Production and Properties of Deproteinized Whey Powders

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ABSTRACT

Spray drying the deproteinized fraction from ultrafiltered whey was investigated, and the effects of processing on the distribution and loss of nutritional components were determined. Concentrates ranging from 30 to 66% solids were prepared and tested for the effects of solids, temperature, viscosity, and lactose crystallization on drying. Concentrates between 40 and 50% solids were readily spray dried whereas concentrates above 50% failed to dry. Attempts to obtain maximum lactose crystallization prior to drying did not prove beneficial. Acidity of the permeate was not a major factor in establishing drying capability. Foam-spray drying with air injection at the rate of 28 to 56 liters/min produced a free flowing powder with moisture content of 1 to 3%. Twelve lots of cottage cheese permeate were dried without appreciable difficulty. Powders were moderately hygroscopic and usually contained less than 1.5% moisture. No loss of nutritional components, e.g., amino acids, minerals, and vitamins, was caused by drying.

INTRODUCTION

The increasing demand for additional sources of food protein, coupled with the growing concern over environmental pollution, have intensified research in cheese whey utilization. The development of reverse osmosis and ultrafiltration equipment and its recent application in the concentration and fractionation of cheese whey have provided the industry with additional means for disposing of this byproduct from cheese manufacture (4, 10, 11, 12). In the ultrafiltration process, two fractions from whey are obtained. One is a relatively high-protein concentrate consisting mostly of lactalbumin and lactoglobulin which is retained by the membrane, and the other is the permeable fraction consisting mostly of lactose and salts.

Although utilization of the protein concentrate appears to be commercially feasible, the disposition of large volumes of deproteinized whey (permeate) still presents a serious problem. The permeate fraction represents about 90% of the original whey volume and contains from 80 to 85% of the original whey solids. Thus, the biological oxygen demand of the permeate is only slightly lower than that of the original whey.

This paper reports the manufacturing variables affecting the concentration and spray drying of cottage and Cheddar whey permeates. Particular emphasis was placed upon the drying of acid permeate from cottage whey since the drying of acid whey is difficult (6). The effects of ultrafiltration and spray drying on the various nutritional components of whey and permeate were determined also.

MATERIALS AND METHODS

Whey Fractionation

Cottage cheese whey was obtained from a local commercial producer. It was received while still hot from cooking (45 to 50 C), cooled to 3 to 5 C in a plate cooler, and stored in refrigerated tanks. Cheddar cheese whey was obtained from our own pilot plant operation, pasteurized immediately to stop further acid development, cooled, and stored under refrigeration.

All wheys were centrifugally clarified with a desludging separator just prior to ultrafiltration.

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[1] Mention of a trademark or proprietary product does not constitute a guarantee or warranty of the product by the U. S. Department of Agriculture, and does not imply its approval to the exclusion of other products that may also be suitable.

A model UF-44S pilot-plant ultrafiltration unit (Abcor, Inc., Cambridge, MA)[1] was used to fractionate cheese whey. The unit contained 20 tubes (2.5cm × 3m) equipped with HFA-180 membranes having a normal retention of 98.2% for whey protein. A temperature of 48 C and inlet-outlet gage pressures of 3.1–1 kg/cm^2 were utilized. Whey was circulated continuously through the tubes until sufficient permeate was removed. Lots of 1100 to 1500 liters were produced for each spray drying trial.

Permeate Condensing

The permeate was preheated to 72 C in a Mallory heater prior to condensing in a single-effect falling film evaporator (Wiegand). Inlet and outlet tube temperatures of 77 and 65 C in the evaporator were sufficient to concentrate the permeate to a solids content of 20 to 30% on the first pass. A second pass concentrated it to 40 to 60% solids.

Permeate Drying

Permeate was dried with a 2.74 m Grey-Jensen cyclone unit equipped with a spray nozzle having a 1-mm diameter orifice. Feed rate was between 200 and 234 kg/h with a hydraulic pressure of 126 kg/cm^2. In most trials, the dryer was operated with inlet and outlet temperatures of 132 and 82 C, respectively. The method of Hanrahan and Webb (6) was used to prepare foam-spray dried powders. Compressed air was injected in-line into the concentrate at the rate of 28 to 85 liters/min depending upon concentrate solids and viscosity. A minimum of 80 to 120 kg of concentrate were spray dried for each lot of powder.

Analytical Procedures

Total solids and moisture content. Total solids of concentrations were determined by the Mojonnier method (1). Moisture content of permeate powders was determined either by the conventional vacuum oven method (1) or the toluol distillation procedure (1).

Viscosity. Viscosities of the concentrates were determined with a Model LVB Brookfield viscometer.

Acidity. The effect of concentrate acidity on drying was determined over a pH range from 4.1 to 6.2. Condensed Cheddar whey permeates were varied for the upper pH values, and cottage whey permeates for the lower. Concentrates below pH 4.5 were prepared by adding concentrated lactic acid to the cottage whey permeate.

Lactose crystallization. Lactose crystallization in the concentrate was varied intentionally to determine its effect on drying and powder stability. Crystallization was encouraged by lactose seeding, stirring, concentrating to high solids, and varying the holding time and temperature of the concentrates. To establish the rate of lactose crystallization occurring after condensing, concentrates of varying solids were prepared. Samples were withdrawn at intervals and the extent of crystallization was determined by the method of Sharp and Doob (14) by using a Perkin-Elmer 141 Polarimeter. Permeate powders were tested for lactose crystallization within 24 h after they were dried. Total lactose was determined by official AOAC method (1).

Protein and nonprotein nitrogen (NPN). Protein was determined by a Kjeldahl procedure (1) and NPN as the soluble nitrogen remaining after protein precipitation with 12% trichloroacetic acid.

Mineral analysis. Samples were prepared for mineral analyses by dry ashing using the method of Garbe (5) and AOAC (1). Major minerals, except phosphorus, were determined with a Heath EU-703 absorption spectrophotometer equipped with a Varian Techtron Model 1000 air acetylene burner. Phosphorus was determined colorimetrically (9). Trace elements were determined by emission spectroscopy.

Hygroscopicity. Hygroscopicity of powder was determined by exposing samples in shallow layers at an atmosphere of 80% relative humidity, and measuring increase in weight during 24-h exposure.

Vitamin assays. Vitamin assays for wheys, permeates, and powders were performed by a private commercial laboratory using standard AOAC methods of analyses (1).

Amino acid assays. Amino acid assays were made of representative samples of Cheddar and cottage wheys and their fractions from ultrafiltration. Only the permeate fractions were tested for free amino acids. The unhydrolyzed permeates also were tested for amines, urea, and related physiological components. Aliquots for each sample were hydrolyzed at 110 C in 6N HCl in ampules sealed under vacuum, and

the amino acid analyses were with a Beckman Model 120 C amino acid analyzer in accordance with the method of Spackman et al. (15). Free amino acids in the permeates were determined according to the procedure of Benson and Patterson (2). The liquid samples were lyophilized and dissolved in pH 2.2 sodium citrate buffer prior to analysis. Available lysine was measured as the difference between total and "blocked" lysine as described by Blom et al. (3). Cystine was determined as cysteic acid after performic acid oxidation followed by acid hydrolysis according to the method of Moore (13). Tryptophan was measured by the procedure of Spies (16).

RESULTS

Composition of Ultrafiltrate

The permeability of whey components in the ultrafiltration of cottage and Cheddar whey is in Table 1. The percentage of protein removal with the HFA-180 membranes was essentially the same for cottage and Cheddar whey with a range of 94 to 99% for both types from separate runs. The lower values for protein removal usually occurred as the result of a small undetected leak in one of the module membranes. The membranes were permeable to lactose, lactic acid, salts, and low molecular weight compounds, resulting in little change between permeates and starting whey. Most of the nitrogen in the permeate was accounted for as nonprotein nitrogen. All milk fat in the wheys remained in the protein concentrate which did not pass through the membranes.

Condensing and Drying of Permeate

Effect of total solids. No difficulty was encountered in condensing the permeate to any solids up to 67%. However, concentrates above 60% total solids were very viscous and on standing for 30 min to 1 h solidified into a firm mass that was difficult to redissolve. In contrast, wheys of comparable total solids remained liquid for long periods. Apparently, the whey proteins retard solidification in condensed whole whey. Agitation of the concentrate was of value in slowing down solidification of the permeate when solids exceeded 50%.

Data in Table 2 show that increasing the solids above 40% markedly increased the viscosity of the concentrate. Increasing holding time and decreasing temperature also caused some increase, particularly at the higher solids concentrations. The viscosity of the lower solids concentrates did not change appreciably with variations in holding time and temperature.

Excessive viscosity of the high solids permeate concentrates made it impossible to pump the concentrate to the dryer and to maintain sufficient hydraulic pressure to atomize the concentrate. The length of time for holding the concentrate prior to drying did not appear to be a factor when the solids did not exceed 50%. Concentrates held between 25 and 42 C dried readily while those held at 10 C were usually too

TABLE 1. Composition of whey permeate in representative ultrafiltration trials.

	Cottage		Cheddar	
	Whey	Permeate	Whey	Permeate
Total solids (%)	6.42	5.8	6.7	5.7
Protein (%)	.53	.02	.60	.01
Fat (%)	.05	< .01	.25	< .01
Lactose (%)	4.4	4.3	5.0	4.9
Lactic acid (%)	.47	.44	.14	.14
Ash (%)	.60	.56	.52	.50
Total nitrogen (mg/g)	1.19	.33	1.30	.26
NPN (mg/g)	.34	.30	.34	.24
Protein N (mg/g)	.85	.03	.95	.01
Soluble N (mg/g)	1.18	.33	1.30	.27
NPN (as % N)	28.6	90.9	26.2	92.3
pH	4.7	4.7	6.1	6.1
Protein removal (%)	. . .	96.0	. . .	98.4

TABLE 2. Effect of total solids, holding time and temperature on the viscosity of permeate concentrate.

Total solids	Temperature	Viscosity time (h)				
		0	.5	1	2	3
(%)	(C)	(Centipoise)				
30	42	7.5	12.5	12.5	12.5	15
40		57.5	100	300	300	300
50		1800	1200	3600	3750	4000
60		5100	7800*	9000*	12,500*	**
66		9000*	**	**	**	**
30	25	10	15	15	15	15
40		82.5	125	400	400	400
50		2000	1500	3600	3750	4000
60		5000	7200	9800*	12,500*	**
66		9800*	**	**	**	**

*Semi solid.

**Solid mass.

viscous to pump and required warming to at least 25 C. In two trials in which the total solids exceeded 50%, the permeate could not be spray dried. In subsequent trials, all concentrates above 50% solids were reduced to the range of 40 to 50% by added water.

Effect of lactose crystallization. Since it was suggested (17) that lactose crystallization in whey powders lessens the tendency for water absorption, controlled crystallization was investigated as a means of increasing drying capability and providing a more stable product. The rate of lactose crystallization in concentrates of different total solids was determined, and the results are in Fig. 1. Most of the lactose crystallized in the 1st h at 42 C. No significant increase in crystallization occurred even when the concentrate was held as long as 16 h.

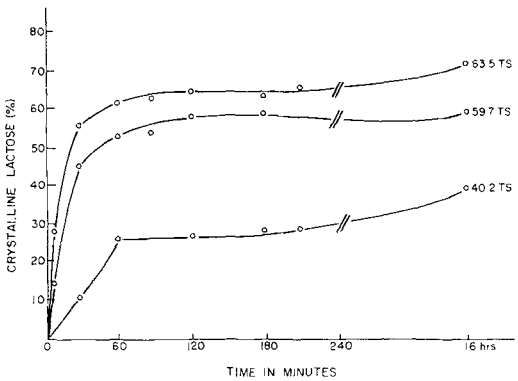

FIG. 1. Rate of lactose crystallization in permeate concentrate at 42 C as determined by the method of Sharp and Doob (14).

The effects of total solids and of varying holding conditions for lactose crystallization are in Table 3. Crystallization of lactose was affected primarily by total solids in the concentrate and holding time, and to a lesser degree by holding temperature and stirring. The greater the total solids, the higher the degree of crystallization. The extent of crystallization in the final concentrate and powder could be predicted closely by the degree of concentra-

TABLE 3. Factors affecting lactose crystallization in permeate concentrate.

Total solids	Holding period		Crystalline lactose
	Time	Temperature	
(%)	(h)	(C)	(%)
41.9	1	40	27.0
41.9	2	40	28.2
41.9	18	40	30.4
41.9	18	10	32.5
45.6	2	40	32.0
52.8	18	10	56.3
52.8*	18	40	54.5
63.8	.5	42	45.0
63.8	1	42	52.9
63.8	18	42	59.3
63.8	18	10	61.3
66.2	2	40	72.4

*Stirred.

tion during condensing. Powders with a high degree of crystallization were obtained by concentrating the permeate to approximately 60% solids, holding the concentrate for 1 to 2 h at 40 C, adjusting to 50% total solids with water and drying. Prolonged holding had little effect on extent of crystallization, and seeding the concentrate with lactose did not increase the rate of crystallization. Neither did dilution of high solids concentrates prior to drying reduce the amount of crystallization.

Effect of pH. Because of the inherent difficulty in drying acid cottage cheese whey, it was anticipated that the acidity of the permeate would be a major factor in drying. In addition to the normal acidities of cottage whey permeate, extreme acid conditions were imposed to determine their effect on drying. Permeates within the range of pH 4.1 to 6.2 dried readily and those with low pH appeared to dry as well as those with high pH.

Effect of foaming. Foam-spray drying or puff drying by air injection was not essential to dry the permeate but was of value in obtaining a drier, free-flowing product. Some evidence of powder sticking and build-up was noted on dryer equipment in conventional drying with one lot of cottage whey permeate.

Properties and Composition of Permeate Powder

Hygroscopicity. All permeate powders remained free-flowing in storage and were moderately hygroscopic regardless of the degree of crystallization in processing.

As shown in Table 4, powders with a high degree of crystallization absorbed moisture at 80% relative humidity at about the same rate as those with low values. No correlation between hygroscopicity of the powders and lactose crystallization could be demonstrated. There was, however, a direct correlation between hygroscopicity and the amount of lactic acid in the powder as indicated by titratable acidity (TA). Cottage whey permeate acidified to pH 4.1 (8.6% TA) was the most hygroscopic while Cheddar whey permeate was the least.

Titratable acidity. If all lactic acid in the cottage whey permeate remained during concentration and drying, the titratable acidity for all lots would have been 9 to 10%. Actual values for the permeate after it was dried were from 6.8 to 8.3% indicating some loss in evaporation. The loss was not measured quantitatively, but lactic acid was readily detected in the condensate from the evaporator. The greater the concentration, the greater the loss of lactic acid and the lower the TA in the powder.

Proximate composition. Ranges and mean values for individual components of cottage and Cheddar whey permeate powders are in Table 5. The data represent values for 11 foam-spray dried and three conventional spray-dried powders.

Amino acids. Individual amino acid analyses

TABLE 4. Hygroscopicity of permeate powder at 80% relative humidity.

Powder source	Titratable acidity	Crystalline lactose	Moisture 6 h	Increase 24 h
		(%)		
Cottage[a]	8.6	32.0	10.3	25.6
Cottage[b]	8.35	27.6	10.2	25.2
Cottage	8.14	65.0	10.2	24.5
Cottage	7.6	38.9	9.8	24.0
Cottage	7.6	67.3	9.7	23.8
Cottage	7.05	69.0	9.6	21.6
Cottage	7.0	57.0	9.5	22.2
Cottage	6.9	59.0	9.0	22.1
Cottage	7.2	66.2	9.2	22.1
Cottage	7.0	65.0	8.9	20.1
Cheddar[c]	1.9	64.9	7.8	17.6
Cheddar	1.8	53.0	7.6	17.5

[a]Cottage whey permeate acidified with lactic acid.

[b]Normal cottage whey permeate.

[c]Cheddar whey permeate.

TABLE 5. Analyses of powders prepared from cottage and Cheddar cheese permeates.

	Cottage		Cheddar	
	Range	Mean	Range	Mean
Moisture (%)	1.27 – 3.05	1.61	.68 – 1.11	.94
Lactose (%)	67.1 – 74.0	70.9	77.6 – 82.2	79.6
Protein (%)[a]	.27 – 1.69	.68	.20 – .43	.38
Total nitrogen (%)	.56 – .61	.57	.46 – .56	.51
Nonprotein nitrogen (%)[b]	.45 – .51	.48	.43 – .50	.47
Fat (%)	< .01	< .01	< .01	< .01
Ash (%)	8.2 – 12.4	10.97	8.07 – 8.60	8.30
Lactic acid (%)	6.9 – 8.3	7.1	1.7 – 1.9	1.8
Crystalline lactose (%)	27.2 – 69.6	57.4	61.2 – 64.2	62.4
Bulk density	.23 – .35	.32	.36 – .48	.39
pH (10% solution)	4.5 – 4.8	4.7	5.8 – 6.1	6.0

[a]Precipitable protein with TCA.

[b]N soluble in 12% TCA.

of liquid cottage and Cheddar wheys and their respective permeates from ultrafiltration are in Table 6. Amino acids from hydrolyzed wheys and permeates are expressed as total amino acids. Free amino acids were determined in the unhydrolyzed permeate samples. Individual amino acid analyses from the two types of whey were close; however, the permeates showed sharp differences. Free amino acids in the cottage whey permeate were usually higher than for Cheddar whey permeate. Amino acid analyses of permeate powders indicated that there was little or no loss of amino acids due to condensing and drying.

TABLE 6. Amino acids in liquid whey and ultrafiltered permeates.

	Total[a]				Free[b]	
	Cottage		Cheddar			
	Whey	Permeate	Whey	Permeate	Cottage permeate	Cheddar permeate
	(mg/100 g)					
Lysine	72.3	4.0	71.6	2.1	2.5	.4
Histidine	15.5	1.0	13.1	.6	.3	.1
Arginine	18.6	.9	18.1	.8	.4	.2
Aspartic acid	80.0	3.4	81.8	2.1	.5	.2
Threonine	40.7	1.6	50.2	.9	< .1	< .1
Serine	37.7	2.1	40.8	1.1	.1	< .1
Glutamic acid	139.1	10.0	140.1	6.1	7.7	2.9
Proline	40.8	3.7	48.4	2.1	5.3	.7
Glycine	16.2	.9	16.8	2.3	.1	.9
Alanine	33.2	1.1	37.1	1.4	.3	.4
Cystine	15.4	.3	9.6	.4	0	0
Valine	43.1	1.9	46.2	1.2	.1	.1
Methionine	14.9	.4	13.8	.3	< .1	< .1
Isoleucine	38.8	1.9	49.8	.9	< .1	< .1
Leucine	85.1	2.4	81.8	1.6	.2	.1
Tyrosine	23.2	1.0	19.0	.5	2.1	.2
Phenylalanine	27.1	1.2	24.5	.8	.4	.1
Tryptophane	15.5	.3	16.3	.5	.1	< .1

[a]Total amino acids after hydrolysis.

[b]Free amino acids prior to hydrolysis.

TABLE 7. Major mineral content of whey and permeates.

	Ash	Calcium	Phosphorus	Magnesium	Sodium	Potassium
	(%)			(mg/100 g)		
Cottage						
Liquid whey	.59	86.0	63	8.9	40	133
Liquid permeate	.56	82.0	62.7	8.2	32	114
Dried permeate	12.2	1410	857.3	155.1	770	2535
Cheddar						
Liquid whey	.52	22.5	42.5	5.8	35	109
Liquid permeate	.50	21.0	49.6	5.3	31.8	108
Dried permeate	8.07	480	637.7	113.4	780	2610

Additional nitrogen compounds in whey permeates were urea, citrulline, asparagine, glutamine, ethanolamine, phosphorylethanolamine, glycerophosphorylethanolamine, phosphorylserine, and phosphorylthreonine. Although quantitative differences were indicated between cottage and Cheddar whey permeates, more samples need to be analyzed to determine these differences.

Mineral composition. Ash and major mineral components of permeates are in Table 7. Liquid samples of permeate showed only a slight reduction in ash content and major minerals from the starting wheys. However, the ash content for the dried cottage cheese whey permeate was consistently greater than for Cheddar whey permeate. Similarly, the mineral content for calcium, phosphorus, and magnesium was consistently higher in dried cottage whey permeate than for dried Cheddar whey permeate. The sodium and potassium content for the two types of permeate were essentially the same. Ranges are in Table 8 for nine trace elements in permeate powders. The greatest variation was in iron content. Iron contamination from equipment and or water supply may have caused this variation.

TABLE 8. Range of trace minerals in permeate powders.

	(ppm)
Aluminum	23.2 – 27.9
Barium	.9 – 1.2
Iron	3.0 – 11.3
Strontium	4.6 – 5.9
Boron	3.0 – 3.7
Copper	1.2 – 2.9
Zinc	30.0 – 33.0
Manganese	.5 – .6
Chromium	2.2 – 3.4

Vitamins. Liquid whey and fractions from ultrafiltration were assayed for seven vitamins that are considered significant in whey and dairy products. Mean assay values are in Table 9 for liquid and dried fractions. Vitamin assays for the liquid samples showed a high degree of variability whereas assays of dried fraction were more uniform and reproducible. No major reduction of vitamins could be detected as the result of ultrafiltration. Vitamin assays for dried permeates were in general agreement with those for dried whey (8).

DISCUSSION

If the process for recovery of whey proteins by ultrafiltration is to become commercially feasible, some provision must be made for disposing of or utilizing vast quantities of deproteinized whey. Advantages and need for drying deproteinized fraction are obvious. However, investigations on the drying of the permeate have not been reported.

Fourteen lots of permeate were spray dried and only two instances was any difficulty encountered in the drying operation. These lots failed to dry primarily because of excessive solids in the concentrate and inability to maintain sufficient hydraulic pressure to the dryer.

High solids permeate concentrates crystal-

TABLE 9. Comparison of vitamins in whey and whey fractions from ultrafiltration.

	Liquid		Dried	
	Whey	Permeate	Protein[a]	Permeate
	(mg/kg concentrate)			
Thiamine	.3	.3	4.5	4.9
Niacin	.9	.4	5.9	5.3
Pantothenic acid	4.0	3.8	38.8	48.8
Riboflavin	1.4	1.2	9.6	14.5
Folic acid	.1	.1	.2	.3
B_{12}	.002	.003	.065	.025
Choline	88.0	59.2	3150	3960

[a]52% protein fraction.

lized and solidified at a much lower total solids content than whole whey concentrates containing protein. Thus, concentrate viscosities for the permeates were more critical than those for drying whole whey. An optimum of 40 to 50% solids was established for drying. Foam-spray drying was superior to conventional drying in assuring a dry product with low moisture. In contrast to the drying of acid cottage cheese whey, the acidity of the permeate did not seem to be an obstacle to drying. Acid permeates dried as readily as sweet permeates; thus, the removal of protein from cottage whey improves its drying capability. A high degree of lactose crystallization prior to drying was not essential to obtain a stable dry powder. Concentrates with low crystallization were foam-spray dried as readily as those with high crystallization.

Compositional data in earlier studies for whey and permeate have shown that most of the components of whey, except the proteins and residual milk fat, pass through the membranes in ultrafiltration. No additional loss of nutrients could be detected as a result of drying. Amino acid composition for cottage and Cheddar whey permeates were similar although more free amino acids were in acid permeates.

Acid whey permeate may be spray dried readily without the problems encountered during the drying of whole whey. The dried product is stable and uniform and should find ready use in various foods, feed formulations, and bacterial media. This provides another outlet for its use (7) and makes removal of the protein a more profitable procedure.

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