

Protein Concentrate from Cheese Whey by Ultrafiltration

F. E. McDONOUGH, W. A. MATTINGLY, and J. H. VESTAL

Dairy Products Laboratory, Eastern Marketing and Nutrition Research Division, ARS, USDA
Washington, D.C. 20250

Abstract

A concentrate containing up to 65% protein in the solids was recovered from cheese whey by the use of tubular cellulose acetate ultrafiltration membranes designed to reject solutes larger than 20,000 molecular weight. Removal of 90% of the water as permeate resulted in a concentrate containing 20% solids with 35 to 37% protein and 50 to 52% lactose in the solids. By diluting and recycling, a concentrate containing 60 to 65% protein and 30 to 35% lactose in the solids was obtained. For 90% reduction in volume, fractionation averaged 12 gallons per square foot of membrane surface per day.

and smaller solutes pass through the membrane and are collected as permeate; larger solutes are retained by the membrane and recovered as concentrate. Thus, reverse osmosis can be applied to the separation of low molecular weight solutes whereas ultrafiltration is applied to the separation of relatively high molecular weight solutes. By proper membrane selection, it is possible to concentrate or fractionate or both.

Concentration of cheese whey has been reported (2, 3, 4, 5, 6, 7, 8, 12), and fractionation studies are being emphasized. Removal of most of the salts, lactose, and water by ultrafiltration would leave an undenatured, high-protein concentrate with unique properties and potential as a premium priced food ingredient. This paper reports on the fractionation of whey into high and low protein portions.

Introduction

Anti-pollution legislation is forcing cheese manufacturers to turn to utilization of whey rather than disposal, but the bulk and perishability of whey makes processing a marginal venture. The price of dried whey for animal feed barely covers operating costs so the most desirable use, from both an economic and nutritional standpoint, is as a human food ingredient. The value of whey may be further enhanced by separating it into its major components of protein, lactose, and salts. The nutritional superiority of whey proteins to many other proteins has been well established (1, 9, 11, 13). These proteins have been commercially available in heat-denatured form for a number of years. Unfortunately, the denatured product is gritty and insoluble and therefore, has only limited use in foods.

During recent years, reverse osmosis and ultrafiltration have been receiving much attention. Ultrafiltration, like reverse osmosis, is a hydraulic pressure activated process, but it separates solution components largely on the basis of molecular size and shape. Unlike reverse osmosis, ultrafiltration does not require high operating pressures to overcome high osmotic pressure, mainly because of the retention capability of the membrane. Both solvent

Experimental Procedures

Whey. Cheddar cheese whey was obtained from the Dairy Products Laboratory pilot plant at the Agriculture Research Center at Beltsville, Maryland. Cottage cheese whey was obtained from a local manufacturer. The whey was pasteurized at 72.8 C for 15 sec, centrifugally clarified, and held at 1.7 to 4.4 C for no longer than three days prior to use.

Equipment. A Model II "Osmostat Separator" developed by Calgon-Havens was used for these tests. The unit consisted of a mobile frame upon which were mounted four tubular modules connected to a belt-driven variable speed positive displacement pump (Moyno Model 9P3SSQ). Pressure was regulated by a spring-loaded valve. Each module contained 18 membrane-lined pressure tubes connected with U-fittings. The tubes, 244 cm long by 1.27 cm diameter, were made of porous fiberglass to provide support for the membrane. The four-module, 72-tube system represented 175 m of continuous flow and a total of 6.2 m² of active membrane (864 cm² per tube). The modules, designated Type 215, utilized cellulose acetate membranes with typical retentions as shown in Table 1.

Analytical. Samples of the original whey, the protein concentrate, and the permeate were analyzed for pH, titratable acidity, total solids

TABLE 1. Typical performance of Calgon-Havens 215 Membranes.^a

Feed	Retention (%)
Polyethylene glycol ^b	90
Sucrose	5
Lactose	6
Sodium chloride	0

^a Pressure, 14.05 kg/cm²; feed rate 7.5 liters per minute.

^b Molecular weight, 20,000.

by the Mojonnier method, ash by combustion at 550 C, lactose by reaction with Dreywood's Anthrone reagent (10), total nitrogen by the Kjeldahl procedure, and nonprotein nitrogen by precipitation with trichloroacetic acid (14).

Procedure. Several preliminary trials were made on new modules to "set" the membrane, to determine operating characteristics, and to gather control data which could be used to detect changes in permeability or rejection. For most tests, 568 to 757 liter-lots of whey were adjusted to 26.7 C in a hollow-jacket stainless steel vat and maintained at that temperature by circulating water through the jacket. A batch procedure was utilized, whereby the whey was recirculated through the system and returned to the holding tank while the permeate was collected in a separate container. Feed rate was 7.5 to 9.5 liters/min and pressure averaged 14 kg/cm² (200 psi). Recirculation was continued until up to 90% of the original volume of whey was removed as permeate. Flux rates (rate of permeation through the membrane) were recorded and samples of concentrate and permeate were collected for analysis at frequent intervals. In several trials, addi-

tional nonprotein solids were removed from the concentrate by diluting it with nine parts of tap water and recycling until a second 90% volume reduction was complete.

Results and Discussion

Retention of Solids. Tables 2 and 3 show typical analyses of fractions from Cottage cheese whey with the Type 215 membrane. Results with Cheddar cheese whey were essentially identical to those reported here for Cottage cheese whey. Table 2 compares percentage composition of whey with concentrate and permeate obtained at two states of processing. Concentrate and permeate "A" are samples taken after an amount equal to 50% of the original volume of whey had been removed as permeate, while concentrate and permeate "B" are samples taken after 90% volume reduction.

The data are more meaningful when converted to dry weights to determine mass balance as shown in Table 3. Using lactose as an example, processing to a 90% volume reduction resulted in a concentrate containing approximately 22% of the original lactose, and a permeate containing 78%. When expressed as retention value, i.e., relationship of lactose removed, to the volume of permeate removed the membrane exhibited a lactose retention of approximately 13%. Similarly, protein retention was 97 to 98% while lactic acid and ash retentions were about 4 to 5%. These retentions, slightly higher than expected from the data in Table 1, are likely the result of a gel-like protein deposit as reported by Lim et al. (5).

It is important to understand the relationship between retention and actual loss with the permeate. A claim that a membrane will pass 100% lactose (0% retention) means literally

TABLE 2. Analyses of ultrafiltration fractions of whey.^a

Sample	Weight (kg)	Total solids	Lactose	Titratable acidity (%)	Ash	Nitrogen	
						Total	Non-protein
Cottage whey	617	7.26	4.7	.52	.612	.145	.051
Concentrate A ^b	308	8.88	5.5	.55	.657	.238	.058
Permeate A	308	5.75	3.8	.49	.570	.047	.040
Concentrate B ^c	51	22.36	11.5	.69	.970	1.320	.188
Permeate B	566	6.00	4.0	.49	.582	.040	.038

^a Havens International Type 215 Membrane.

^b After 50% volume reduction.

^c After 90% volume reduction.

TABLE 3. Dry weights of ultrafiltration fractions of whey.^a

	Original whey	Concentrate A	Permeate A	Quantity in permeate (%)	Retention ^b (%)	Concentrate B	Permeate B	Quantity in permeate (%)	Retention ^b (%)
	(kg)	(kg)	(kg)	(%)	(%)	(kg)	(kg)	(%)	(%)
Total solids	44.76	27.38	17.73	39.6	20.8	11.36	33.96	75.8	17.3
Lactose	29.00	16.96	12.17	41.9	16.2	6.29	22.64	78.0	13.4
Ash	3.77	2.03	1.76	46.7	6.6	.49	3.29	87.3	4.8
Lactic acid	3.21	1.70	1.51	47.1	5.8	.35	2.78	86.5	3.9
Total nitrogen	.89	.73	.15	16.2	67.6	.67	.23	25.4	71.8
Non-protein N	.31	.18	.13	43.4	13.2	.10	.21	68.1	24.3
Protein ^c	3.71	3.56	.09	2.3	95.4	3.67	.09	2.3	97.4

^a Based on results from Table 1.

^b Based on per cent permeate removed.

^c Protein nitrogen \times 6.38.

that lactose passes through the membrane at the same rate as the water. Therefore, removal of 100% lactose from the whey could be achieved only by removing 100% of the water. Data in Table 3 show a lactose retention of 16% at 50% volume reduction. Thus, when 50% of the volume had been removed, 41.9% of the lactose had been removed whereas at 90% volume reduction, 78% of the lactose had been removed (Table 3). Obviously, the degree of shift in the protein-to-lactose ratio was directly related to degree of permeate removed. When the volume was reduced 90%, a concentrate was obtained containing 20 to 22% solids. On a dry weight basis, this "skimmilk equivalent" concentrate contained 35 to 37% protein and 50 to 52% lactose.

Higher ratios of protein-to-lactose were prepared by diluting the 35% protein concentrate with water and repeating ultrafiltration. A

comparison of dry weight percentages of the original whey, the "B" concentrate, and the "washed" concentrate are shown in Figure 1. The original whey had a protein-to-lactose ratio of 1:7.8, concentrate "B" had a 1:1.6 ratio, and in the "washed" concentrate the ratio had shifted to 2:1. Note also that the "washed" concentrate was practically devoid of ash and lactic acid. If membranes can be improved to permit 0% retention of lactose, rather than the 13% reported here, concentrates with significantly higher protein could be produced. One company reportedly has such membranes and has obtained up to 70% protein with a single, undiluted fractionation.

Fractionation rates. The rate of fractionation, expressed as gallons per square foot per hour, is a critical design parameter because it determines the membrane area requirement, or plant size, and thus capital cost. Figure 2 shows the average rate at 14 kg/cm² (200 psi) and 32.2 C of liquid whey processing, or fractionation, to different levels of volume reduction up to 90%. As shown earlier, relatively high volume reductions were necessary to achieve a significant increase in the protein-to-nonprotein solids ratio. A "skimmilk equivalent" concentrate was produced by 90% volume reduction at an average of 0.5 gallon per square foot per hour. Processing rates for the recycled, diluted concentrate were comparable to those shown; therefore the cost of producing a concentrate with 65% protein in the solids would be about twice that of the 35% protein con-

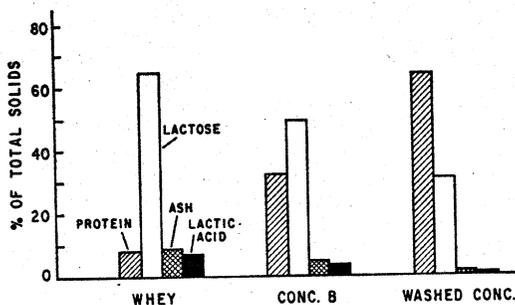


FIG. 1. Comparison of ratio of solids of whey and ultrafiltration fractions.

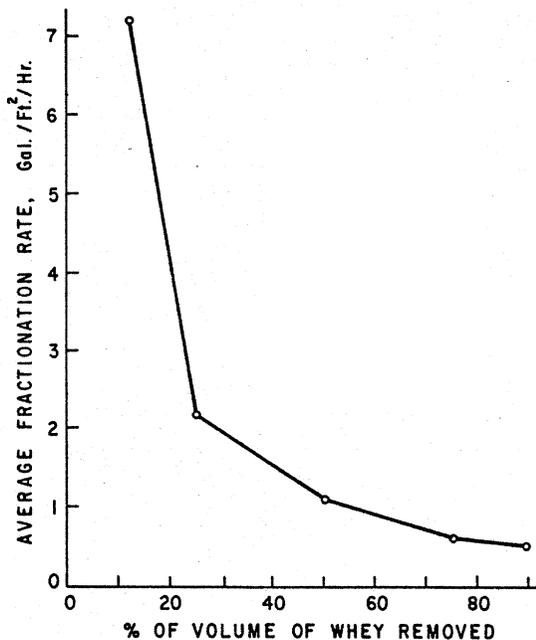


FIG. 2. Effect of extent of concentration on average whey fractionation rates. (Note: English system used to coincide with terminology of commercial membrane manufacturers.)

centrate. Insufficient data are available to make an accurate economic forecast, but considering capital cost and processing rates, it appears that the process will be economical.

In contrast to reverse osmosis, which requires pressures in excess of 42.2 kg/cm² (600 psi), ultrafiltration utilizes more open membranes which are not as dependent on osmotic pressure. These membranes are more susceptible to compaction from high pressures. Preliminary studies in our laboratory revealed a 50% decline in flux rates due to compaction at 31.6 kg/cm² (450 psi). The membranes used for this report have been operated for over 300 hr at 14 kg/cm² (200 psi) with no sign of deterioration. Other manufacturers offer ultrafiltration membranes designed to operate under 3.5 kg/cm² (50 psi).

High protein concentrates are attractive from the standpoint of increased utilization of whey and increased profits, but a pollution problem involving disposal of the permeate remains. This clear yellow-green liquid, with a solids content only slightly less than the original whey, is high in lactose and almost devoid of protein. Research is now under way to determine pos-

sible uses of this product as a source of lactose or animal feed.

References

- (1) Block, R. J., D. Bolling, K. W. Weiss, and G. Zweig. 1953. Studies on bovine whey proteins. *Arch. Biochem., Biophys.*, 47: 88.
- (2) Dunkley, W. L. 1969. Concentrating and fractionating whey. Report of a Symposium on Reverse Osmosis in Food Processing. January 23, 1969, by WURDD, ARS, USDA, Albany, California.
- (3) Fenton-May, R. I., C. G. Hill Jr., and C. H. Amundson. 1971. Use of ultrafiltration/reverse osmosis systems for the concentration and fractionation of whey. *J. Food Sci.*, 36: 14.
- (4) Horton, B. S., R. L. Goldsmith, S. Hossain, and R. R. Zall. Membrane separation processes for the abatement of pollution from cottage cheese whey. Presented at the Cottage Cheese and Cultured Milk Products Symposium, University of Maryland, College Park, Maryland, March 11, 1970.
- (5) Lim, T. H., W. L. Dunkley, and R. L. Merson. 1970. Role of protein in reverse osmosis of Cottage cheese whey. *J. Dairy Sci.*, 53: 645.
- (6) Marshall, P. G., W. L. Dunkley, and E. Lowe. 1968. Fractionation and concentration of whey by reverse osmosis. *Food Technol.*, 22: 969.
- (7) McDonough, F. E. 1968. Whey concentration by reverse osmosis. *Food Eng.*, 40: 124.
- (8) McDonough, F. E., and W. A. Mattingly. 1970. Pilot-plant concentration of cheese whey by reverse osmosis. *Food Technol.*, 24: 88.
- (9) Mitchell, H. H., and R. J. Block. 1946. Some relationships between the amino acid content of proteins and their nutritive values for the rat. *J. Biol. Chem.*, 163: 559.
- (10) Morris, D. L. 1948. Quantitative determination of carbohydrates with Dreywood's anthrone reagent. *Science*, 107: 254.
- (11) Osborne, T. B., and L. B. Mendel. 1924. The nutritive value of lactalbumin. *J. Biol. Chem.*, 59: 13.
- (12) Peri, C., and W. L. Dunkley. 1971. Reverse osmosis of Cottage cheese whey. 1. Influence of composition of the feed. *J. Food Sci.*, 36: 25.
- (13) Riggs, L. K., A. Beaty, and B. Mallon. 1955. Nutritive value of whey powder protein. *J. Agr. Food Chem.*, 3: 333.
- (14) Rowland, S. J. 1938. The determination of the nitrogen distribution in milk. *J. Dairy Res.*, 9: 42.