.0%
5	2.05	6.01	2.9	8.7	2.05	2.02	1.0	1.0	30.11	1.88	30.22	5.7%
6	1.52	4.91	3.2	8.4	1.52	1.62	1.1	1.1	25.10	1.49	25.19	6.0%
7	1.06	4.05	3.8	7.7	1.06	1.08	1.0	1.0	24.27	1.05	24.33	5.7%
8	1.05	5.19	5.0	7.8	1.05	1.21	1.2	1.2	24.02	1.15	24.09	5.7%

Trial 26 Continued – First Elution

Appendix Table 26b: First Elution Calculations

Sample Description	Lactose Eluted g	g Lactose absorbed[1]	Total Elution Yield	Absolute Errors
1	0.267	0.528	50%	1%
2	0.154	0.300	51%	2%
3	0.155	0.268	58%	2%
4	0.114	0.214	53%	2%
5	0.118	0.216	55%	2%
6	0.100	0.184	54%	2%
7	0.071	0.125	56%	4%
8	0.075	0.137	55%	3%

Table Note:
1) Lactose absorbed (g) was calculated in trial 25, Appendix 25b. Solution in excess of the resin slurry solution was discarded. This discarded lactose was deducted from the absorbed lactose in Appendix 25b to produce the g Lactose absorbed in Appendix Table 26b above. Discarded lactose was calculated as the sample container (6.50g) plus the Resin Slurry (Appendix Table 26a) plus the Lactose Solution (Appendix Table 26a) less the Resin Slurry and container weight after discarding the solution (Appendix Table 26a).

Trial 26 continued – Second Elution

Appendix Table 26c: Results

Sample Description	Resin Slurry	Lactose Solution	Initial F:R	Resin Slurry + container	Resin Slurry	RO Water	Wash BV	Total Wash BV	Dish Weight	Sample	Ashed Sample	TS
1	9.77	5.68	0.6	16.7	9.77	9.78	1.0	2.0	25.62	9.37	25.79	1.8%
2	3.98	3.99	1.0	10.8	3.98	4.00	1.0	2.0	28.13	3.76	28.20	1.7%
3	3.05	4.62	1.5	9.7	3.05	2.82	0.9	2.0	28.63	2.67	28.68	2.0%
4	2.02	4.07	2.0	8.8	2.02	2.06	1.0	2.1	28.55	1.92	28.60	2.4%
5	2.23	5.06	2.3	8.7	2.23	2.10	0.9	1.9	24.03	1.91	24.08	2.5%
6	1.60	4.51	2.8	8.3	1.60	3.41	2.1	3.2	24.27	3.13	24.33	1.7%
7	1.10	4.03	3.7	7.5	1.10	2.26	2.1	3.1	24.02	1.93	24.06	1.9%
8	1.12	5.01	4.5	7.7	1.12	1.34	1.2	2.4	28.63	1.20	28.67	3.2%

Appendix Table 26d: Calculations

Sample Description	Lactose Eluted g	Total g Lactose eluted	Total Elution Yield	Absolute Errors
1	0.179	0.445	84%	1%
2	0.068	0.222	74%	2%
3	0.058	0.212	79%	2%
4	0.051	0.164	77%	3%
5	0.052	0.170	79%	3%
6	0.057	0.158	86%	3%
7	0.042	0.112	90%	4%
8	0.044	0.119	87%	4%

Trial 26 continued – Third Elution

Appendix Table 26e: Results

Sample Description	Resin Slurry	Lactose Solution	Initial F:R	Resin Slurry + container	Resin Slurry	RO Water	Wash BV	Total Wash BV	Dish Weight	Sample	Ashed Sample	TS
1	9.77	5.68	0.6	16.7	9.77	9.89	1.0	3.0	27.97	9.46	28.03	0.6%
2	3.98	3.99	1.0	10.9	3.98	4.01	1.0	3.0	23.58	3.58	23.61	0.7%
3	3.05	4.62	1.5	9.6	3.05	3.51	1.2	3.2	30.07	3.33	30.10	0.9%
4	2.02	4.07	2.0	8.7	2.02	2.20	1.1	3.2	24.08	2.08	24.11	1.0%
5	2.23	5.06	2.3	8.7	2.23	2.34	1.0	3.0	28.28	2.26	28.31	1.2%
6	1.60	4.51	2.8	8.4	1.60	2.24	1.4	4.6	30.11	2.32	30.13	0.7%
7	1.10	4.03	3.7	7.7	1.10	2.12	1.9	5.0	28.18	2.06	28.20	0.7%
8	1.12	5.01	4.5	7.7	1.12	2.49	2.2	4.6	29.56	2.30	29.57	0.8%

Appendix Table 26f: Calculations

Sample Description	Lactose Eluted g	Total g Lactose eluted	Total Elution Yield	Absolute Errors
1	0.056	0.501	95%	1%
2	0.028	0.249	83%	2%
3	0.033	0.245	91%	3%
4	0.023	0.187	88%	3%
5	0.029	0.199	92%	3%
6	0.018	0.176	95%	4%
7	0.015	0.127	102%	5%
8	0.019	0.138	101%	5%

Trial 26 continued – Fourth Elution

Appendix Table 26g: Results

Sample Description	Resin Slurry	Lactose Solution	Initial F:R	Resin Slurry + container	Resin Slurry	RO Water	Wash BV	Total Wash BV	Dish Weight	Sample	Ashed Sample	TS
1	9.77	5.68	0.6	16.9	9.77	9.64	1.0	4.0	24.02	9.60	24.05	0.3%
2	3.98	3.99	1.0	11.1	3.98	4.77	1.2	4.2	24.02	5.04	24.04	0.4%
3	3.05	4.62	1.5	9.7	3.05	3.92	1.3	4.5	24.08	3.87	24.09	0.4%
4	2.02	4.07	2.0	8.7	2.02	2.44	1.2	4.4	24.99	2.22	25.00	0.5%
5	2.23	5.06	2.3	8.6	2.23	2.44	1.1	4.1	24.27	2.03	24.28	0.5%
6	1.60	4.51	2.8	8.2	1.60	2.23	1.4	6.0	28.81	2.01	28.82	0.2%
7	1.10	4.03	3.7	7.6	1.10	2.32	2.1	7.1	29.56	2.13	29.56	0.2%
8	1.12	5.01	4.5	7.7	1.12	2.26	2.0	6.6	30.05	2.06	30.06	0.2%

Appendix Table 26h: Calculations

Sample Description	Lactose Eluted g	Total g Lactose eluted	Total Elution Yield	Absolute Errors
1	0.027	0.529	100%	2%
2	0.020	0.269	90%	3%
3	0.014	0.259	97%	3%
4	0.012	0.199	93%	4%
5	0.011	0.210	97%	4%
6	0.004	0.180	98%	4%
7	0.003	0.131	105%	6%
8	0.005	0.143	104%	6%

Trial 27

Description: Batch, Anion, Aqueous, Ambient temperature, Variable resin size, Fixed F:R

Objective: Investigate if resin sample size (fixed F:R) influences lactose absorption.

Trial Notes: Lactose concentration in aqueous lactose solution = 15.00%

Appendix Table 27a: Results

Sample Description	Resin Slurry	Lactose Solution	Feed:Resin	Dish Weight	Sample	Ashed Sample	TS
1	1.52	4.91	3.2	25.52	4.42	26.07	12.5%
2	0.96	2.90	3.0	30.71	2.55	31.03	12.4%
3	3.28	9.86	3.0	22.46	8.92	23.55	12.3%
4	0.80	2.36	3.0	27.33	2.02	27.58	12.3%

Appendix Table 27b: Product Calculations

Sample Description	Lactose Remaining In Solution	Lactose Absorbed to resin	g Lactose added to resin	g Lactose remaining in solution	g Lactose Absorbed to resin	g Lactose Absorbed/ g resin
1	81.3%	18.7%	0.75	0.61	0.14	0.093
2	80.6%	19.4%	0.44	0.36	0.09	0.090
3	79.9%	20.1%	1.51	1.21	0.31	0.093
4	80.4%	19.6%	0.36	0.29	0.07	0.089

Appendix Table 27c: Resin Water Adjustment Calculations

7.0% Water fraction of resin slurry.					
Sample Description	Free Water	g Lactose added	Total Solution	Lactose Solution Concentration	TS
1	0.11	0.75	5.02	15.0%	12.5%
2	0.07	0.44	2.96	15.0%	12.4%
3	0.23	1.51	10.09	15.0%	12.3%
4	0.06	0.36	2.42	15.0%	12.3%

Appendix Table 27d: Resin Water Adjusted Lactose Absorption Calculations

Sample Description	Adjusted Lactose Absorbed to resin	g Lactose added to resin	g Lactose absorbed to resin	g Lactose Absorbed/ g resin
1	17%	0.75	0.13	0.082
2	17%	0.44	0.08	0.080
3	18%	1.51	0.27	0.083
4	17%	0.36	0.06	0.078

Trial 27 Continued – First Elution

Description: Batch, Spent Anion, Aqueous Lactose, Ambient temperature

Objective: To investigate the recovery of lactose with wash water from spent anion resin.

Trial Notes: Used spent resin from trial 27.
Total Elution yield is the cumulative results for the trial

Appendix Table 27e: First Elution Results

Sample Description	Resin Slurry	Lactose Solution	Initial F:R	Resin Slurry + container	Resin Slurry	RO Water	Wash BV	Total Wash BV	Dish Weight	Sample	Ashed Sample	TS
1	1.52	4.91	3.2	8.4	1.52	1.62	1.1	1.1	25.10	1.49	25.19	6.0%
2	0.96	2.90	3.0	7.6	0.96	2.07	2.2	2.2	24.36	1.84	24.43	3.9%
3	3.28	9.86	3.0	10.6	3.28	3.38	1.0	1.0	28.82	3.70	29.04	6.0%
4	0.80	2.36	3.0	7.4	0.80	1.87	2.3	2.3	25.32	1.78	25.39	3.8%

Appendix Table 27f: First Elution Calculations

Sample Description	Lactose Eluted g	g Lactose absorbed[1]	Total Elution Yield	Absolute Errors
1	0.100	0.184	54%	3%
2	0.079	0.104	76%	3%
3	0.232	0.408	57%	3%
4	0.075	0.088	85%	4%

Table Note:
1) Lactose absorbed (g) was calculated in trial 27, Appendix 27b. Solution in excess of the resin slurry solution was discarded. This discarded lactose was deducted from the absorbed lactose in Appendix 27b to produce the g Lactose absorbed in Appendix Table 27b.

above. Discarded lactose was calculated as the sample container (6.50g) plus the Resin Slurry (Appendix Table 27d) plus the Lactose Solution (Appendix Table 26d) less the Resin Slurry and container weight after discarding the solution (Appendix Table 26d).

Trial 27 continued – Second Elution

Appendix Table 27g: Results

Sample Description	Resin Slurry	Lactose Solution	Initial F:R	Resin Slurry + container	Resin Slurry	RO Water	Wash BV	Total Wash BV	Dish Weight	Sample	Ashed Sample	TS
1	1.52	4.91	3.2	8.3	1.52	3.41	2.2	3.3	24.27	3.13	24.33	1.7%
2	0.96	2.90	3.0	7.7	0.96	2.04	2.1	4.3	24.99	2.06	25.01	0.9%
3	3.28	9.86	3.0	10.1	3.28	3.34	1.0	2.0	27.34	3.05	27.41	2.5%
4	0.80	2.36	3.0	7.3	0.80	1.91	2.4	4.7	25.11	1.60	25.11	0.4%

Appendix Table 27h: Calculations

Sample Description	Lactose Eluted g	Total g Lactose eluted	Total Elution Yield	Absolute Errors
1	0.057	0.158	86%	3%
2	0.020	0.099	95%	5%
3	0.082	0.314	77%	1%
4	0.008	0.082	94%	6%

Trial 27 continued – Third Elution

Appendix Table 27i: Results

Sample Description	Resin Slurry	Lactose Solution	Initial F:R	Resin Slurry + container	Resin Slurry	RO Water	Wash BV	Total Wash BV	Dish Weight	Sample	Ashed Sample	TS
1	1.52	4.91	3.2	8.4	1.52	2.24	1.5	4.8	30.11	2.32	30.13	0.7%
2	0.96	2.90	3.0	7.5	0.96	2.45	2.6	6.8	25.52	2.14	25.53	0.3%
3	3.28	9.86	3.0	10.2	3.28	3.37	1.0	3.1	26.91	3.32	26.95	1.2%
4	0.80	2.36	3.0	7.5	0.80	2.50	3.1	7.8	22.46	2.56	22.47	0.3%

Appendix Table 27j: Calculations

Sample Description	Lactose Eluted g	Total g Lactose eluted	Total Elution Yield	Absolute Errors
1	0.018	0.176	95%	4%
2	0.006	0.106	101%	6%
3	0.040	0.354	87%	2%
4	0.009	0.091	104%	7%

Trial 27 continued – Fourth Elution

Appendix Table 27k: Results

Sample Description	Resin Slurry	Lactose Solution	Initial F:R	Resin Slurry + container	Resin Slurry	RO Water	Wash BV	Total Wash BV	Dish Weight	Sample	Ashed Sample	TS
1	1.52	4.91	3.2	8.2	1.52	2.23	1.5	6.2	28.81	2.01	28.82	0.2%
2	0.96	2.90	3.0	7.7	0.96	2.68	2.8	9.6	28.28	2.71	28.28	0.1%
3	3.28	9.86	3.0	10.1	3.28	9.89	3.0	6.1	23.58	3.35	23.58	0.1%
4	0.80	2.36	3.0	7.3	0.80	4.86	6.1	13.9	30.11	1.91	30.11	-0.1%

Appendix Table 27l: Calculations

Sample Description	Lactose Eluted g	Total g Lactose eluted	Total Elution Yield	Absolute Errors
1	0.004	0.180	98%	4%
2	0.002	0.108	103%	7%
3	0.022	0.376	92%	2%
4	-0.001	0.090	103%	9%

Trial 28

Description: Batch, Anion, DMSO/lactose, Ambient temperature

Objective: To investigate the evolution of moisture during DMSO trials.

Trial Notes: Lactose concentration in DMSO/lactose solution = 15.00%
Washed resin with three extractions of DMSO before addition

Appendix Table 28a: Results

Sample Description	Sample No.	Resin Slurry	Solution	Feed:Resin	Dish Weight	Sample	Ashed Sample	TS
DMSO/Lac	1		1.00		10.75	1.61	11.04	17.7%
DMSO/Lac	2		1.00		11.33	1.59	11.60	17.3%
DMSO/Lac/Resin	3	3.14	8.08	2.6	9.09	1.67	9.31	13.1%
DMSO/Lac/Resin	4	3.09	7.96	2.6	10.74	1.75	10.96	12.6%
DMSO/Resin	5	3.07	7.93	2.6	10.81	1.62	10.81	0.0%
DMSO/Resin	6	3.10	8.01	2.6	8.96	1.64	8.96	-0.1%

Appendix Table 28b: Product Calculations

Sample Number	Lactose Remaining In Solution	Lactose Absorbed to resin	g Lactose added to resin	g Lactose remaining in solution	g Lactose absorbed to resin	g Lactose Absorbed/ g resin
1	100.0%					
2	100.0%					
3	74.7%	25.3%	1.41	1.06	0.36	0.114
4	72.2%	27.8%	1.39	1.01	0.39	0.125
5	0.0%					
6	-0.5%					

Trial 28 continued – Moisture Analysis

Appendix Table 28c: Results and Calculations; Moisture Results

Sample Number	Moisture Observations 1	2	3	Totals	Mean	Standard Deviation
1	3.74	3.81	4.68	12.22	4.07	0.52
2	3.51	3.77	4.43	11.71	3.90	0.48
3	6.13	6.09	6.52	18.75	6.25	0.24
4	6.46	7.26	6.61	20.33	6.78	0.43
5	5.65	6.61	7.02	19.28	6.43	0.70
6	6.20	6.80	6.74	19.74	6.58	0.33
				102.02	5.67	

Appendix Table 28d: Moisture Result 95% Confidence Interval Calculations

Sample Number	Error Range	95% Confidence Levels LCL	UCL
1	0.47	3.60	4.55
2	0.45	3.45	4.35
3	0.32	5.93	6.57
4	0.43	6.35	7.20
5	0.55	5.88	6.97
6	0.38	6.20	6.96

Appendix Table 28e: Results and Calculations; Moisture-Squared Results

Sample Number	Moisture Observations^2 1	2	3	Totals
1	13.96	14.51	21.87	50.35
2	12.29	14.21	19.62	46.12
3	37.63	37.05	42.57	117.25
4	41.71	52.70	43.68	138.08
5	31.89	43.74	49.25	124.88
6	38.41	46.24	45.46	130.12
				606.80

Trial 28 continued – Moisture Analysis

Appendix Table 28f: Statistical Analysis; All Moisture Results

Null Hypothesis that samples 1,2,3,4,5 & 6 are from different populations.

	Sum of Squares	Degrees of Freedom	Mean Square	F_o	$F_{0.01,2,4}$
SS_{Factor}	-276.17	2	-138.08	-1.81	18.00
SS_{Error}	304.75	4	76.19		
SS_T	28.58	6			

Conclusion: No statistical evidence to suggest Null Hypothesis is correct.

Appendix Table 28g: Statistical Analysis; Moisture Results containing resin

Null Hypothesis that samples 3,4,5 & 6 are from different populations.

	Sum of Squares	Degrees of Freedom	Mean Square	F_o	$F_{0.01,2,3}$
SS_{Factor}	-126.71	2	-63.35	-1.48	30.82
SS_{Error}	128.85	3	42.95		
SS_T	2.14	5			

Conclusion: No statistical evidence to suggest Null Hypothesis is correct.

Trial 28 continued – Moisture Evolution

Objective: Calculate the expected moisture evolved from lactose absorption.

Information:

$Mr(C_6H_{12}O_6)$ = 184 g/mole

$Mr(H_2O)$ = 18 g/mole

From trial 16 (Figure 18) the approximate lactose absorption for a F:R ratio of 2.6 is:

0.06 g lactose/g resin

Lactose Absorbed

$n = m/Mr$

$= 0.06/184$

$= 3.26 * 10^{-4}$ moles

Mole Ratio

$C_6H_{12}O_6$: H_2O

1:1

Water Eluted

Therefore the water eluted is $3.26 * 10^{-4}$ moles

$m = 3.26 * 10^{-4}$ moles * 18 g/mole

$= 5.87 * 10^{-3}$ g

Moisture increase for trial 28:

$= 5.87 * 10^{-3}$ g/ 8 g solution

= **0.07%** increase in moisture due to lactose absorption.

Trial 29

Description: Batch, A847S resin, Aqueous, Ambient temperature

Objective: To provide data of lactose absorption with A847S resin.

Trial Notes: Lactose concentration in aqueous lactose solution = 14.94%

Appendix Table 29a: Results

Sample Description	Resin Slurry	Lactose Solution	Feed:Resin	Dish Weight	Sample	Ashed Sample	TS
Feed		2.47		24.68	2.19	25.02	15.5%
1	9.93	5.02	0.5	29.64	2.07	29.81	8.4%
2	4.08	4.03	1.0	29.58	2.14	29.81	10.7%
3	3.04	4.53	1.5	24.47	2.60	24.78	12.2%
4	2.23	4.41	2.0	29.52	2.13	29.79	12.8%
5	1.97	4.96	2.5	28.12	3.10	28.53	13.3%
6	1.63	5.02	3.1	22.46	2.74	22.83	13.7%
7	0.98	4.09	4.2	24.00	2.87	24.41	14.2%
8	1.20	7.66	6.4	29.40	4.62	30.07	14.5%

Appendix Table 29b: Product Calculations

Sample Description	Lactose Remaining in Solution	Lactose Absorbed to resin	g Lactose added to resin	g Lactose remaining in solution	g Lactose absorbed to resin	g Lactose Absorbed/ g resin
Feed	100.0%					
1	54.1%	45.9%	0.78	0.42	0.36	0.036
2	69.1%	30.9%	0.63	0.43	0.19	0.047
3	78.4%	21.6%	0.70	0.55	0.15	0.050
4	82.3%	17.7%	0.69	0.56	0.12	0.054
5	85.7%	14.3%	0.77	0.66	0.11	0.056
6	88.3%	11.7%	0.78	0.69	0.09	0.056
7	91.2%	8.8%	0.64	0.58	0.06	0.057
8	93.6%	6.4%	1.19	1.11	0.08	0.063

Appendix Table 29c: Resin Water Adjustment Calculations

7.0% Water fraction of resin slurry.					
Sample Description	Free Water	g Lactose added	Total Solution	Lactose Solution Concentration	TS
1	0.70	0.78	5.71	13.6%	8.4%
2	0.29	0.63	4.32	14.5%	10.7%
3	0.21	0.70	4.74	14.8%	12.2%
4	0.16	0.69	4.57	15.0%	12.8%
5	0.14	0.77	5.10	15.1%	13.3%
6	0.11	0.78	5.13	15.2%	13.7%
7	0.07	0.64	4.16	15.3%	14.2%
8	0.08	1.19	7.74	15.4%	14.5%

Appendix Table 29d: Resin Water Adjusted Lactose Absorption Calculations

Sample Description	Adjusted Lactose Absorbed to resin	g Lactose added to resin	g Lactose absorbed to resin	g Lactose Absorbed/ g resin
1	34%	0.78	0.26	0.027
2	24%	0.63	0.15	0.037
3	17%	0.70	0.12	0.039
4	14%	0.69	0.10	0.044
5	12%	0.77	0.09	0.045
6	9%	0.78	0.07	0.045
7	7%	0.64	0.05	0.046
8	5%	1.19	0.06	0.053

Trial 30

Description: Batch, Spent A847S, Aqueous Lactose, Ambient temperature

Objective: To investigate the recovery of lactose with wash water from spent anion resin.

Trial Notes: Used spent resin from trial 29.
Total Elution yield is the cumulative results for the trial

Appendix Table 30a: First Elution Results

Sample Description	Resin Slurry	Lactose Solution	Initial F:R	Resin Slurry + container	Container	Resin Slurry	RO Water	Wash BV	Total Wash BV	Dish Weight	Sample	Ashed Sample	TS
First Elution													
1	9.93	5.02	0.5	16.9	6.38	9.93	9.96	1.0	1.0	30.72	2.27	30.78	2.7%
7	0.98	4.09	4.2	7.8	6.40	0.98	1.23	1.3	1.3	24.03	1.39	24.11	5.9%
8	1.20	7.66	6.4	8.2	6.51	1.20	1.53	1.3	1.3	23.96	1.49	24.06	6.6%
Second Elution													
1	9.93	5.02	0.5	6.4	16.8	9.93	10.12	1.0	2.0	21.59	2.45	21.61	0.8%
Third Elution													
1	9.93	5.02	0.5	6.4	17.1	9.93	11.30	1.1	3.2	29.41	2.72	29.41	0.3%
Fourth Elution													
1	9.93	5.02	0.5	6.4	16.5	9.93	11.31	1.1	4.3	24.03	3.13	24.03	0.0%

Appendix Table 30b: First Elution Calculations

Sample Description	Lactose Eluted g	Total Lactose Eluted, g	g Lactose absorbed[1]	Total Elution Yield	Absolute Errors
First Elution					
1	0.273		0.403	68%	6%
7	0.082		0.118	69%	3%
8	0.098		0.152	65%	3%
Second Elution					
1	0.079	0.352		87%	1%
Third Elution					
1	0.034	0.386		96%	2%
Fourth Elution					
1	0.000	0.386		96%	2%

Table Note:

1) Lactose absorbed (g) was calculated from information in Appendix Table 29a and Appendix Table 30a. First the lactose added to the solution in trial 29 was calculated as the lactose solution (5.015g) added multiplied by the lactose solution TS (15.54%). Then the lactose discarded was calculated. This was calculated from Appendix Table 30a. The mass of the lactose solution discarded was calculated as the container (6.38g), resin (9.93g) and lactose solution (5.015g) weights less the resin slurry + container weight (16.85g) before addition of RO water. The discarded mass was then multiplied by the supernatant TS (8.40%) from Appendix Table 29a. This provided the lactose mass discarded (0.3763g). When subtracted from the lactose mass added to the resin (0.7793g), this provided a more accurate measure of the resin absorbed (0.4030g). The lactose absorption calculated in trial 29 and that calculated in trial did not differ greatly. For the lactose elution yields this number greatly effects the result. Therefore this calculation was used for recovery yields only.

Trial 31

Description: Batch, A845 resin, Aqueous, Ambient temperature

Objective: To provide data of lactose absorption with A845 resin.

Trial Notes: Lactose concentration in aqueous lactose solution = 14.94%

Appendix Table 31a: Results

Sample Description	Resin Slurry	Lactose Solution	Feed:Resin	Dish Weight	Sample	Ashed Sample	TS
Feed		2.47		25.88	7.95	27.12	15.6%
1	9.87	5.20	0.5	24.92	2.07	25.10	8.8%
2	4.05	4.40	1.1	29.33	2.37	29.59	11.0%
3	2.94	4.46	1.5	27.92	2.33	28.20	12.1%
4	1.97	3.99	2.0	30.26	2.22	30.54	12.8%
5	1.90	4.51	2.4	24.35	3.09	24.76	13.0%
6	1.52	4.57	3.0	23.08	2.17	23.37	13.4%
7	0.98	4.48	4.6	28.25	2.59	28.61	14.2%
8	1.05	6.02	5.7	29.11	2.26	29.44	14.4%

Appendix Table 31b: Product Calculations

Sample Description	Lactose Remaining in Solution	Lactose Absorbed to resin	g Lactose added to resin	g Lactose remaining in solution	g Lactose absorbed to resin	g Lactose Absorbed/ g resin
Feed	100.0%					
1	56.9%	43.1%	0.81	0.46	0.35	0.035
2	70.8%	29.2%	0.68	0.48	0.20	0.049
3	77.5%	22.5%	0.69	0.54	0.16	0.053
4	82.5%	17.5%	0.62	0.51	0.11	0.055
5	83.9%	16.1%	0.70	0.59	0.11	0.060
6	86.2%	13.8%	0.71	0.61	0.10	0.065
7	91.3%	8.7%	0.70	0.64	0.06	0.062
8	92.3%	7.7%	0.94	0.86	0.07	0.068

Appendix Table 31c: Resin Water Adjustment Calculations

9.0% Water fraction of resin slurry.					
Sample Description	Free Water	g Lactose added	Total Solution	Lactose Solution Concentration	TS
1	0.89	0.81	6.09	13.3%	8.8%
2	0.36	0.68	4.76	14.4%	11.0%
3	0.26	0.69	4.72	14.7%	12.1%
4	0.18	0.62	4.16	14.9%	12.8%
5	0.17	0.70	4.68	15.0%	13.0%
6	0.14	0.71	4.71	15.1%	13.4%
7	0.09	0.70	4.57	15.3%	14.2%
8	0.09	0.94	6.11	15.3%	14.4%

Appendix Table 31d: Resin Water Adjusted Lactose Absorption Calculations

Sample Description	Adjusted Lactose Absorbed to resin	g Lactose added to resin	g Lactose absorbed to resin	g Lactose Absorbed/ g resin
1	34%	0.78	0.26	0.027
2	24%	0.63	0.15	0.037
3	17%	0.70	0.12	0.039
4	14%	0.69	0.10	0.044
5	12%	0.77	0.09	0.045
6	9%	0.78	0.07	0.045
7	7%	0.64	0.05	0.046
8	5%	1.19	0.06	0.053

Trial 32

Description: Batch, Spent A845, Aqueous Lactose, Ambient temperature

Objective: To investigate the recovery of lactose with wash water from spent anion resin.

Trial Notes: Used spent resin from trial 31.
Total Elution yield is the cumulative results for the trial

Appendix Table 32a: First Elution Results

Sample Description	Resin Slurry	Lactose Solution	Initial F:R	Resin Slurry + container	Container	Resin Slurry	RO Water	Wash BV	Total Wash BV	Dish Weight	Sample	Ashed Sample	TS
First Elution													
1	9.87	5.20	0.5	6.4	16.6	9.87	9.98	1.0	1.0	23.47	2.20	23.53	2.6%
8	1.05	6.02	5.7	6.4	7.8	1.05	1.05	1.0	1.0	28.50	1.13	28.57	6.4%
Second Elution													
1	9.87	5.20	0.5	6.4	16.9	9.87	10.12	1.0	2.0	27.70	3.24	27.72	0.8%
Third Elution													
1	9.87	5.20	0.5	6.4	16.7	9.87	11.44	1.2	3.2	24.69	3.06	24.70	0.3%
Fourth Elution													
1	9.87	5.20	0.5	6.4	16.4	9.87	10.27	1.0	4.2	29.11	3.62	29.12	0.1%

Appendix Table 32b: First Elution Calculations

Sample Description	Lactose Eluted g	Total Lactose Eluted, g	g Lactose absorbed[1]	Total Elution Yield	Absolute Errors
First Elution					
1	0.254		0.380	67%	7%
7	0.072		0.108	67%	4%
Second Elution					
1	0.081	0.008		88%	1%
Third Elution					
1	0.032	0.003		97%	2%
Fourth Elution					
1	0.004	0.000		98%	2%

Table Note:

1) Lactose absorbed (g) was calculated from information in Appendix Table 31a and Appendix Table 32a. First the lactose added to the solution in trial 31 was calculated as the lactose solution (5.204g) added multiplied by the lactose solution TS (15.55%). Then the lactose discarded was calculated. This was calculated from Appendix Table 32a. The mass of the lactose solution discarded was calculated as the container (6.40g), resin (9.87g) and lactose solution (5.20g) weights less the resin slurry + container weight (16.63g) before addition of RO water. The discarded mass was then multiplied by the supernatant TS (8.84%) from Appendix Table 31a. This provided the lactose mass discarded (0.4296g). When subtracted from the lactose mass added to the resin (0.8092g), this provided a more accurate measure of the resin absorbed (0.380g). The lactose absorption calculated in trial 31 and that calculated in trial did not differ greatly. For the lactose elution yields this number greatly effects the result. Therefore this calculation was used for recovery yields only.

Trial 33

Description: Batch, A847S resin, Aqueous, Ambient temperature

Objective: To investigate lactose absorption with varying lactose feed concentrations.

Trial Notes: Lactose concentration in aqueous lactose solution = 30.00%
Lactose solution heated in microwave to dissolve lactose. Condensation formed. From dilution ratios TS were calculated for each feed concentration. These were based:

1) on 30% TS (no evaporation) &
2) the 30% sample TS analysis (evaporation)

Appendix Table 33a: Results

Sample Description	RO Water	Lactose Solution	Dish Weight	Sample	Ashed Sample	TS	No evaporation	Evaporation in 30% sample
0%	4.09	0.00	25.49	4.09	25.48	-0.01%	0.00%	0.00%
6%	6.44	1.60	28.63	2.68	28.81	6.96%	5.96%	6.96%
12%	4.80	3.21	25.32	2.79	25.71	13.93%	12.02%	14.05%
18%	3.23	4.81	30.08	2.84	30.67	20.83%	17.96%	20.98%
24%	1.60	6.40	27.27	2.72	28.03	28.05%	23.99%	28.03%
30%	0.00	8.20	23.41	2.36	24.24	35.05%	30.00%	35.05%

Appendix Table 33b: Results

Sample Description	Resin Slurry	Lactose Solution	Feed:Resin	Dish Weight	Sample	Ashed Sample	TS
1	1.97	4.34	2.2	24.26	2.34	24.26	0.0%
2	5.04	5.15	1.0	24.00	3.38	24.17	5.0%
3	4.95	5.00	1.0	29.41	2.69	29.68	10.1%
4	4.95	5.00	1.0	24.68	2.44	25.05	15.1%
5	5.03	5.04	1.0	28.87	2.48	29.36	20.04%
6	5.03	5.54	1.1	28.81	2.55	29.47	25.88%

Appendix Table 33c: Product Calculations

Sample Description	Lactose Remaining in Solution	Lactose Absorbed to resin	g Lactose added to resin	g Lactose remaining in solution	g Lactose absorbed to resin	g Lactose Absorbed/ g resin
1			0.00	0.00	0.00	0.000
2	71.3%	28.7%	0.36	0.26	0.10	0.020
3	72.2%	27.8%	0.70	0.50	0.19	0.039
4	72.6%	27.4%	1.04	0.76	0.29	0.058
5	71.4%	28.6%	1.41	1.01	0.40	0.080
6	73.8%	26.2%	1.94	1.43	0.51	0.101

Appendix Table 33d: Resin Water Adjustment Calculations

9.0% Water fraction of resin slurry.					
Sample Description	Free Water	g Lactose added	Total Solution	Lactose Solution Concentration	TS
1	0.18	0.00	4.52	0.0%	0.0%
2	0.45	0.36	5.60	6.4%	5.0%
3	0.45	0.70	5.44	12.8%	10.1%
4	0.45	1.04	5.45	19.1%	15.1%
5	0.45	1.415	5.497	25.7%	20.0%
6	0.45	1.94	5.99	32.4%	25.9%

Appendix Table 33e: Resin Water Adjusted Lactose Absorption Calculations

Sample Description	Adjusted Lactose Absorbed to resin	g Lactose added to resin	g Lactose absorbed to resin	g Lactose Absorbed/ g resin
1	0%	0.00	0.00	0.000
2	21%	0.36	0.07	0.015
3	20%	0.70	0.14	0.028
4	19%	1.04	0.20	0.040
5	20%	1.41	0.29	0.057
6	19%	1.94	0.36	0.072

Trial 34

Description: Batch, Spent A847S, Aqueous Lactose, Ambient temperature

Objective: To investigate the recovery of lactose with wash water from spent anion resin with varying lactose feed concentrations.

Trial Notes: Used spent resin from trial 33.
Total Elution yield is the cumulative results for the trial

Appendix Table 34a: First Elution Results

Sample Description	Resin Slurry	Lactose Solution	Initial F:R	Resin Slurry + container	Container	Resin Slurry	RO Water	Wash BV	Total Wash BV	Dish Weight	Sample	Ashed Sample	TS
First Elution													
6% Sample	5.04	5.15	1.02	11.3	6.4	5.04	5.01	1.0	1.0	23.92	2.26	23.95	1.4%
12% Sample	4.95	5.00	1.01	11.5	6.5	4.95	5.03	1.0	1.0	24.36	2.76	24.44	3.0%
18% Sample	4.95	5.00	1.01	11.4	6.4	4.95	5.01	1.0	1.0	26.75	2.94	26.88	4.1%
24% Sample	5.03	5.04	1.00	11.3	6.5	5.03	5.22	1.0	1.0	29.64	2.12	29.76	5.5%
30% Sample	5.03	5.54	1.10	11.3	6.4	5.03	5.23	1.0	1.0	23.47	2.60	23.67	7.5%
Second Elution													
6% Sample	5.04	5.15	1.0	11.4	6.4	5.04	5.04	1.0	2.0	24.00	3.33	24.02	0.4%
30% Sample	5.03	5.54	1.1	11.5	6.4	5.03	5.11	1.0	2.0	26.93	3.34	27.01	2.4%
Third Elution													
6% Sample	5.04	5.15	1.0	11.3	6.4	5.04	5.01	1.0	3.0	29.52	2.83	29.52	0.1%
30% Sample	5.03	5.54	1.1	11.4	6.4								
Fourth Elution													
6% Sample	5.04	5.15	1.0	11.4	6.4								

Appendix Table 34b: First Elution Calculations

Sample Description	Lactose Eluted g	Total Lactose Eluted, g	g Lactose absorbed[1]	Total Elution Yield	Absolute Errors
First Elution					
6% Sample	0.066		0.094	70%	14%
12% Sample	0.142		0.196	72%	6%
18% Sample	0.217		0.285	76%	4%
24% Sample	0.255		0.363	70%	4%
30% Sample	0.383		0.482	79%	2%
Second Elution					
6% Sample	0.022	0.089		94%	5%
30% Sample	0.123	0.506		105%	1%
Third Elution					
6% Sample	0.007	0.096		101%	7%
30% Sample	0.032	0.003			

Table Note:

1) g Lactose absorbed was calculated from information in Appendix Table 31a and Appendix Table 32a. Details of the calculation are described in Appendix, Trial 30.

Trial 35

Description: Moisture assessment of all resin slurries used.

Objective: To investigate the recovery of lactose with wash water from spent anion resin with varying lactose feed concentrations.

Trial Notes: Adjusted resin slurries to similar consistency.
Weighed sample then removed free moisture by pipette.

Appendix Table 35a: Resin Slurry Moisture Results

Resin	Sample Type	Sample	Dry Sample	Water	Free Moisture
A103S	Main Sample	23.34	22.20	1.14	4.9%
A103S	Spooned Sample	6.72	6.25	0.46	6.9%
C100H	Main Sample	28.66	27.03	1.63	5.7%
C100H	Spooned Sample	5.64	5.24	0.41	7.2%
IRA95	Main Sample	27.36	25.79	1.58	5.8%
IRA95	Spooned Sample	6.80	6.37	0.44	6.4%
A847S	Main Sample	29.80	27.33	2.48	8.3%
A847S	Spooned Sample	6.21	5.83	0.38	6.2%
A845	Main Sample	26.90	24.82	2.09	7.8%
A845	Spooned Sample	5.62	5.11	0.50	9.0%

Conclusion: For all trials samples were spooned when weighed. A845 resin approximately 9.0% free moisture, all remaining resins are approximately 7% free moisture in the samples. Trials 14-16 were DMSO trials with A103S resin. In an attempt to eliminate water from the resin, the resin slurries were partially dried. It is estimated that the free moisture of the resin for these trials was 2%.