ABSTRACT

Procedures for manufacture of whey protein products are based on known behavior of whey components under defined conditions. Properties that have been exploited commercially include: molecular size differences (ultrafiltration, gel filtration), insolubility of protein at high temperature, charge characteristics (demineralization, protein removal by ion exchange), aggregation by polyphosphates, and crystallization of lactose. Numerous other isolation procedures have been investigated. Chemical, physical, and functional characteristics vary according to method of manufacture. Capital costs for most of these processes are high. As yields are characteristically low, careful economic analysis is necessary. Mass and energy balances must be prepared accurately as these provide the bases for such calculations.

For the customer, consistency of quality is paramount. Hence, the manufacturer must have an understanding of his raw material and must be able to manipulate its properties or his process to achieve such consistency. Attention to hygiene, staff training, quality assurance, and provision of technical resources to cope with the more demanding processes are necessary.

The principal processes for manufacture of whey protein concentrates are reviewed, together with general characteristics of the resultant products. Criteria for commercial evaluation of new processes are discussed.

INTRODUCTION

Production of whey protein products is well established in the dairy industries of many countries. The expanding market prospects of the base milk products that generate whey (cheese and casein) over the last 10 yr, combined with escalating effluent treatment costs, have increased the need to process whey into useful products (47, 61).

The development of new processes for the recovery of protein from whey during the last 10 yr has resulted in substantial increase of the range of whey protein products available. Prior to 1971, insoluble lactalbumin, the product that results from heat-induced precipitation of whey protein, was produced commercially in several countries. Total manufacture was small, however, because of its limited applications in formulations of food products. The lactose industry also produced an early type of whey protein concentrate in the form of mother liquor, which has been used for many years as a stock food.

Major developments in several solids-liquid separation technologies have been utilized by the dairy industry to produce whey protein products with good functional, nutritional, and flavor properties. A number of comprehensive reviews of both traditional and modern methods of whey protein product manufacture have been prepared (4, 17, 35, 48, 61, 79, 89, 104). Despite these developments, the food industry was initially reluctant to introduce these ingredients into new formulations. The few commercially successful examples of whey protein developments by the mid-1970’s appear to have resulted from manufacturer-end user cooperation, where the end user had a specific application in mind. Hence, the introduction of such products was more a case of technology “push” rather than “pull”, whereas the latter is usually the stronger commercial force (105).

The behavior of whey proteins in real food systems proved to be highly complex in many instances, and several whey products were inconsistent in performance. These observations plus technical difficulties with the new technologies all hindered their acceptance by the food industry. However, major progress in all
these problem areas has been achieved in the last 5 yr.

From developments of the last decade, several conclusions can be made. There are several important marketing and process design criteria that should be used to assess the potential success of any new protein recovery process. These will be addressed, following a brief review of the types of processes and products now available.

**WHEY PROTEIN RECOVERY PROCESSES**

The principal processing methods in the manufacture of whey protein products may be classified by the physicochemical properties exploited to effect protein separation. The commercial processes now known to be (or to have been) in operation are based on the following:

**Heat Precipitation**

Whey proteins may be precipitated (and thereby rendered insoluble in water) by heating whey at acid or near-neutral pH. The critical variables that affect these processes have been described (61, 90, 94). Acid whey must be heated to at least 90°C and maintained at such temperatures for at least 10 min to achieve maximum yields. For sweet wheys, good yields can be obtained by heating at pH between 6.0 and 6.5, although products so derived have higher mineral concentrations than those of acid whey unless pH adjustment to 4.6 is effected prior to protein removal (21, 22, 71, 75, 96). The precipitate so formed is firmer and more readily separated than that formed in unacidified whey.

Processes for recovery of lactalbumin have been described (61, 90). Whey is heated and held (in batch or continuous mode), precipitated protein is removed by settling (static or accelerated), and the precipitate then is washed, reseparated, and dried. The basic process flows are outlined in Figure 1. In modern plants, high speed centrifuges such as clarifiers and decanters are used to effect primary and secondary (after washing) separations. Ring, fluid bed, roller, and spray dryers have been used to obtain the finished product. Typical yields are 4.2 to 5.2 kg/m³.

Process refinements investigated have included demineralization prior to heating (49, 81, 88), preconcentration by reverse osmosis and ultrafiltration (10, 36, 72), and continuous, high temperature reaction (120°C for 8 min at pH 6) (85). Most processes result in an insoluble product, but through heating whey to 95°C at pH 2.5 to 3.5, then adjusting to pH 4.5 prior to separation, it has been claimed that a product soluble at pH 5 can be produced (73, 74). The use of additives such as salts (NaCl, CaCl₂) and cysteine to improve product yields has been investigated (102).

**Molecular Size Differences**

*Ultrafiltration.* By 1981, ultrafiltration (UF) had become the most widely used process for recovery of soluble whey protein concentrate (WPC). It was estimated then that up to 8% of the world's whey was processed by UF (67). The process has been described extensively (20, 28, 56, 61, 97, 100). The development of robust, synthetic, cleanable membranes, and the refinement of continuous operation using multi-stage, recycle loops, and diafiltration have been significant factors contributing to the success of this process. Membrane configurations designed to

![Figure 1. Process schematic for production of heat precipitated whey protein (lactalbumin) showing alternative methods of dewatering and drying (61).](image-url)
Figure 2. Process schematic for production of whey protein concentrates by continuous stages-in-series ultrafiltration (61).

Gel Filtration (GF). This useful laboratory technique for separation of solution components also has been used commercially for recovery of WPC (18, 25, 54, 55). The hydrated gel acts as a molecular sieve in that small molecular weight components are able to enter the solvent phase within the gel beads. Protein molecules remain in the solvent phase surrounding the beads. High and low molecular weight fractions then can be recovered. Products of 30 to 80% protein can be manufactured. The process is expensive to install and operate, and the yield, at 65% of the protein in whey, is low. It also is subject to fouling and microbial contamination. It appears that it is no longer in commercial operation.

Precipitation by Complexing Agents

Numerous complexing agents have been used experimentally to recover protein from whey; of these, polyphosphates appear to be the only group to be used commercially for this purpose (61). Long-chain polyphosphates will precipitate protein from whey at low pH e.g., 2.5. Typically, potassium polymetaphosphate and sodium hextametaphosphate are used. The precipitate so formed is removed by centrifugation, washed, and then subjected to pH alteration and calcium addition to remove the phosphate. Cation (particularly calcium) removal prior to phosphate addition reduces the amount of phosphate required and results in recovery of up to 90% of the original whey protein (33). Further refinements also have been described (70).

To achieve acceptable powder densities (.35 to .5 g/cm³), it is normally necessary to concentrate the retentate prior to spray drying. Low-temperature processing is necessary because of the heat sensitivity of the product, but suitable equipment is readily available. The spray drying process for this product is conventional. Product yield varies with protein content but ranges from 15 to 6 kg/m³ over the range 30 to 80% protein, dry. Between 90 and 95% of the protein in the whey is recovered.

Several important factors in successful operation of UF plants have been identified (65). These have included whey quality, whey management, water quality, process integration (with upstream and downstream operations), and quality assurance.
Removal of Lactose and Minerals

Composition of whey can be modified by removal of lactose and minerals to give whey protein products of 15 to 40% protein, dry. For many years, the lactose industry has produced a protein concentrate in the form of mother liquor, the material that remains after lactose has been crystallized and separated from concentrated whey. The product, known as delactosed whey powder, contains about 25% protein. It has been used as a stock food because of the extensive protein denaturation in the process (61). In more modern plants, with lower temperature-shorter residence time evaporators, the product is more functional and, therefore, of greater value.

Development of demineralization methods in the last 10 yr has led to introduction of demineralized and delactosed and demineralized whey powders. Electrodialysis (ED) and ion exchange (IE) (or combinations of both) have been used. The ED process is the more capital intensive, but it now has been confirmed by several workers that for up to 70% ion removal from cheese whey, it is the more economical choice in terms of total costs (operating plus capital servicing) (61, 62). If 90% mineral removal is required, then ion exchange or a combination of ED plus IE will be necessary. Both processes have been described (ED: 19, 39, 61, 93; IE: 19, 39, 61).

Delactosed whey powder has a high mineral concentration (up to 25%). Processes have been described whereby preconcentrated whey (up to 30% TS (total solids)) is subjected to ED, the whey is concentrated to 60% TS, lactose is crystallized and removed, and the remaining liquid is concentrated and spray dried (30, 61). The resultant product may contain up to 35% protein. Lactose removal prior to ED also has been described (61). Such mineral removal enhances the value of the protein product.

Adsorption Properties

Adsorption techniques, based on the ion exchange properties of whey protein, are currently under serious investigation. Because whey proteins are amphoteric, solid phase charged adsorption media can be used to remove them from solution under appropriate conditions. Media suitable for this purpose have included regenerated cellulose (43, 84, 86), titania plus alumina (101), and silica (69). Of these, cellulose- and silica-based systems have progressed to semi-commercial operation.

Regenerated cellulose is used in the “Vistec” (BioIsolates) process. The development of sulfoethyl cellulose resins of high charge density (1.1 meq/g) has enhanced its commercial prospects (J. Ayers, personal communication). Whey is first decationized, then mixed with resin in a stirred tank reactor. After separation of the protein-resin complex from the deproteinated whey, the protein is desorbed at pH 9. Ultrafiltration is used to concentrate (and demineralize) the protein solution, which then is spray dried. Protein yield is 85%, and the dried WPC may contain as much as 95% protein. Because lipid molecules are not adsorbed by the media, such products have low fat concentrations.

Silica-based adsorbents are used in the “Spherosil” process. For acid whey, an adsorbent with strong cation exchange properties is used. For sweet whey, an anion exchanger is necessary, and as immunoglobulins are not adsorbed at this higher pH, a second (weak cation) exchanger must be used. Eluants used are ammonium hydroxide (from cation exchangers) and hydrochloric acid (from anion exchangers). As with the Vistec process, ultrafiltration and spray drying are then necessary. Protein yield is 90%.

Incorporation in Other Products

Cheeses. In the last 10 yr, there has been considerable interest in increasing yields of several traditional cheeses by incorporation of whey protein (1, 9, 11, 46, 83). Heat-precipitated protein may be added to cheese milk, as in the “Centriwhey” process (7), to increase cheese yields by 10 to 14%. Over 90% of the original whey protein may be recovered. Ultrafiltration now is used extensively to produce a liquid “precheese” from either whole or skim milk, particularly for soft cheese manufacture (41, 106). Yield increases of up to 30% have been claimed. Incorporation of whey protein into cheeses can cause abnormal textures.

1J. Ayers, Department of Biochemistry, Massey University, New Zealand, June 1, 1983.
and flavors, however, and adjustment of the traditional processes is often necessary to overcome this.

**Whey and Yeast.** Yeast is produced by fermentation of lactose in some countries using whey as the primary feedstock (61). Nutritional value and yield of the dried yeast are increased if whey proteins in the growth medium are recovered also as part of the finished product. Methods reported have included partial fermentation of whey followed by spray drying of fermented liquid (61), heat precipitation of protein prior to or after fermentation (27), and ultrafiltration of the fermented liquid to effect separation of protein plus yeast from permeable solutes (77).

**Coprecipitates.** Heating skim milk sufficiently to denature whey proteins causes interactions of casein with whey protein. This well-established phenomenon is the basis for manufacture of coprecipitated casein and whey protein products. These are obtained when heated skim milk is acidified or treated with calcium salts and the resultant curd is washed and dried (87, 98). Up to 75% of the original whey proteins can be recovered in this manner.

**Whey Blends.** Numerous references have been made in the product development literature to “whey blends,” which are blended mixtures of whey products (whey powder, delactosed whey powder, WPC, etc.) with other ingredients such as caseinates, vegetable protein isolates, etc. (5, 6). Properties of such blends vary widely. They are often made for specific product applications and have become a significant opportunity for whey processors. Manufacturing appears to be mostly by dry blending, although some of the literature makes reference to wet blending and treatments such as heating before spray drying to achieve desired functional characteristics.

**Texturized Whey Protein**

Some work has been reported on production of texturized products from whey proteins. Dispersions of whey protein are pumped under pressure through an orifice and coagulated in acidic solutions, such as hydrochloric acid with soluble calcium (52) and acetic acid with sodium chloride (40). Whey protein does not appear to be effective for this purpose in comparison with other proteins.

**Noncommercial Procedures**

Numerous other isolation procedures have been investigated, although it is not known whether any of these has been practiced commercially. Methods reported to date have included: precipitation by carboxymethyl cellulose (109, 110), ferric salts (3), alcohol (76), tannin (44), polyacrylic acid (37, 99), sodium lauryl sulfate (14), bentonite (29), bentonite and lignosulfonate (12), chitosan (8), and tannery waste (103); coprecipitation with plant proteins (80, 92); ion exchange (50); foam concentration (15, 108); dialysis (16); ultra-centrifugation (26); electrogelation (45); glycerol extraction (82); use of calcium oxide (13); enzymes (59); and chromatographic techniques (51).

Principal commercial methods for manufacture of whey protein products are in Figure 3.

**WHEY PROTEIN PRODUCTS**

Compositions of wheys and whey protein products have been described extensively (47, 65, 91). Although no official standards of identity have been established in the US, the United States Department of Agriculture (USDA) does recognize the following product descriptions: whey powders; partially delactosed whey powder; partially demineralized whey powder (not more than 7% ash); partially delactosed, partially demineralized whey powder; demineralized whey powder (less than...
1.5% ash), and whey protein concentrate (which must contain more than 25% protein, dry basis).

Yields and protein concentrations of these products, as made by the available commercial procedures, are summarized in Table 1.

**ELEMENTS OF PROCESS DESIGN**

From literature descriptions of principal recovery processes, they share many common steps. A schematic outline of a general protein extraction process is depicted in Figure 4. Regardless of the protein separation process, much of the equipment required is common to all processes. If food grade products are to be manufactured, hygienic design of building and plant will be required. Tanks, pumps, valves, instruments, and many other capital-related items will be needed in all options. This has significant cost implications, as a recovery step that, on its own, appears simple and inexpensive, will simply become part of a larger, integrated operation. Hence, emphasis must be placed on the entire whey process, from the point of whey separation right through to packaged, finished product.

In addition, whey plants often are established either in or adjacent to existing milk plants where the primary products have been cheese or casein. Such plants may not have been built with whey processing in mind. The substantial service requirements, in terms of utilities and staff resources, that a whey plant may require may be a major burden to an existing plant. In some instances (2, 107), establishment of centralized whey processing facilities on greenfield sites has overcome this problem.

Some of the important elements in the design of, and the planning for, whey protein recovery processes are detailed here.

**Characteristics of the Raw Material.** Extensive information is available about the compositions of wheys (24, 47, 63, 91), but it is better to establish actual characteristics of the whey generated in the plant where a protein plant is planned. As yield is so critical to success of such operations, a relatively small error in the estimate of “true” protein [(total-nonprotein) × 6.38] can have serious economic consequences. Also, the microbiological quality of the whey should be known, as should its physical condition (e.g., presence of insoluble
Figure 4. Process schematic of a generalized whey protein recovery operation.

matter and fat), to determine the types of treatment required.

Types of Finished Products to be Manufactured. This selection must be based on the types of protein products that the food industry is prepared to buy. A possible conclusion from the experience of the last decade is that dairy technologists must be prepared to communicate with food product development groups so that they can learn of potential opportunities. They then should be prepared to work with the customer to develop specific, end-use applications. The decision also is likely to be based, however, on dairy industry policies prevailing in the country where the plant is located. Effluent disposal costs and geographic location also may be contributing factors. Where effluent disposal costs are high, whey may have a high negative cost. Manufacture of low-cost, nonfunctional WPC products of 25 to 35% protein content then may be logical, although presumably this can be at the expense of commodity products such as nonfat dry milk (NFDM). Once the product range has been identified, finished product specifications can be prepared.

Capacity. For year-round dairy industries, the capacity of a whey protein plant often will equal the maximum quantity of whey available in that factory or, if whey is to be transported, in a larger area. For a seasonal industry, matching capacity to peak whey availability has not been economic. In New Zealand, provided the excess whey could be disposed of, a plant of capacity equal to 80% of peak seasonal supply was the most economic choice (95).

Mass and Energy Balances. These are among the most critical aspects of a feasibility study. Information so generated will provide much of the basis for calculations of operating cost. To do an accurate mass balance, it is necessary to know raw material compositions, mass partitioning of whey components during the process, finished product specification, and quantities and compositions of each unpreventable loss. Flow rates of steam and water also must be quantified. This is done best by studying the engineering flow diagram and using known performance information on the principal process items. Theoretical yields, supplied by some equipment suppliers, should be checked thoroughly as these often will exceed what can be achieved in practice. For whey, another important factor is partitioning of nonprotein nitrogen NPN compounds and "true" protein (precipitated in 12% trichloroacetic acid). In several processes, NPN is not recoverable. The "protein" content of whey often is quoted as .75 to .90%, but the true protein content is usually in the range .5 to .65% for the purpose of most recovery processes. A small detail of a larger mass balance is shown in Figure 5 to illustrate a concise method of expression. For energy balance, steam and power consumptions must be known.

Provided the engineering flow diagram has been constructed accurately, and by working from first principles, it is possible to construct accurate predictions of product yield (kilograms finished product per cubic meter of whey processed) and consumptions of utilities (metric tons steam, kilowatt hours, and cubic meter for steam, power, and water, respectively, expressed per ton of finished product).

Drawings. The scope of the project will be defined, in increasing order of complexity, by the block diagram, process schematic, process
Figure 5. Method of expressing mass balance of whey components over successive steps in a whey protein recovery process.
TABLE 2. Some strategies relating to production of high value (functional) and lower value (less functional) whey protein products.

<table>
<thead>
<tr>
<th>High value</th>
<th>Lower value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Detailed knowledge of whey</td>
<td>Effluent disposal often a factor</td>
</tr>
<tr>
<td>Products of uniform quality</td>
<td>May want deproteinated lactose stream</td>
</tr>
<tr>
<td>Good process security</td>
<td>Use existing plant</td>
</tr>
<tr>
<td>Specialist plant, often new</td>
<td>Less demanding specifications</td>
</tr>
<tr>
<td>Maximum yield</td>
<td>Less company support</td>
</tr>
<tr>
<td>Achievement of specifications</td>
<td></td>
</tr>
<tr>
<td>Strong technical support</td>
<td></td>
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<tr>
<td>- QA, research and development,</td>
<td></td>
</tr>
<tr>
<td>engineering</td>
<td></td>
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<tr>
<td>Good staff training</td>
<td></td>
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<td>High management skill</td>
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Completion of Flow Diagrams. If the engineering diagram can be completed as part of the feasibility study (unfortunately, this often is not the case), the precision of capital cost estimation can be improved considerably. Preparation of equipment schedules, with each item sized and priced, is then possible. Because whey protein processes are often unique to their particular factories, the use of costing factors can be misleading. The flow diagram often will show when existing facilities, such as steam supply lines and CIP vessels, are inadequate to meet demands of the new process.

Identification of Process Control Strategies. Process control and instrumentation is costly but can be cost effective if selected properly.

Accurate Estimates of Installation Costs. Accurate cost estimates are difficult, and skill is obtained largely by experience.

Involvement of the Right People. Production, engineering, and accounting personnel all should be consulted, particularly for inputs on how the new plant will affect existing operations.

Accurate Estimates of Project Costs. Fees for consultants and architects, plus internal engineering and administration costs must be identified and charged to the project.

Escalations and Contingencies. Current inflation rates must be considered. The standard allowance for contingencies is 10% on the sum of all other costs.

Operating Cost

The principal components of both capital and operating costs are identified in Figure 6. A significant aspect of the cost of many whey protein products, particularly those produced by more capital-intensive technologies, is the high ratio of standing or fixed costs to total costs. Hence, the achievement of high product yields and good performance against specification assume considerable commercial significance. A further important component is the cost of whey itself. In some countries, this has a negative value because of the high costs of effluent disposal. In some US cities, this may exceed 30 ¢/kg BOD (biologic oxygen demand) (W. J. Harper, personal communication). In other countries, whey may have a positive cost. Overall, estimates of capital and operating costs are vitally important to the success of the process. Such work needs careful research, not just in terms of the new protein recovery technology but in terms of the way it will affect other company operations.

Figure 6. Principal components of capital and operating costs of whey protein recovery processes.

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2W. J. Harper, New Zealand Dairy Research Institute, Private Bag, Palmerton North, New Zealand, April 4, 1983.

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Planning for Production

Before the plant is commissioned, staff training in production, laboratory, and maintenance departments should be conducted. This can prove highly beneficial, leading to shortened commissioning times.

CONCLUSIONS

Technologies for the recovery of protein from whey are now established. Yields are normally low, particularly for highly purified products. The total capital cost is high for most of these processes, regardless of the cost of the protein recovery step itself, because of costs of ancillary plant. Hence, in selecting a process, emphasis must be on a plant that conforms to regulatory requirements, is reliable to operate, and gives product consistent with the marketing and production strategies of the company. Close liaison with customers helps in the identification of significant product quality attributes. It also can lead to identification of key process control variables that must be controlled to give consistent product.

REFERENCES

30 Hargrove, R. E., F. E. McDonough, D. E. La Croix, and J. A. Alford. 1976. Production and
38 Holericka Int. B. V. 1980. Procedure for the processing of milk, whey, or ultrafiltration permeate of milk or whey to make milk products such as dried whey, lactose, and protein concentrate. Dutch Pat. Appl. 7,810,035.


110 Zadow, J. G., and R. D. Hill. 1978. The formation of complexes between whey proteins and carbo-

xymethyl cellulose modified with constituents of increased hydrophobicity. J. Dairy Res. 45:85.