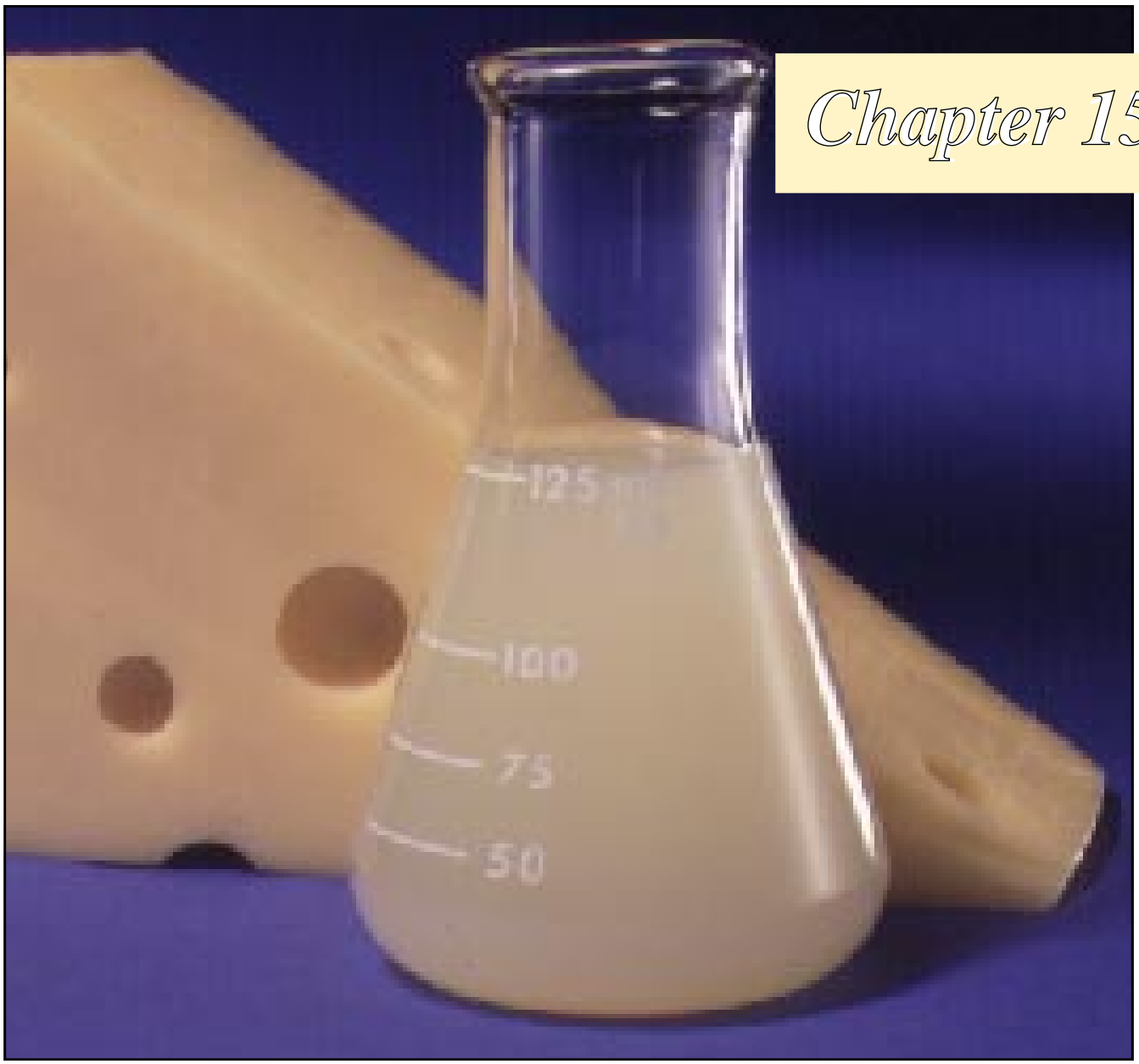


Chapter 15

Whey processing

Whey, the liquid residue of cheese and casein production, is one of the biggest reservoirs of food protein still remaining largely outside human consumption channels. World whey output, at approximately 120 million tonnes in 1990, contains some 0.7 million tonnes of relatively high-value protein, equal to the protein contents of almost 2 million tonnes of soya beans. Yet, despite the chronic protein shortage in large parts of the world, a very considerable proportion of the total whey output is still wasted - the proportion of wastage was roughly 50% in 1989-1990.

Whey comprises 80–90% of the total volume of milk entering the process and contains about 50% of the nutrients in the original milk: soluble

protein, lactose, vitamins and minerals. Whey as a by-product from the manufacture of hard, semi-hard or soft cheese and rennet casein is known as sweet whey and has a pH of 5.9 – 6.6. Manufacture of mineral-acid precipitated casein yields acid whey with a pH of 4.3 – 4.6. Table 15.1 shows approximate composition figures for whey from cheese and casein manufacture.

Table 15.1

Approximate composition of whey, %

Constituent	Cheese whey %	Casein whey %
Total solids	6.4	6.5
Water	93.6	93.5
Fat	0.05	0.04
True protein	0.55	0.55
NPN (non-protein nitrogen)	0.18	0.18
Lactose	4.8	4.9
Ash (minerals)	0.5	0.8
Calcium	0.043	0.12
Phosphorus	0.040	0.065
Sodium	0.050	0.050
Potassium	0.16	0.16
Chloride	0.11	0.11
Lactic acid	0.05	0.4

Table 15.2

Examples of utilisation of whey and whey products.

Whey product	Whey	Whey concentrate or powder					Whey protein conc. or powder				Lactose	
	Liquid whey	Natural	Sweetened	Demineralised	Deproteinised	Delactosed	Demineralised	Delactosed	Demineralised and delactosed		Crude	Refined
Animal feed	•	•		•	•	•						
Human consumption												
Baby food				•			•	•	•			•
Diet food				•			•	•	•		•	•
Sausages				•			•					
Soups		•	•	•								
Bakery products	•	•		•			•					
Salad dressings		•		•								
Whey spread/cheese		•										
Cheese, natural processed		•		•								
Beverages	•								•			
Confectionery		•	•	•								•
Pharmaceutical products												•
Yeast products	•											
Industrial products											•	•

Whey is very often diluted with water. The figures above relate to undiluted whey. As to the composition of the NPN fraction, about 30% consists of urea. The rest is amino acids and peptides (gluco macro peptide from renneting action on casein). Table 15.2 lists some fields of application for whey and whey products.

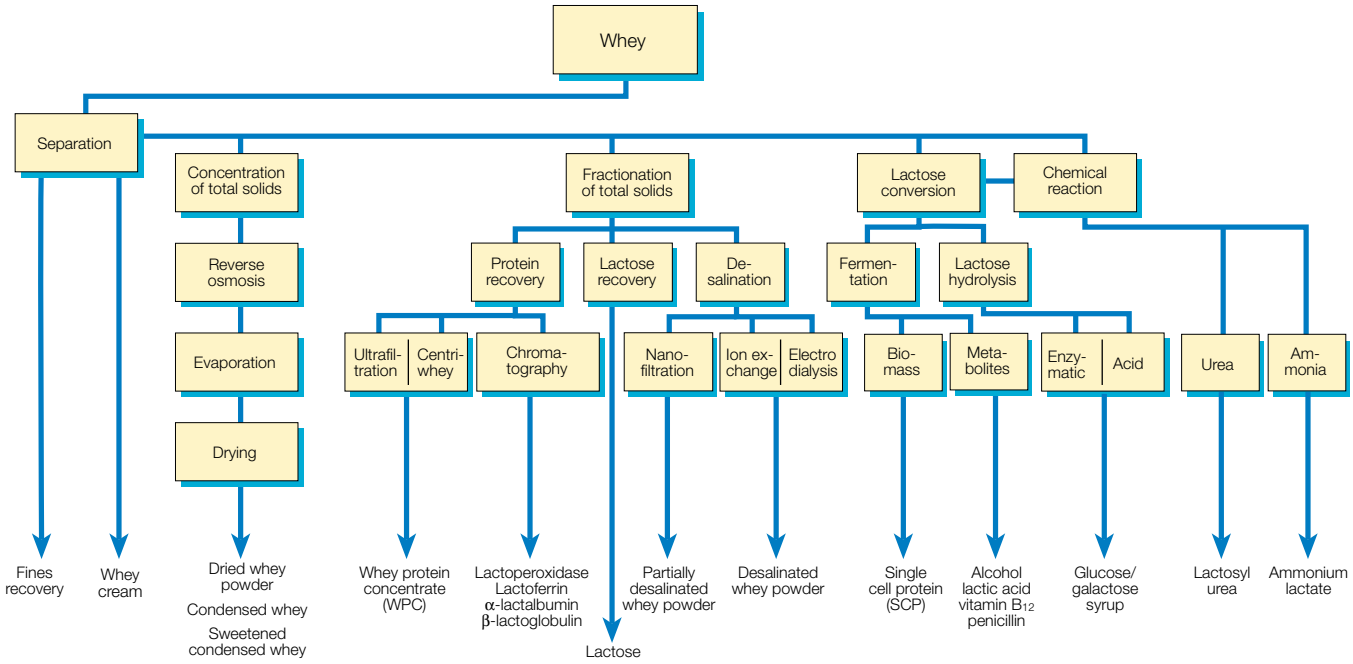


Fig. 15.1 *Whey processing alternatives.*

Although whey contains valuable nutrients, it is only in recent years that new commercial processes have been developed for the manufacture of high-quality whey products.

The block diagram in figure 15.1 summarises various processes used in the treatment of whey and its end products. Regardless of the subsequent treatment of the whey, the first stage is separation of fat and casein fines, figure 15.2 – partly to increase the economic yield and partly because these constituents interfere with subsequent treatment.

Production of whey powder, demineralised whey powder, lactose and delactosed whey powder predominates. However, a gradual shift is in progress towards new and interesting products that will transform the image of whey from an unwanted byproduct to an important raw material for the manufacture of quality products. Some of the products currently in use are described in this chapter.

Different whey processes

Whey must be processed as soon as possible after collection, as its temperature and composition promote the growth of bacteria. Otherwise the whey should be quickly cooled down to about 5°C to temporarily stop bacterial growth.

If legally permitted, whey can be preserved by addition of sodium bisulphite, typically 0.4 % calculated as sulphur dioxide (SO_2), or hydrogen peroxide (H_2O_2), typically 0. 2 % of a 30 % H_2O_2 solution.

Casein fines recovery and fat separation

Casein fines are always present in whey. They have an adverse effect on fat separation and should therefore be removed first. Various types of separation devices can be utilised, such as cyclones, centrifugal separators or rotating filters, figure 15.2.

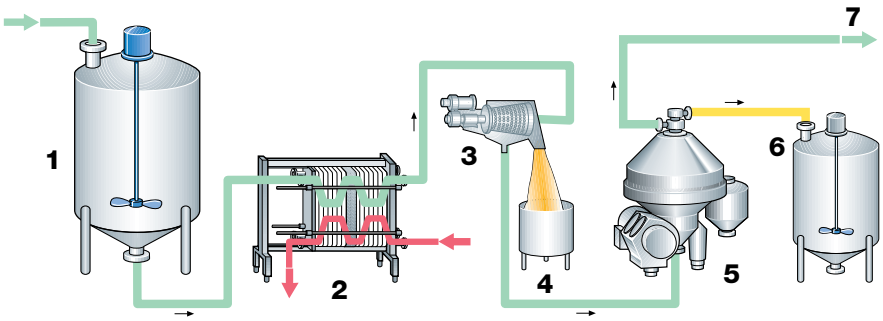


Fig. 15.2 *Fines and fat separation from whey.*

Fat is recovered in centrifugal separators.

The fines are often pressed in the same way as cheese, after which they can be used in processed cheese and, after a period of ripening, also in cooking.

The whey cream, often with a fat content of 25 – 30%, can be re-used in cheesemaking to standardise the cheese milk; this enables a corresponding quantity of fresh cream to be utilised for special cream products.

Cooling and pasteurisation

Whey which is to be stored before processing must either be chilled or pasteurised as soon as the fat has been removed. For short-time storage, 10 – 15 hours, cooling is usually sufficient to reduce bacterial activity. Longer periods of storage require pasteurisation of the whey.

Concentration of total solids

Concentration

Whey concentration traditionally takes place under vacuum in a falling-film *evaporator* with two or more stages. Evaporators with up to seven stages have been used since the mid-seventies to compensate for increasing energy costs. Mechanical and thermal vapour compression have been introduced in most evaporators to reduce evaporation costs still further.

RO (reverse osmosis) plants of tubular design have also been installed in many plants for preconcentration before the whey is sent back to the farmers and before being evaporated to final concentration.

After evaporation to 45 – 65% total solids, the concentrate is cooled rapidly to about 30°C in a plate heat exchanger and transferred to a triple-jacketed tank for further cooling to 15 – 20°C accompanied by constant stirring. This may continue for 6 – 8 hours to obtain the smallest possible crystals, which will give a non-hygroscopic product when spray dried.

Concentrated whey is a supersaturated lactose solution and, under certain conditions of temperature and concentration, the lactose can sometimes crystallise before the whey leaves the evaporator. At concentrations above a DM content of 65% the product can become so viscous that it no longer flows.

For more information on *RO* and *evaporators* see chapter 6, sections 6.4 and 6.5.

Drying

Basically whey is dried in the same way as milk, i.e. in drum or spray dryers, see under milk powder in chapter 17.

The use of *drum* dryers involves a problem: it is difficult to scrape the layer of dried whey from the drum surface. A filler, such as wheat or rye bran, is therefore mixed into the whey before drying to make the dried product easier to scrape off.

Spray drying of whey is at present the most widely used method of dry-

ing. Before being dried, the whey concentrate is usually treated as mentioned above to form small lactose crystals, as this results in a non-hygroscopic product which does not go lumpy when it absorbs moisture.

Acid whey from cottage cheese and casein production is difficult to dry due to its high lactic acid content. It agglomerates and forms lumps in the spray dryer. Drying can be facilitated by neutralisation and additives, such as skimmilk and cereal products, but this type of whey is not processed nowadays.

Fractionation of total solids

Protein recovery

Whey proteins were originally isolated through the use of various precipitation techniques, but nowadays membrane separation (fractionation) and chromatographic processes are used in addition to both precipitation and complexing techniques. The process that has been most extensively used for separation of whey proteins from whey serum is heat denaturation. The precipitated protein formed by this process is either insoluble or sparingly soluble depending on the conditions prevailing at denaturation; it is called heat-precipitated whey protein (HPWP).

Fink and Kessler (1988) state that a maximum whey protein denaturation rate of 90% is possible for all denaturable fractions. Proteose peptone, comprising some 10% of the fraction, is considered undenaturable.

Native whey proteins, as constituents of whey powders, can easily be produced by careful drying of whey. Because of their unfavourable composition, they have only a limited application in foodstuffs (only some 11% protein and a high lactose and ash content). Isolation of native whey proteins has therefore been developed. The native whey proteins obtained by membrane separation or ion exchange possess good functional properties, as to solubility, foaming, emulsion formation and gelling.

Protein recovery by UF

Native protein concentrates have a very good amino acid profile with high proportions of available lysine and cysteine.

Whey protein concentrates (WPC) are powders made by drying the retentates from ultrafiltration of whey. They are described in terms of their protein content, % protein in dry matter, ranging from 35% to 85%. To make a 35% protein product the liquid whey is concentrated about 6-fold to an approximate total dry solids content of 9%.

Example: 100 kg of whey yields approximately 17 kg of retentate and 83 kg of permeate at close to 6-fold (5.88) concentration. Table 15.3 shows the compositions of the feed (whey) and the resulting retentate and permeate.

Table 15.3

Composition of whey and resulting retentate and permeate.

Component	Weight in 100 kg Ordinary whey		Weight in 17 kg Retentate		Weight in 83 kg Permeate	
	kg	%	kg	%	kg	%
True protein	0.55	0.55	0.55	3.24	0	0
Lactose	4.80	4.80	0.82	4.82	3.98	4.80
Ash	0.80	0.80	0.14	0.82	0.66	0.80
NPN*	0.18	0.18	0.03	0. 18	0.15	0.18
Fat	0.03	0.03	0.03	0.18	0	0
Total DM	6.36	6.36	1.57	9.24	4.79	5.78

* NPN = Non-protein nitrogen

% protein in dry matter according to the values in table 15.3:

$$\frac{100 \times 0.55}{1.57} = 35$$

In concentration most of the true protein, typically > 99%, is retained together with almost 100% of the fat. The concentrations of lactose, NPN and ash are generally the same in the retentate serum and permeate as in the original whey, but a slight retention of these components is reported. The overall retention figures, however, depend very much on:

- The type of membrane
- The flux
- The character of the feed (prediluted with water, pre-concentrated after demineralisation, etc.)

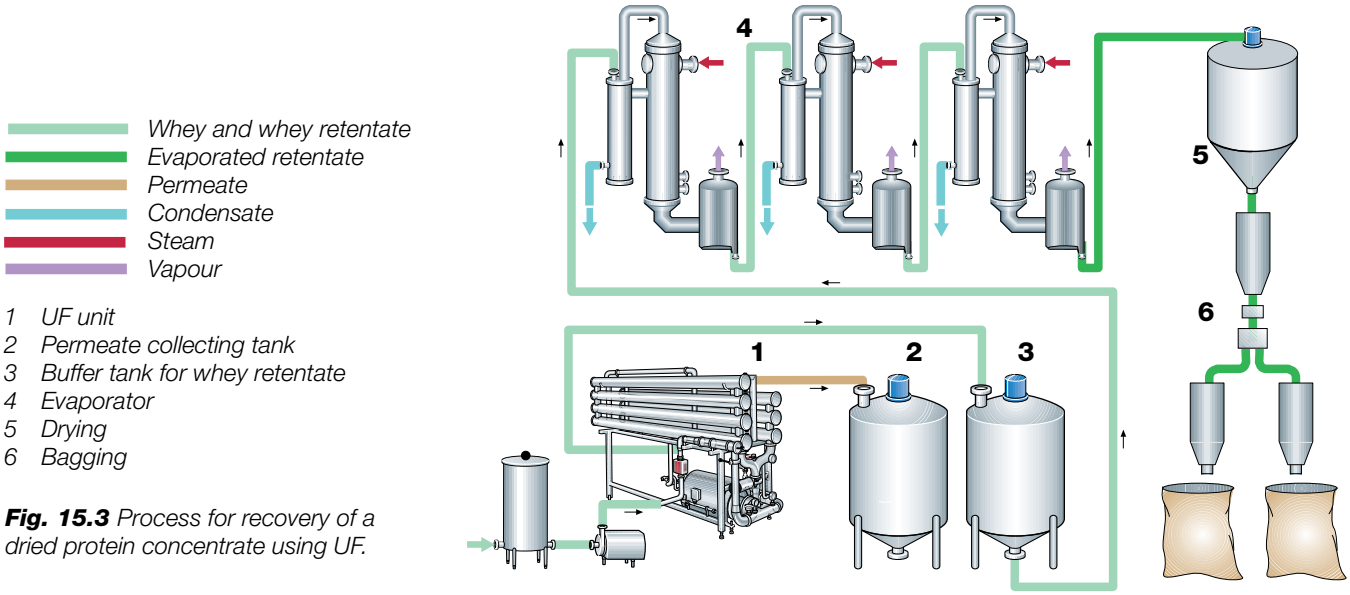


Fig. 15.3 *Process for recovery of a dried protein concentrate using UF.*

To obtain an 85% protein concentrate the liquid whey is first concentrated 20 – 30-fold by direct ultrafiltration to a solids content of approximatively 25%; this is regarded as the maximum for economic operation. It is then necessary to diafilter the concentrate to remove more of the lactose and

Table 15.4

Composition in % of some whey protein concentrate powders

Product	1	2	3	4
Protein in dry matter	35	50	65	80
Moisture	4.6	4.3	4.2	4.0
Crude protein (Nx6.38)	36.2	52.1	63.0	81.0
True protein	29.7	40.9	59.4	75.0
Lactose	46.5	30.9	21.1	3.5
Fat	2.1	3.7	5.6	7.2
Ash	7.8	6.4	3.9	3.1
Lactic acid	2.8	2.6	2.2	1.2

Product specification:

1 Skimmilk substitute, 35% protein in dry matter
2 Protein supplement to other foods, 50% protein in dry matter
3 Practical limit of protein by ultrafiltration alone, 65% protein in dry matter
4 Product of ultrafiltration plus diafiltration, 80% protein in dry matter

ash and raise the concentration of protein relative to the total dry matter. Diafiltration is a procedure in which water is added to the feed as filtration proceeds in order to wash out low molecular components which will pass through the membranes, basically lactose and minerals.

Table 15.4 shows the compositions of some typical whey protein concentrate (WPC) powders.

A process line for production of dried protein using UF is shown in figure 15.3. About 95% of the whey is collected as permeate, and protein concentrations as high as 80 – 85% (calculated on the DM content) can be obtained in the dried product. For further details about UF see chapter 6.4, membrane filters.

Defatting of whey protein concentrate (WPC)

Defatted WPC powder containing 80 – 85% protein dry matter is a very interesting option for some applications, e.g. as a replacement for white of egg in whipped products such as meringues and as a valuable ingedient in various foods and fruit beverages.

Treatment of the whey retentate from a UF plant in a microfiltration (MF) plant can reduce the fat content of 80 – 85% WPC powder from 7.2% to less than 0.4%. Microfiltration also concentrates fat globule membranes and most of the bacteria in the MF retentate, which is collected and disposed of separately. The defatted MF permeate is routed to a second UF plant for further concentration; this stage also includes diafiltration.

As figure 15.4 shows, the whey is preheated (1) and separated (2) to recover as much fat as possible in the form of 25 – 30% cream. This cream can be re-used for fat standardisation of cheese milk. The separation stage also removes fines. After this the whey is pasteurised (1) and cooled to about 55 – 60°C before being transferred to an intermediate holding tank.

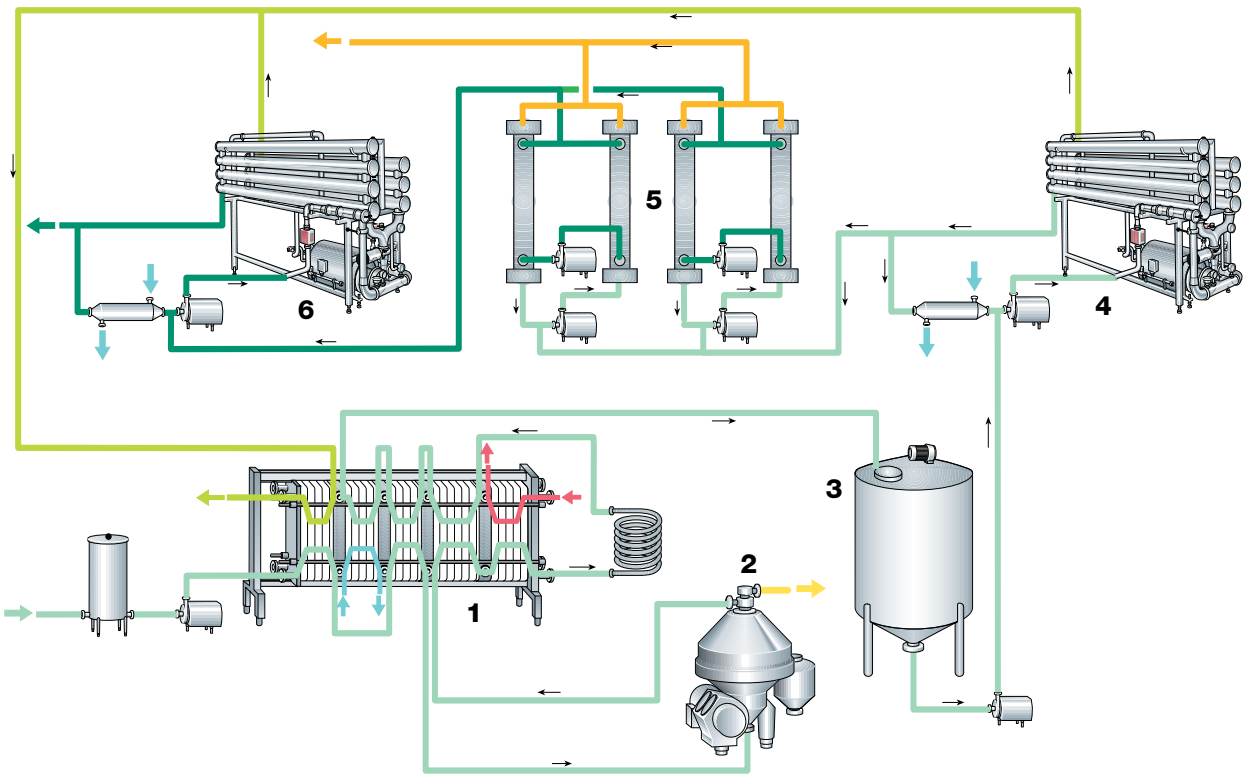
After a hold the whey is pumped to the first UF plant (4), where it is concentrated about threefold. The retentate is pumped to the MF plant (5), while the permeate goes to a collecting tank after regenerative cooling (1).

The retentate from MF treatment, which contains most of the fat and bacteria, is collected separately, and the defatted permeate is forwarded to further ultrafiltration with diafiltration (6). The resulting WPC with about 20 – 25% DM is then spray-dried to reduce the moisture content to a maximum of 4% before bagging.

Whey/retentate
Cream
Permeate
Defatted protein retentate for drying
High fat retentate from MF plant
Cooling medium
Heating medium

1 *Pasteuriser*
2 *Whey cream separator*
3 *Holding tank*
4 *First UF plant*
5 *MF plant*
6 *Second UF plant*

Fig. 15.4 *Process for defatting of whey protein retentate.*

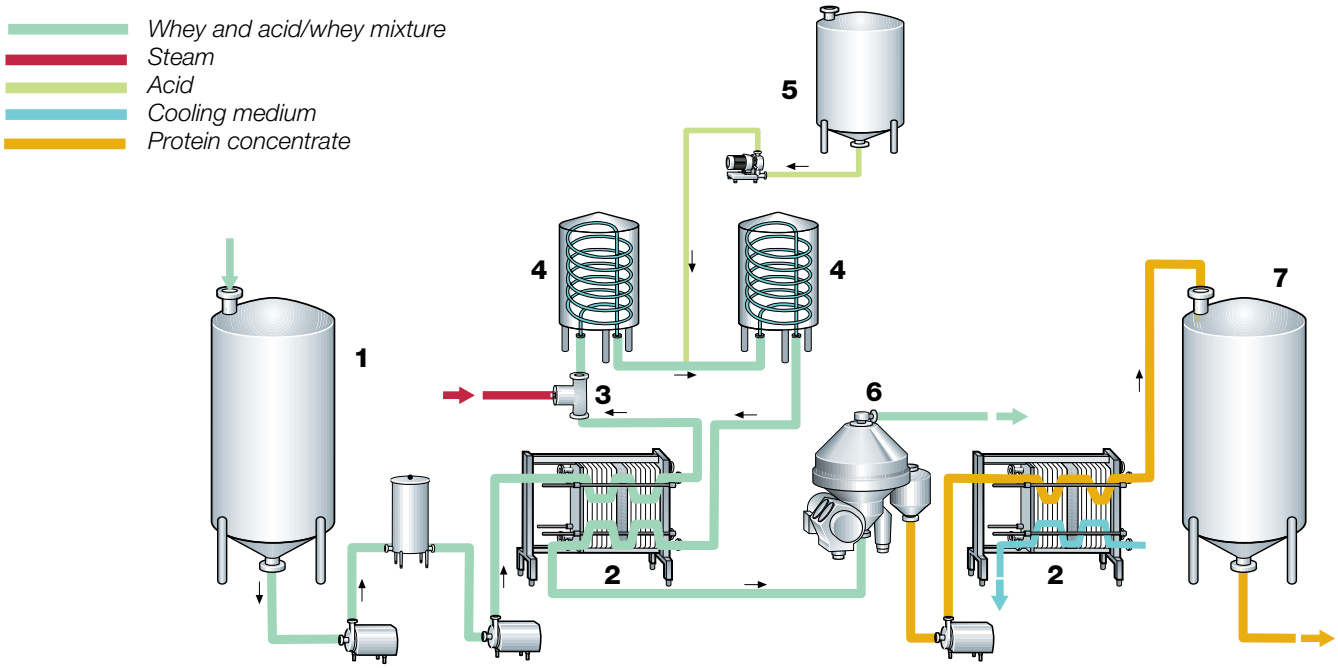


Recovery of denatured whey protein

In general, serum protein or whey proteins cannot be precipitated by rennet or acid. It is however possible to precipitate whey proteins with acid if they are first denatured by heat. The process is divided into two stages:

- Precipitation (denaturing) of the protein by a combination of heat treatment and pH adjustment,
- Concentration of proteins by centrifugal separation.

Denatured whey proteins can be mixed with cheese milk prior to renneting; they are then retained in the lattice structure formed by the casein molecules during coagulation. This discovery led to intensive efforts to find a method of precipitating and separating whey proteins as well as a technique for optimising the yield while retaining the characteristic aroma and texture of the cheese in question.



1 *Whey collecting tank*
2 *Plate heat exchanger*
3 *Steam injector*
4 *Holding tube*
5 *Acid tank*
6 *Clarifier*
7 *Collecting tank for denaturated whey protein*

Fig. 15.5 *Recovery of denatured whey proteins.*

Figure 15.5 shows the Centri-Whey process line for manufacture of denatured whey proteins. After pH adjustment the whey is pumped via an intermediate tank (1) to a plate heat exchanger (2) for regenerative heating. The temperature of the whey is raised to 90 – 95°C by direct steam injection (3) before it passes through a tubular holding section (4) with a holding time of 3 – 4 minutes. Acid is introduced during this stage to lower the pH. The acid is either organic or inorganic (e.g. lactic acid or edible hydrochloric acid) as stipulated.

Those proteins that can be, and have been, modified by heat are precipitated within 60 seconds in a tubular holding section (4).

After regenerative cooling to about 40°C the precipitated proteins are separated from the liquid phase in a solids-ejecting clarifier (6). The clarifier discharges, at intervals of about 3 minutes, the accumulated protein in the form of a 12 – 15% concentrate of which about 8 – 10% is protein. This method results in 90 – 95% recovery of the coagulable proteins.

The addition of concentrated whey protein to cheese milk – principally in the manufacture of soft and semi-hard cheeses – causes only minor changes in the coagulating properties. The structure of the curd becomes finer and more uniform than with conventional methods. The processed whey proteins are more hydrophilic than casein. In the making of Camembert cheese, for example, an increase in yield of 12% has been reported.

Chromatographic isolation of lactoperoxidase and lactoferrin

Generally speaking, use of natural bioactive agents is of very great interest in products like infant formulas, health foods, skin creams and toothpaste. Examples of such components are the bioactive proteins lactoperoxidase (LP) and lactoferrin (LF) existing at low contents in whey, typically 20 mg/l of LP and 35 mg/l of LF. The Swedish Dairies Association (SMR) has developed a patented process based on chromatography for isolation of these proteins from cheese whey on an industrial scale.

The basic principle underlying the process is the fact that both LP and LF have isoelectric points in the alkaline pH area, 9.0 – 9.5, which means that these proteins are positively charged at the normal pH of sweet whey, 6.2 – 6.6, while the rest of the whey proteins e.g. β-lactoglobulin, α-lactalbumin and bovine serum albumin are negatively charged in the same pH range. A fundamentally suitable process for isolation of LP and LF is therefore to pass a specially designed cation exchange resin for selective adsorption. The LP and LF molecules thus bind to the negatively charged functional group of the cation exchanger by charge interaction, leading to fixation of these molecules on the ion exchange resin, while the other whey proteins pass through because of their negative charge.

To make the process industrially viable, some basic criteria have to be satisfied. One of them is the need for a "particle-free" whey to maintain a high flow rate during the loading phase, because very large volumes of whey have to pass the ion exchange resin to achieve saturation. Cross-flow microfiltration (MF) with a pore size of 1.4 µm operated under a uniform transmembrane pressure (UTP) has proved to be a successful technique for getting particle-free whey. Stable flux of 1 200 – 1 500 l/m^{2}h is easily sustained for 15 – 16 hours. This type of pretreatment of the whey avoids build-up of increasing back pressure over the ion exchange column.

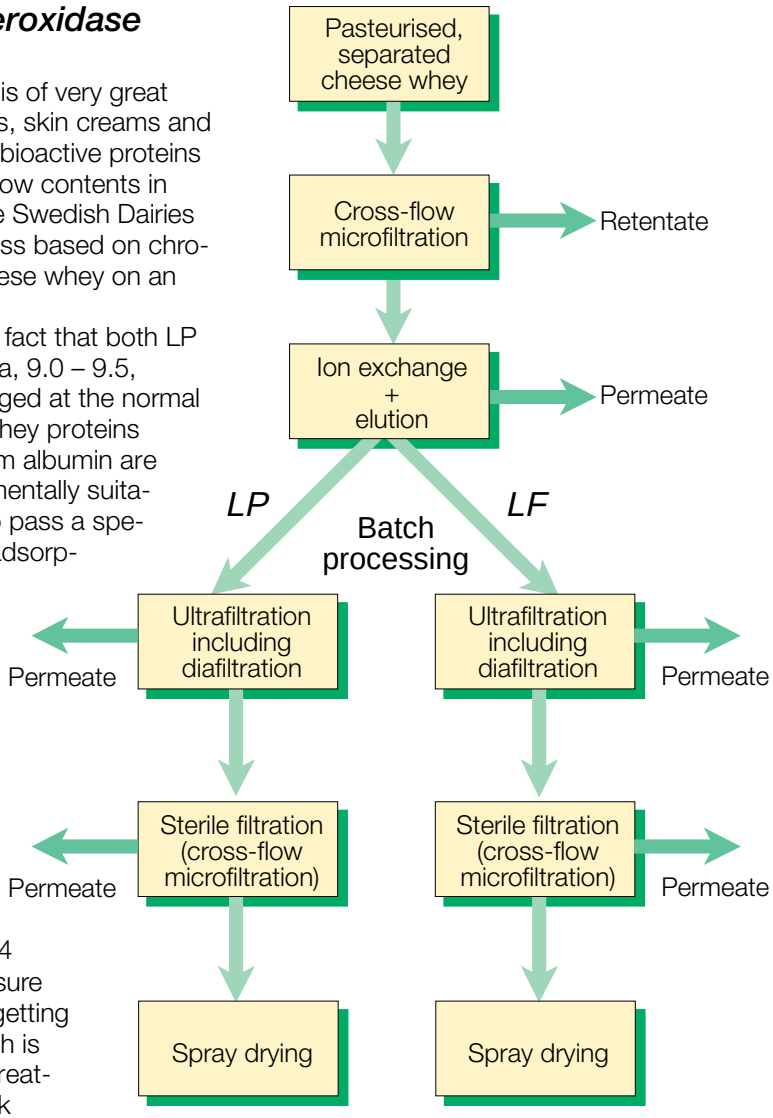


Fig. 15.6 *Block diagram for isolation of lactoperoxidase (LP) and lactoferrin (LF) from whey.*

The ion exchange resin has a capacity to adsorb altogether 40 – 45 g of LP and LF per litre of resin before breakthrough occurs. With a resin bed volume of 100 l, almost 100 000 l of whey can be treated per cycle.

With properly chosen conditions for elution of adsorbed bioactive proteins on the column it is possible to obtain very pure fractions of LP and LF. Salt solutions of different strengths are used for this step. The proteins in the eluates occur in fairly concentrated form, of the order of 1% by weight. The ion exchange step thus concentrates LP and LF by a factor of almost 500 compared to the original whey. Further processing of the eluates by UF and diafiltration yields very pure protein products, appr. 95% purity. Finally, after sterile filtration in a cross-flow microfilter with 0.1 – 0.2 µm pores, the protein concentrates are spray dried. The overall process is illustrated in figure 15.6.

Lactose recovery

Lactose is the main constituent of whey. There are two basic methods of recovery, depending on the raw material:

- Crystallisation of the lactose in untreated but concentrated whey;
- Crystallisation of lactose in whey from which the protein has been removed by UF or some other method before concentration.

Both methods produce a mother-lye, molasses, which can be dried and used as fodder. The feed value can be increased considerably if the molasses is desalinated and if high-quality proteins are added.

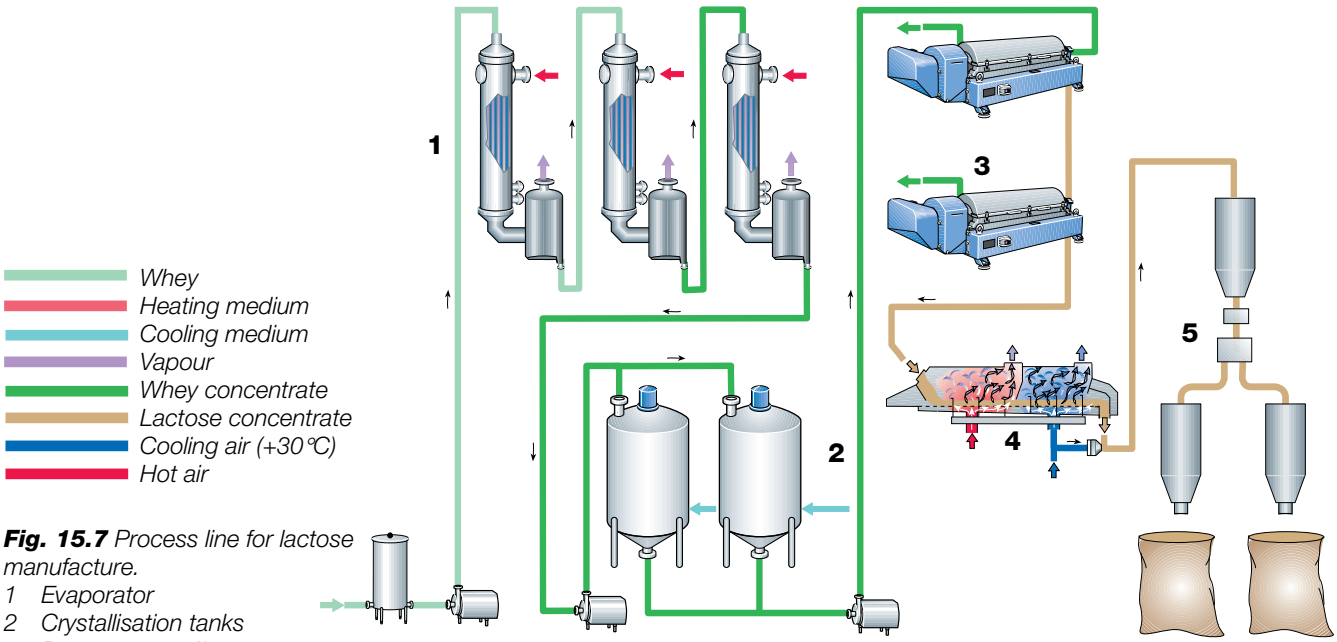


Fig. 15.7 *Process line for lactose manufacture.*
1 Evaporator
2 Crystallisation tanks
3 Decanter centrifuges
4 Fluidised-bed dryer
5 Packing

Crystallisation

The crystallisation cycle is determined by the following factors:

- Crystal surface available for growth
- Purity of the solution
- Degree of saturation
- Temperature
- Viscosity
- Agitation of the crystals in the solution

Several of these factors are mutually related to each other, for example degree of saturation and viscosity.

Figure 15.7 shows a production line for manufacture of lactose. The whey is first concentrated by evaporation to 60 – 62% DM and then transferred to crystallisation tanks (2) where seed crystals are added. Crystallisation takes place slowly according to a predetermined time/temperature programme. The tanks have cooling jackets and equipment for control of the cooling temperature. They are also fitted with special agitators.

After crystallisation the slurry proceeds to decanter centrifuges (3) for separation of the crystals, which are dried (4) to a powder and following grinding, typically in a hammer mill, and sifting the lactose is packed (5).

For efficient and simple separation of lactose crystals from the mother liquor, crystallisation must be arranged so that the crystals exceed 0.2 mm in size – the larger the better for separation.

The degree of crystallisation is determined in principle by the quantity of β-lactose converted to the desired α-lactose form, and the cooling of the concentrate must therefore be carefully controlled and optimised.

Lactose separation

Various types of centrifuges can be used for harvesting lactose crystals. One is the horizontal decanter centrifuge, figure 15.8, which operates continuously and has a screw conveyor for unloading the lactose. Two machines are installed in series. The lactose from the first is reprocessed in the second for more efficient separation. During separation, impurities are washed from the lactose so that a high degree of purity is obtained. The residual moisture content of the lactose after the second separation stage is < 9% and pure lactose accounts for about 99% of the dry solids.

Drying

The lactose is dried after separation to a residual moisture content of 0.1–0.5%, depending on the future use of the product. The temperature during drying should not exceed 93°C, as β-lactose is formed at higher temperatures. The drying time must also be taken into consideration. During quick

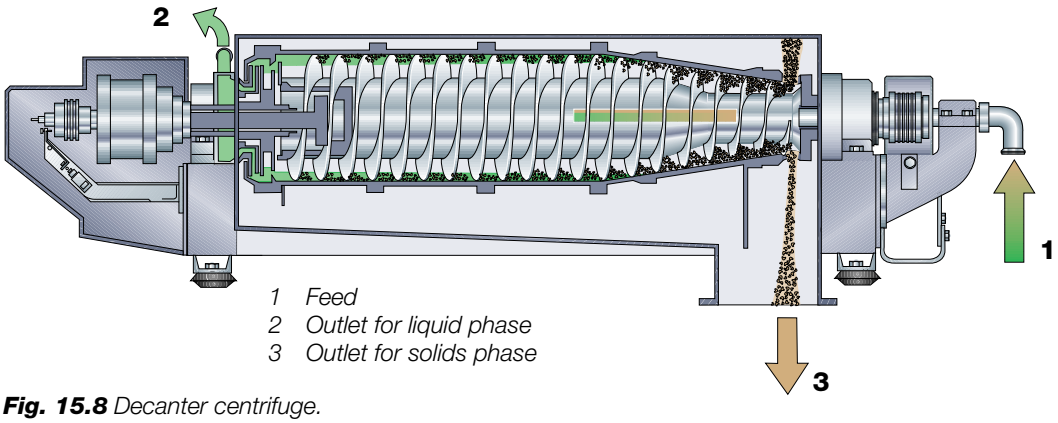


Fig. 15.8 *Decanter centrifuge.*

drying a thin layer of amorphous (shapeless, non-crystalline) lactose tends to form on the α-hydrate crystal, and this may later result in formation of lumps. Drying usually takes place in a fluidised bed drier. The temperature is maintained at 92°C and the drying time is 15 – 20 minutes. The dried sugar is transported by air at a temperature of 30°C, which also cools the sugar.

The crystals are normally ground to a powder immediately after drying and are then packed.

Refining of lactose

A higher degree of purity is required for some applications, e.g. pharmaceutical manufacturing processes. Lactose for such use must therefore be further refined. During refining the lactose is re-dissolved in hot water to a concentration of 50%. Active carbon, phosphate and a filtration agent are added at the same time. After filtration the lactose solution is transferred to a tank where crystallisation takes place. The purified lactose is then separated, dried, ground and packed.

Demineralisation (Desalination)

As whey has a fairly high salt content, about 8 – 12% calculated on dry weight, its usefulness as an ingredient in human foods is limited. By having the whey demineralised, various fields of application can however be found for whey which is partially (25 – 30%) or highly (90 – 95%) demineralised.

Partially demineralised whey concentrate can for instance be used in the manufacture of ice-cream and bakery products or even in quarg, whereas *highly* demineralised whey concentrate or powder can be utilised in formulas for infants and, of course, in a very wide group of other products.

Principles of demineralisation

Demineralisation involves removal of inorganic salts together with some reduction in the content of organic ions such as lactates and citrates.

The *partial* demineralisation is mainly based on utilisation of cross-flow membranes specially designed to “leak” particle species that have radii in the nanometer (10^{-9} m) range. This type of filtration is called nanofiltration (NF).

The *high* degree desalination is based on either of two techniques:

- Electrodialysis
- Ion exchange

Partial demineralisation by NF

By using a specially designed “leaky” RO membrane small particles like certain monovalent ions, e.g. sodium, potassium, chloride and small organic molecules like urea and lactic acid can escape through the membrane together with the aqueous permeate. This membrane process is known by various names such as ultraosmosis, “leaky” RO and nanofiltration (NF).

Because of their greater compactness, *spiral wound membranes* are most often used in new installations today (1994). For further information about this type of membrane see chapter 6.4, membrane filters.

Examples of permeation rates of normal sweet whey constituents during nanofiltration are given in table 15.5.

As the table shows, reduction of the chloride content in sweet whey can be as high as 70% and that of sodium and potassium 30 – 35%. The reason for this difference in elimination of ions is the need of maintaining an electrochemical balance between negative and positive ions.

A critical aspect of nanofiltration in whey processing is that the leakage of lactose must be kept to a minimum (<0.1%) to avoid problems with high BOD (biological oxygen demand) in the waste water (permeate). Installation of NF equipment in whey processing can be considered in the following situations :

- As a low-cost alternative to diminish the salty taste of ordinary sweet whey powder;
- As a preliminary step to more complete demineralisation of whey by electrodialysis and ion exchange;
- For acid removal in hydrochloric and lactic acid casein whey; note that the permeation rate is low for lactate ions but high for free lactic acid molecules;
- For salt reduction in salted whey (e.g. salt drippings in Cheddar cheese production).

Table 15.5

Permeation rates of normal sweet whey constituents during nanofiltration

Conditions		**Reduction**	**%**
Final DM	22%	Potassium	31
Concentration factor	4 x	Sodium	33
Temperature	21°C	Chloride	67
Pressure	2.5 MPa (25 bar)	Calcium	3
		Magnesium	4
		Calcium	3
		Magnesium	4
		Phosphorus	6
		Citrate	0
		Lactate	<3
		Ash	25
		Total DM	3
		NPN	27
		Lactose	<0.03

High degree demineralisation

Electrodialysis

Electrodialysis is defined as the transport of ions through non-selective semi-permeable membranes under the driving force of a direct current (DC) and an applied potential. The membranes used have both anion and cation exchange functions, making the electrodialysis process capable of reducing the mineral content of a process liquid, for example seawater or whey.

Figure 15.9 is a schematic picture of an electrodialysis unit. It consists of a number of compartments separated by alternate cation and anion exchange membranes which are spaced about 1 mm or less apart. The end compartments contain electrodes. There can be as many as 200 cell pairs between each pair of electrodes.

The two electrodes at each end of the cell stack have separate rinse channels as shown in figure 15.9, through which a separate acidified stream is circulated to protect the electrodes from chemical attack.

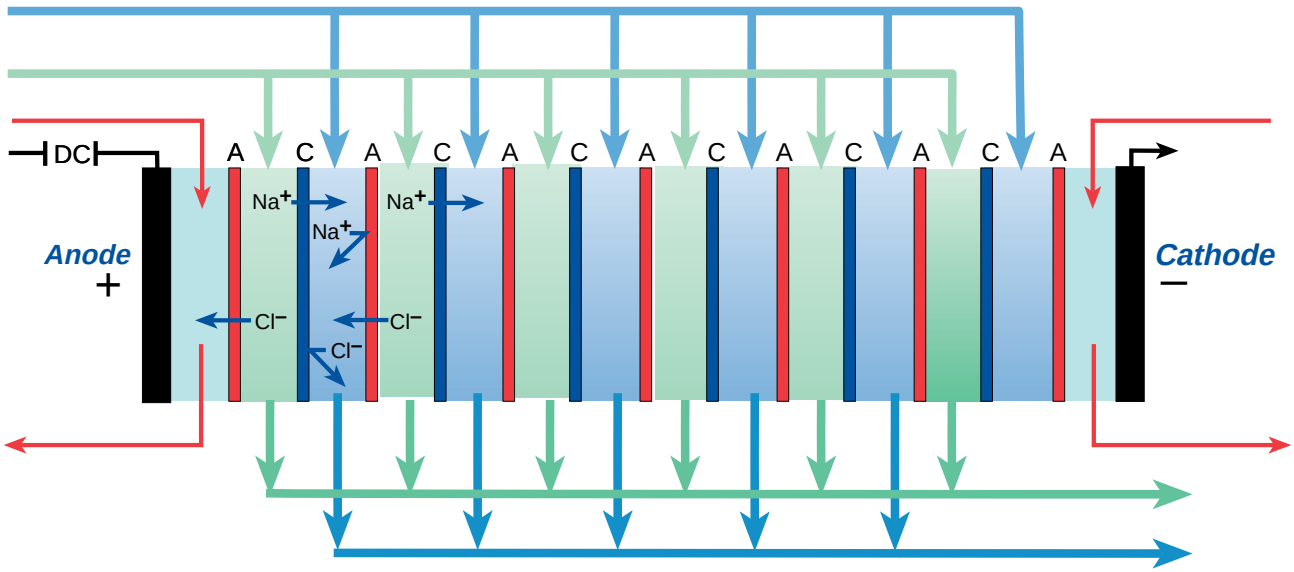


Fig. 15.9 *Cell packs for electrodialysis.*

A = Anions = positively charged
C = Cations = negatively charged
DC = Direct Current

Whey
Salt (brine) solution
Electrode rinse solution

For whey treatment, the whey feed and acidified brine pass through alternate cells in the stack, whose construction can be likened to that of a plate heat exchanger or plate sheet ultrafiltration module.

Operating principle

Alternate cells in the electrodialysis stack act as concentration and dilution cells respectively. Whey is circulated through the dilution cells, and a 5% brine carrier solution through the concentration cells.

When direct current (DC) is applied across the cells, cations attempt to migrate to the cathode and anions to the anode as shown in figure 15.9. However, completely free migration is not possible because the membranes act as barriers to ions of like charge. Anions can pass through an anion membrane, but are stopped by a cation membrane. Conversely, cations can pass through a cation membrane but not an anion membrane. The net result is depletion of ions in the whey (dilution) cells. The whey is thus demineralised, to an extent determined by the ash content of the whey, residence time in the stack, current density and flow viscosity.

The electrodialysis plant can be run either continuously or in batches. A batch system, which is often used for demineralisation rates above 70%, can consist of one membrane stack over which the process liquid, e.g. whey, is circulated until a certain ash level is reached. This is indicated by the conductivity of the process liquid. The holding time in a batch system can be as long as 5 – 6 hours for 90% demineralisation at 30 – 40°C. Preconcentration of the whey to 20 – 30% DM is desirable with regard to capacity utilisation and electric power consumption. The whey concentrate should be clarified before it enters the electrodialysis unit.

The high process temperature means that there is a risk of bacteriological growth taking place in the product. A bacteriostatic compound such as hydrogen peroxide is therefore often added to the whey, when allowed. The process liquid heats up during the process, so a cooling stage is needed to maintain the process temperature. In a continuous plant, consisting of five membrane stacks in series, the holding time can be reduced to 10 – 40 minutes. The maximum demineralisation rate of such a plant is often limited to about 60 – 70%. In relation to capacity, the installed membrane area is much larger in a continuous plant than in a batch plant.

An electrodialysis plant can easily be automated and furnished with a programmed CIP system. The cleaning sequence normally includes water rinse, cleaning with an alkaline solution (max. pH 9), water rinse, cleaning with hydrochloric acid (pH 1) and a final water rinse. A typical cleaning programme takes 100 minutes.

Power supply and automation

Direct current is used in the electrodialysis plant, which should have facilities for regulating current in the range of 0 – 185 A and voltage in the range of 0 – 400 V. Flow rates, temperatures, conductivity, pH of process water and product, product inlet pressure, pressure difference between the stacks and current, as well as voltage over each membrane stack, are monitored and controlled during production.

Limiting factors in electrodialysis

A major limiting factor for using electrodialysis in dairy processing is the cost of replacing membranes, spacers and electrodes, which constitutes 35 – 40% of the total running costs in the plant. Replacement is necessary due to fouling of the membranes, which in turn is caused by:

- Precipitation of calcium phosphate on the cation exchange membrane surfaces
- Deposition of protein on the anion exchange membrane surfaces.

The first problem can be handled by proper flow design over the membrane surface and regular acid cleaning.

Protein deposits are the main factor in shortening the lifetime of the anion membranes. The background to this problem is as follows: at the normal pH of whey, the whey proteins can be regarded as large negative ions (anions) and move as such under the influence of the electrical field in the stack. These molecules, being too large to pass through the anion exchange membranes, are deposited as a thin protein layer on the faces of the anion exchange membranes in the whey compartments. Techniques such as polarity reversal can be used to dislodge these deposited materials from the membrane.

Although frequent high-pH cleaning removes most of the deposits, disassembly of the stack for manual cleaning is recommended at intervals of 2 – 4 weeks.

The processing cost of electrodialysis depends very much on the demineralisation rate. Increasing the capacity in steps from 50% to 75% to 90% doubles the processing cost per step. This means that it is four times as expensive per kilo of product solids to demineralise to 90% than to 50%; the reason is that plant capacity is reduced at 90% demineralisation.

Water treatment, electric power, chemicals and steam account for the operating costs of a demineralisation plant. Waste water treatment is a particularly heavy item. During production lactose leaks through the membranes at a rate of 7 – 10% at 90% demineralisation. The phosphate removed from whey also accumulates in the waste stream. The cost of electric power amounts to 10 – 15% of the processing cost, while the chemicals used in the process, mainly hydrochloric acid, account for less than 5%. The cost of steam used for preheating the product and cooling costs for control of process temperature are 10 – 15%, depending on the demineralisation level.

Electrodialysis is best for demineralisation levels below 70%, where it is very competitive compared to ion exchange.

Electrodialysis is best for demineralisation levels below 70%, where it is very competitive compared to ion exchange.

Ion exchange

In contrast to electrodialysis, the process which removes ionisable solids from solutions on a continuous electro-chemical basis, an ion exchange process employs resin beads to adsorb minerals from solution, in exchange for other ionic species. The resins have a finite capacity for this, so that when they are completely saturated, the adsorbed minerals must be removed and the resins regenerated before reuse. Normally the resins are used in fixed columns of suitable design.

Ion exchange resins are macromolecular porous plastic materials, formed into beads with diameters in the range of 0.3 to 1.2 mm for technical applications. Chemically they act as insoluble acids or bases which, when converted into salts, remain insoluble. The main characteristic of ion exchange resins is their capacity to exchange the mobile ions they contain for ions of the same charge sign, contained in the solution to be treated. A

simple example of this reaction is shown for sodium chloride removal, where R is the exchange group bound to the insoluble resin.

Cation exchange	$R - H + Na^+ = R - Na + H^+$	resin in H^+ form
Anion exchange	$R - OH + Cl^- = R - Cl + OH^-$	resin in OH^- form

The reaction above is deliberately written as an equilibrium, because the direction in which the reaction goes depends on the ion concentration in the liquid and in the solids phase of the resin. The equilibrium is characterised by a constant. On regeneration the reaction is reversed when the sodium-laden ion exchange resin is treated with, say, a 4% hydrochloric acid solution. The high concentration of hydrogen ions in the acid drives the equilibrium to the left.

The equilibrium constant varies depending on ion species, which gives the selectivity of ion exchange processes. Generally speaking, multivalent ions have higher selectivity than monovalent ones and ions of the same valence are selected by size, large ions having higher selectivity. For cations typically found in dairy process streams, selectivity decreases in the order $Ca^{2+} > Mg^{2+} > K^+ > Na^+$.

Similarly, anion exchange selectively can be classified in the following way: citrate^{3-} > HPO_4^{2-} > NO^{3-} > Cl^-.

In practice this means that the ion exchanger, after being exhausted by a liquid containing different ion species, will exist in different forms along the length of the column as described in figure 15.10. This figure shows what happens in a column treating ordinary raw water in a cation exchanger loaded in the hydrogen ion form. The situation after regeneration with acid is also shown. It can be seen that the ions that remain longest in the cation-exchange column are Na ions. This can be understood from the selectivity order described above.

Going back to the picture of the exhausted cation-exchange column in the figure, the segregated distribution of ions means that Na ions leak first, followed by Mg and Ca ions. An initial ion leakage in the exhaustion phase may occur when the ion exchanger is not fully regenerated, but after a while the Na ions are eluted and replaced by H ions (see figure 15.10). The status of the lower part of the ion exchanger determines the leakage of ions from the process liquid.

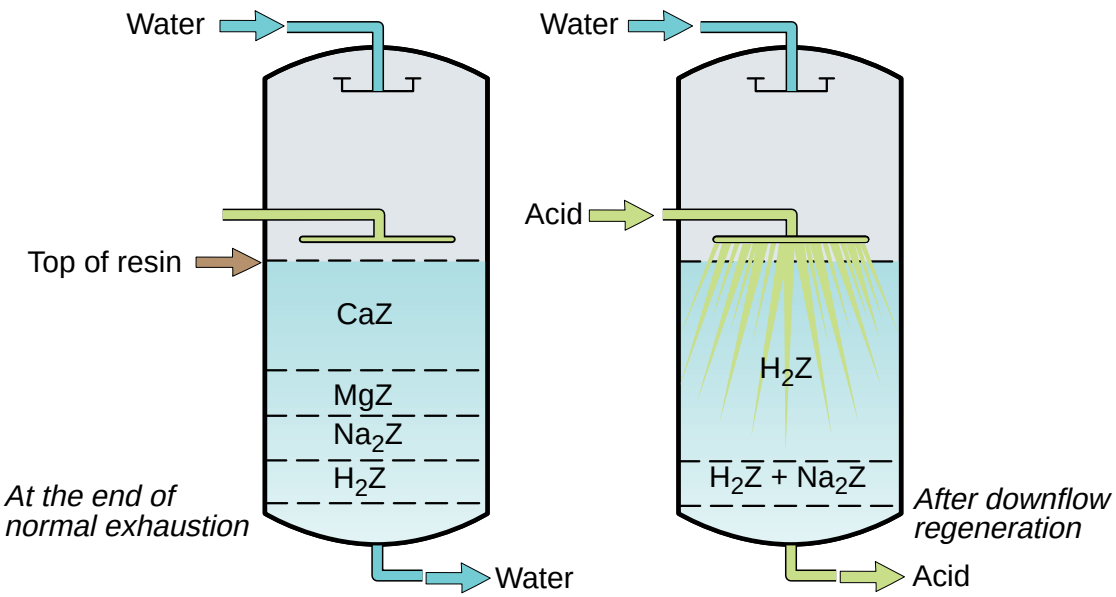


Fig. 15.10 *Cation exchanger resin bed before and after regeneration with acid.*

Ion exchange resin characteristics

Ion-exchange resins in industrial use today are based on polymeric plastic materials to build up the porous matrix structure. Common materials are polystyrene/divinyl benzene and polyacrylate. Functional groups are chemically bound to this matrix structure. Typical groups are:

• Sulphonic groups	$-SO_3^- H^+$	(strong acid cation exchanger)
• Carboxyl groups	$-COO^- H^+$	(weak acid cation exchanger)
• Quatenary amine	$N^+ OH^-$	(strong base anion exchanger)
• Tertiary amines	$-NH^+ OH^-$	(weak base anion exchanger)

Both strong base and strong acid exchangers are fully ionised in the whole pH interval, 0 – 14. Weak base and weak acid ion exchangers have a restricted pH area in which they are active. Weak acid cation exchangers cannot normally be used in the low pH range, 0 – 7, because the carboxyl groups are mainly present in their free acid form as determined by their acid/base dissociation constant (often expressed as $pK_a = -10$ logarithm of the dissociation constant). At pH values higher than pK_a the carboxylic groups are in their salt form, and can consequently participate in ion exchange reactions. As a contrast, weak base anion exchange resins are only active in the low pH range, 0 – 7.

From the ease-of-regeneration point of view it is beneficial to use weak resins whenever possible. They can be regenerated with acid/base respectively with an excess of only 10 – 50% of the theoretically needed amount. Strong resins require an acid/base excess of 300 – 400% compared with the theoretical value for regeneration. For demineralisation according to the classical procedure, a strong acid cation exchanger regenerated in the hydrogen form is combined with a weak base anion exchanger working in the free base (hydroxyl) form. It is not possible to use a weak acid cation exchanger instead of a strong one, because of the very advantageous equilibrium for exchange of cations for the hydrogen bound to the hydroxylic groups.

Other important characteristics of ion exchangers, which are not further discussed, are:

- Ion exchange capacity
- Swelling properties
- Mechanical strength
- Fluidisation during backflushing of the bed
- Pressure drop
- Flow-velocity restrictions
- Water rinse requirements after regeneration

Ion exchange processes for demineralisation

Demineralisation by ion exchange has long been an established process for water treatment but has also been adopted during the past two decades for "de-ashing" of whey. Whey is not a uniform product as to composition. Whey from an acid casein/cheese curd has a pH of 4.3 – 4.6, while the pH of sweet whey is 6.3 – 6.6. The main difference between these two types of whey, apart from the acidifying medium, is the high level of calcium phosphate in the acid whey. It is good practice to use the cations as the base for calculating the salt load in whey because the anions, e.g. citrates and phosphates, are involved in proteolytic reactions. This complicates the calculation of specific ion contents. The figures for cation content are typical of sweet and acid whey respectively and are shown in table 15.6.

Whey can consequently be characterised as a liquid with a high salt load which, as a consequence, results in short cycles when ion exchange is

Table 15.6
Cation contents of sweet and acid whey.

Ion	Sweet whey		Acid whey	
	%	meq/l	%	meq/l
Na	0.050	22.0	0.050	22.0
K	0.160	41.0	0.160	41.0
Ca	0.035	17.5	0.120	60.0
Mg	0.007	5.8	0.012	10.0
Total		86.3		133.0

applied. This in turn results in high costs for regeneration chemicals, if they are not recovered.

Conventional ion exchange for demineralisation

A simple demineralisation plant using ion exchange is shown in figure 15.11. The whey first enters the strong cation exchanger, loaded in H form, and continues to anion exchange in a weak base anion exchanger in its free base form. The ion exchange columns are rinsed and regenerated separately with dilute hydrochloric acid and sodium hydroxide (ammonia). Once a day the columns are disinfected with a small amount of active chlorine solution.

The following net reactions take place during demineralisation (NaCl is used to illustrate the salts of whey and R represents the insoluble resin exchange site).

Cation exchange: $R - H + Na^+ + Cl^- \longrightarrow R - Na + H^+ + Cl^-$

Anion exchange: $R - OH + H^+ + Cl^- \longrightarrow R - Cl + H_2O$

The various flows in the ion exchange process include the following steps:

- Exhaustion
  10 – 15 bed volumes of whey can be treated per regeneration. The bed volume is based on the bed volume of the cation exchanger.
- Regeneration
- Displacement of whey
- Backflushing
- Contact with regeneration solution
- Water rinse

The ion exchange columns are often made of rubber-lined mild steel to avoid corrosion problems. The conical shape is used specially for the anion exchanger to allow for swelling of the bed during transition from the free base form to the salt form.

Counter-current flow is often used for regeneration of the cation exchanger. Thus when whey is treated in downward flow, regeneration takes place in upward flow. This system reduces consumption of regeneration

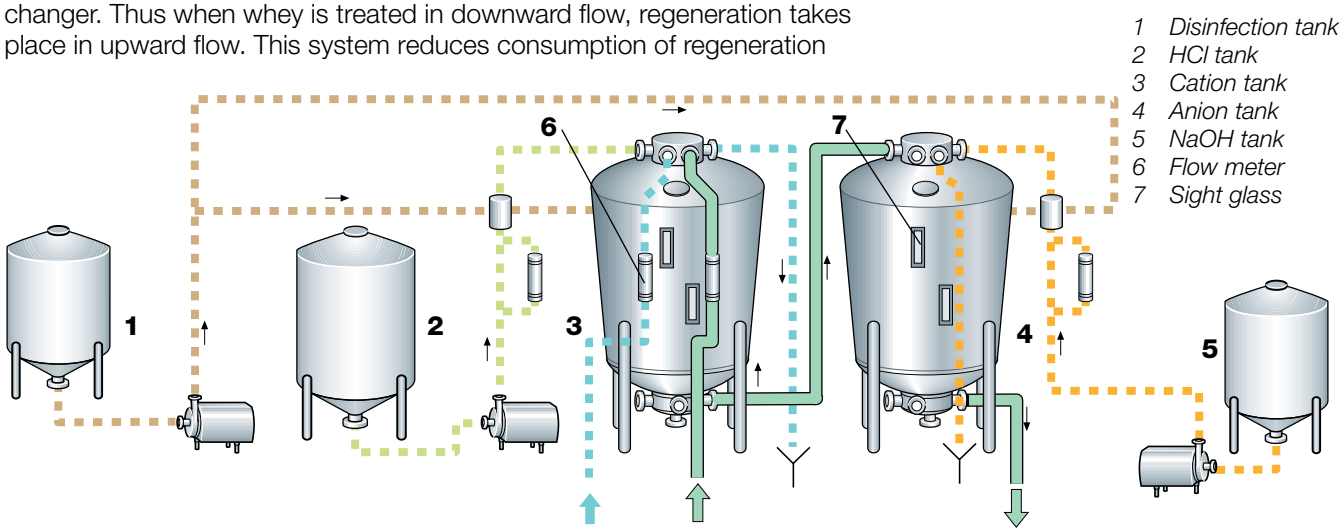


Fig. 15.11 *Plant for demineralisation of cheese whey by classical ion exchange.*
The dotted lines are used in the regenerative and sanitation phases.

chemicals by as much as 30—40%, but at the expense of a more complicated design. The plant can easily be automated. Two or three parallel ion exchange systems are needed for a continuous flow of whey. A normal cycle time is six hours, four of which are used for regeneration.

Process limitations

Whey is a liquid with a high salt load, which means short runs between regenerations. It also means a high consumption of regeneration chemicals and a high salt load in the waste from both ash removal and the required surplus of regeneration chemicals. Rinse-water consumption is also high, especially for washing out excess sodium hydroxide from the weak anion resin.

Losses of whey proteins occur on the columns due to denaturation/absorption. This is caused by great pH variation in the whey during the ion exchange process. Consumption of regeneration chemicals accounts for 60 – 70% of the operating costs of the process.

The process is primarily designed for 90% demineralisation, but any demineralisation rate can be chosen if a by-pass system is used.

An alternative ion exchange process

In order to reduce consumption of regeneration chemicals and thus also create a better waste situation for a demineralisation plant, the R & D department of the Swedish Dairies Association, SMR, has developed an alternative ion exchange process. In this process, the unit operations of which are illustrated in figure 15.12, the whey first enters the anion column containing a weak base resin regenerated in the bicarbonate form (HCO_3^-). During anion exchange the whey anions are exchanged for HCO_3^- ions. After this the whey enters the cation column, containing a weak acid cation exchange resin regenerated in the ammonium form (NH_4^+). During the passage of the whey through this column the whey cations are exchanged for NH_4 ions. Thus after the process the whey salts are exchanged for ammonium bicarbonate ($NH_4\ HCO_3$). The reactions can be summarised in the following formulae, where NaCl is used to represent the whey salts and R represents the insoluble resin exchange site.

Anion exchange : $R - HCO_3 + Na^+ + Cl^- \text{——} R - Cl + Na^+ + HCO_3^-$

Cation exchange: $R - NH_4 + Na^+ + HCO_3^- \text{——} R - Na + NH_4^+ + HCO_3^-$

$NH_4\ HCO_3$ is a thermolytic salt which decomposes to NH_3, CO_2 and H_2O when heated. It is then volatilised during the subsequent evaporation of the whey, offering the possibility of recovering the NH_3 and CO_2 stripped off the whey to make up a new regeneration solution ($NH_4\ HCO_3$). Part of the spent regeneration solution containing excess $NH_4\ HCO_3$ is collected for stripping in a distillation tower (about 100% excess $NH_4\ HCO_3$ is used).

Figure 15.13 shows the layout of a full-scale SMR process. The whey is first routed to the anion exchange column in HCO_3 form and then to the cation exchange column in NH_4 form. The ion exchange systems are paired, one working while the other is being regenerated. The cycle time is four hours.

After passing the ion exchange unit (1) the cooled whey is used for heat recovery in the absorption column and as cooling medium in the condenser (2) connected to the distillation tower (9). Then the whey enters the evaporator (3) and finally the demineralised whey concentrate is spray dried (10). The condensate from evaporator stage 2, which is especially rich in ammonia, is separated from the other condensate streams and continues to the absorption tower (4) where it forms the liquid base for the new regeneration solution. The condensates from evaporator stages 1 and 2 are used for cleaning the ion exchange resins. The ammonia is thus recovered to a great extent. Most of the carbon dioxide stripped off during evaporation is recovered in gaseous form in the exhaust gases from the mechanical vacuum pump of the evaporator. This gas flows directly into the bottom of the absorption column, where it is completely absorbed together with other inlet streams to form $NH_4\ HCO_3$. Recovery is not total, so the absorption tower is fitted with lines for injection of fresh 25% NH_3 solution and CO_2.

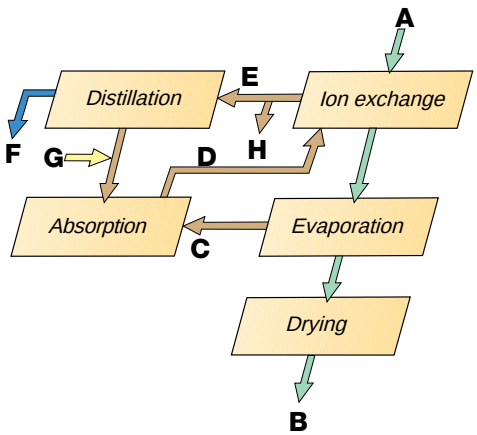


Fig. 15.12 *Unit operations of the SMR process.*

A Whey feed
B Demineralised whey powder
C Condensate with NH_3 and CO_2
D Amonium bicarbonate NH_4HCO_3
E Used regeneration solution
F Waste water
G CO_2 + NH_3 addition
H Magnesium ammonium phosphate

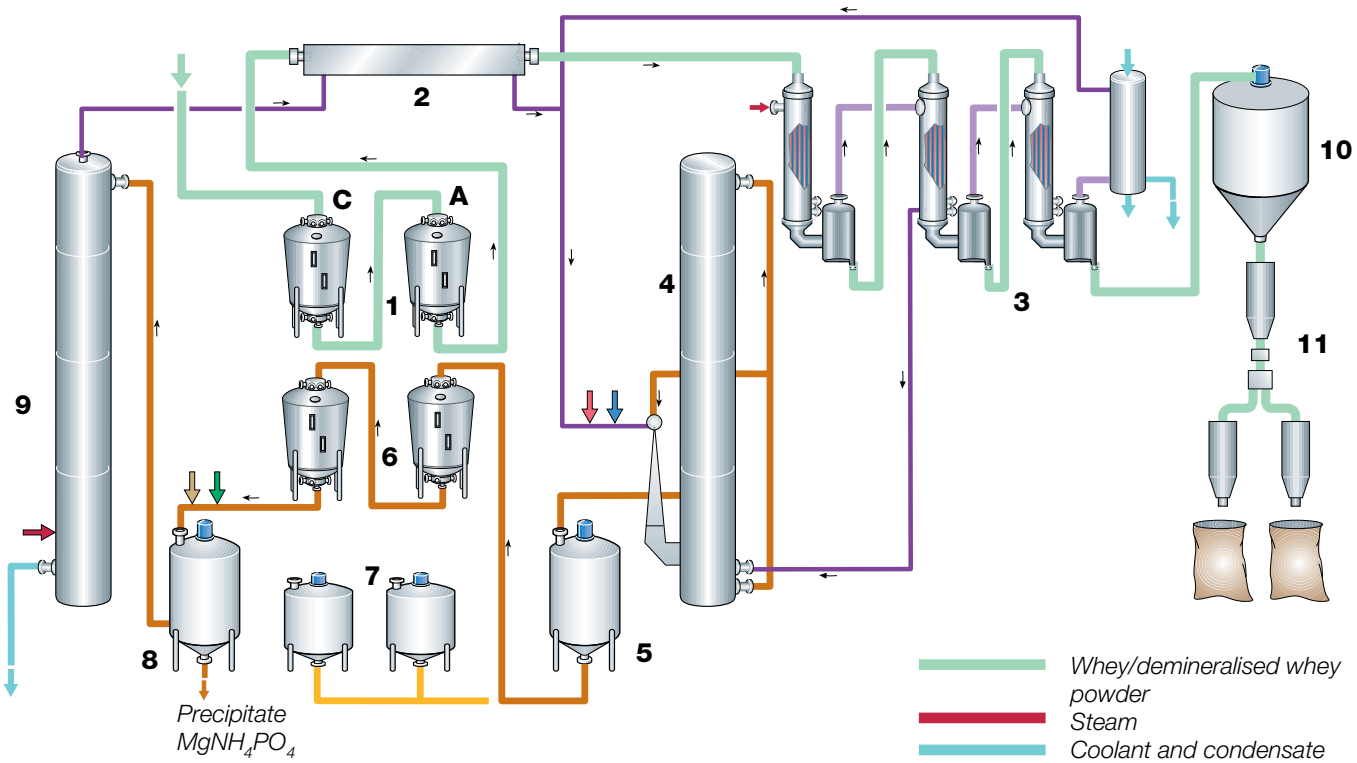


Fig 15.13 *Flow diagram of a full-scale production plant for demineralisation of whey powder.*

A Anion exchanger *C Cation exchanger*

1 Ion exchangers treating whey
2 Condenser
3 Evaporator
4 Absorption tower
5 Tank for new regeneration solution
6 Ion exchangers on regeneration
7 Tanks for NH_3 and HCl
8 Tank for used regeneration solution
9 Distillation tower
10 Spray dryer
11 Bagging

The part of the regeneration solution which is rich in NH_4HCO_3 is collected in a tank (8), where the phosphate is precipitated by addition of $MgCl_2$ after a minor pH adjustment with NaOH. When the precipitate of magnesium ammonium phosphate ($MgNH_4PO_4$) has settled, the supernatant liquid is pumped to the top of the distillation tower (9) and at the same time preheated in a plate heat exchanger (not shown) using the bottom liquid as the heating medium. About 10% of the liquid is stripped off as vapour, which in turn is condensed by the ion-exchange treated whey.

The SMR process has the following characteristics:

- Low running costs due to recovery of the regeneration chemicals;
- Low losses of whey solids and only half the salt discharge compared to a classical ion exchange process
- Small variations in pH during ion exchange (6.5 – 8.2), resulting in minimum damage to the whey proteins
- High demineralisation efficiency, over 90%
- Low operating temperature (5 – 6°C), enhancing the bacteriological status of the end product
- High yield of whey solids compared to classical ion exchange and electrodialysis
- Optimum heat recovery

Process limitations and costs

In most cases, depending on the costs of chemicals, the operating costs of the SMR process are 30 – 70% lower than those of the classical ion exchange process. Like all systems of demineralisation, electrodialysis and traditional ion exchange, this process is sensitive to high Ca contents in the feed stream, so it is advisable to use pH adjustment and heating as pretreatment stages before demineralisation. With this technique 80% of the calcium phosphate in the acid whey can easily be precipitated and refined for use in animal fodder or even for human consumption.

The equipment for the process includes more components than the classical ion exchange process. The capital costs are therefore higher, but

these must be weighed against the benefits of low operating costs and improved plant environment.

Lactose conversion

Lactose hydrolysis

Lactose is a disaccharide consisting of the monosaccharides glucose and galactose, see figure 15.14. Lactose exists in two isomeric forms, α-lactose and β-lactose. They differ in the spatial arrangement of the hydroxyl group at the C atom in the glucose molecule, and thereby also, amongst other things in:

- Solubility
- Crystal shape
- Melting point
- Physiological effect

Lactose can be split hydrolytically, i.e. by bonding of water, or by an enzyme. The lactose-splitting enzyme β-galactosidase belongs to the hydrolase group. Figure 15.14 shows enzymatic splitting of lactose into galactose and glucose.

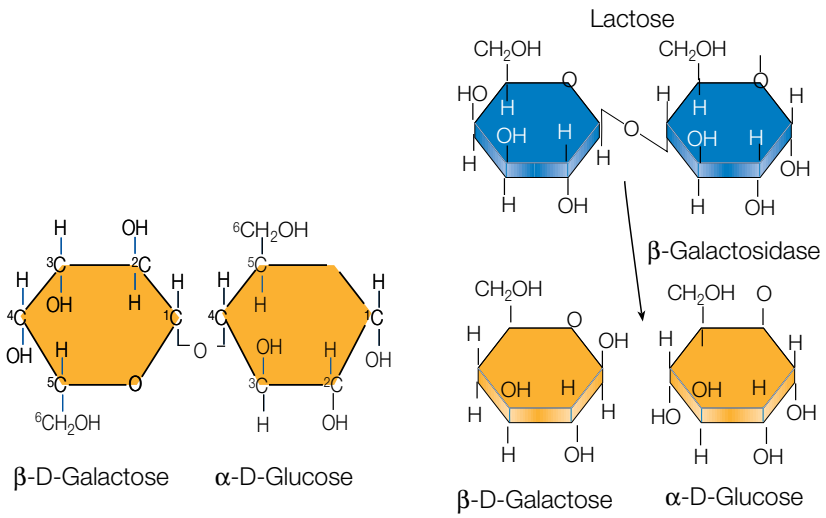


Fig. 15.14 *Chemical structure of lactose and lactose splitting.*

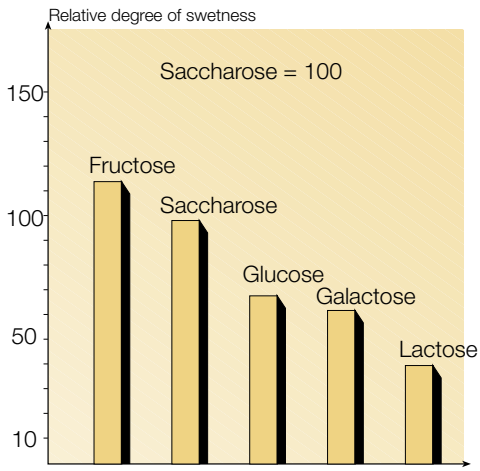


Fig. 15.15 *Degree of sweetness of different types of sugar.*

Lactose is not nearly as sweet as other types of sugar. This is shown in figure 15.15, which indicates the relative degree of sweetness of different types of sugar. Hydrolysis of lactose consequently results in considerably sweeter products.

Some people lack the enzyme that decomposes lactose and therefore cannot drink or eat any significant quantities of milk products. This is called lactose intolerance. Hydrolysis of the lactose in the milk products allows these people to utilise the high-quality proteins, vitamins, etc. in milk products.

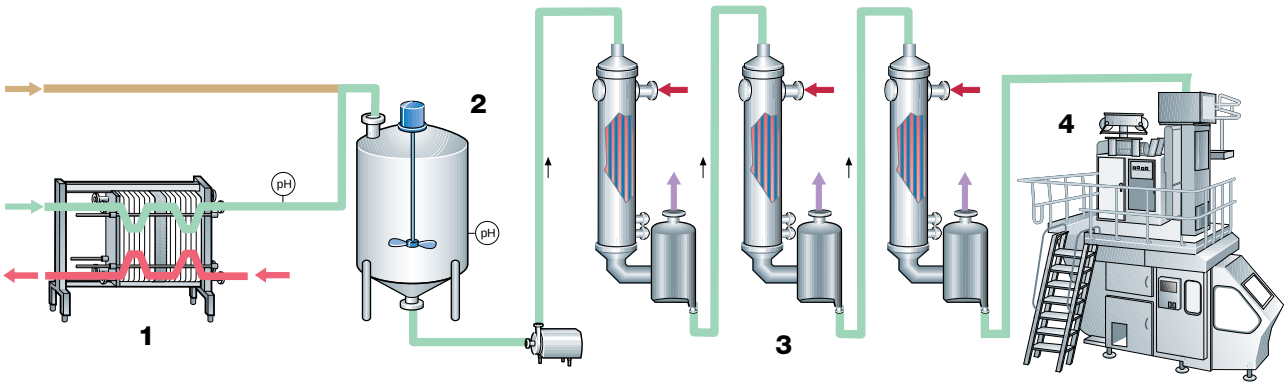
Some defects, such as sandy texture in ice-cream (crystallisation of lactose) are practically eliminated by lactose hydrolysis.

Enzymatic hydrolysis

Figure15.16 shows a process for enzymatic hydrolysis of lactose in whey.

Pretreatment in the form of demineralisation is not essential, but it improves the taste of the final product. After hydrolysis the whey is evaporated. A syrup with a dry solids content of 70 – 75% is then obtained. 85% of the lactose in this syrup is hydrolysed and can be used as a sweetener in the baking industries and in the manufacture of ice-cream.

During production the enzyme is inactivated by heat treatment or by pH



Whey
Lactase
Heating media
Steam
Vapour

1 Pasteuriser
2 Tank for hydrolysis
3 Evaporator
4 Filling

Fig. 15.16 *Plant for enzymatic hydrolysis of lactose in whey.*

adjustment. It cannot be used again. Instead of using free enzymes, it is now possible to bind the enzyme to different types of water-soluble and non-water-soluble carriers. Such systems with immobilised enzymes can be used for continuous lactose hydrolysis. The enzyme, which is expensive, is not consumed and can be used to hydrolyse large amounts of product. This increases the profitability. The technique has not yet been developed to any great extent.

Acid hydrolysis

Lactose can also be split by means of acids in conjunction with heat treatment or by passing a cation exchanger in hydrogen form at high temperature, around 100°C. The required degree of hydrolysis is determined by selection of pH, temperature and holding time. As brown discoloration occurs during hydrolysis of the whey, active carbon treatment is recommended.

Chemical reaction

It has been established that non-protein nitrogen products can be used as partial replacement for natural protein in ruminants because certain microbes in the cattle rumen can synthesize protein from urea and ammonia. However, in order to get a balanced feed of nitrogen and energy, urea and ammonia have to be transformed into more suitable forms, which slowely release nitrogen to the rumen for improved protein synthesis.

Lactosyl urea and ammonium lactate are two such products based on whey.

Lactosyl urea

Briefly, the procedure for production is as follows: after separation the whey is concentrated up to 75% DM, typically in two steps. After addition of urea and edible sulphuric acid the whey concentrate is held at 70°C for 20 hours in a jacketed tank provided with agitator. Under these conditions the urea reacts with the lactose to form lactosyl urea.

Following the reaction period the product is cooled and transported to a factory producing concentrated feed (pellets for instance) or direct to farmers.

Ammonium lactate

The process technique involves fermentation of the lactose in whey into lactic acid and maintaining the pH with ammonia, resulting in formation of ammonium lactate. After concentration to 61.5% DM the product is ready for use.