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SPATIAL DISTRIBUTION OF MILK CONSTITUENTS IN POWDERS MADE BY DIFFERENT DRYING TECHNIQUES

SUMMARY

Layers of milk solids were washed from the surfaces of five different types of powder granules by using a vacuum to draw successive 50-ml aliquots of water rapidly through a bed containing a mixture of 5 g of milk powder and 60 g of 20-30 mesh sand.

Analysis of the washings for total solids, fat, lactose, and protein, as well as determination of the freezing point and conductivity of the solutions, demonstrated that some migration of low molecular weight solutes towards the surface occurred during both the spray and foam drying processes. Instantizing apparently increased the amount of osmotically active material at the surfaces of two of the four samples studied. Foam-dried whole milk powder granules were found to be the most uniform in composition. No relationship could be established between the observed orientation of milk constituents within the powder granules and their dispersibility.

A mathematical analysis of a model washing process is presented, as well as evidence for the conformation of the described technique with the model system.

Different types of milk powders made available by recently developed drying techniques allow a more thorough study of factors influencing their dispersibility.

Bockian et al. (3) directed attention to the influence of the relative spatial distribution of the different milk constituents in the milk powder particle on the dispersibility of the dehydrated material. They reported that, in instantized skimmilk powders, the osmotically active constituents are concentrated on the exterior of the particle, thereby enhancing dispersibility. This observation was based on data obtained by use of a washing technique which removed layers of milk constituents from individual powder particles by rapidly sweeping a convenient volume of water through a bed of milk powder.

Van Kreveld and Verhoog (7) suggest that when the powder particle is wetted, the osmotically active components dissolve and immediately diffuse from the particle into the water; the other milk components then dissolve into an aqueous solution of milk salts and lactose. Therefore, the simple wetting of the powder particle during the washing process would be responsible for the observed effects which Bockian et al. (3) ascribed to instantizing.

This paper reports the establishment of criteria a true washing technique should satisfy and the development of a modification of the technique of Bockian et al. (3) which satisfies these criteria. Results obtained using this modification to study the spatial orientation of milk components in the powder granules produced by various drying techniques are also presented in terms of the concentration of various milk constituents found in the successive layers removed from the powder granules by a systematic washing of their exposed surfaces.

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MATERIALS AND METHODS

The foam-dried milk powders used in this study were prepared using the procedure of Sinnamon et al. (6). The process entails pasteurization, condensation to 50% total solids, homogenization, incorporation of nitrogen gas, and drying the resultant foam at low temperatures in a vacuum shelf dryer.

The spray-dried skimmilk powder and whole milk powder No. 1 were prepared using a 9-ft diam Swenson[1] spray dryer operating with 40% solids in the feed, an inlet temperature of 280 F, and an outlet temperature varying between 185 and 195 F. Spray-dried whole milk powder No. 2 and instantized skimmilk powders used were commercial products obtained through regular trade channels.

[1] Reference to certain products or companies does not imply an endorsement by the Department over others not mentioned.

The dispersibility of each powder was measured using the method of Sinnamon et al. (6).

To remove successive layers of solids from the powder particles, 5 g of the milk powder being studied were thoroughly mixed with 60 g of Ottawa 20-30 mesh sand and layered in a coarse sintered glass funnel of 7.5-cm internal diameter. A piece of Whatman No. 2 filter paper[1] of like diameter was placed over the powder-sand mixture. The entire assembly was connected to a line pulling a 29-in. vacuum through a 250-ml suction flask. A 50-ml aliquot of water was poured onto the filter paper and the vacuum immediately applied. This aliquot was drawn through the sand-milk powder bed in less than 1 sec. The wash water was collected and successive washings were made in rapid fashion by two operators. The interval between washings was reduced to 5 sec or less. With some powders, seven successive washings could be made. All washings were done at room temperature. Subsequently, each fraction was analyzed for total solids, nitrogen, lactose and, in the case of whole milk powders, total fat. The freezing point depressions effected by the solids in the wash water as well as the conductivity of the solutions were determined.

The Ottawa sand used was obtained from the Fisher Scientific Company,[1] Silver Spring, Maryland. Before each washing study, the sand was soaked 24 hr in hot concentrated hydrochloric acid. It was then washed with tap water, then distilled water. The distilled water washing was continued until determinations showed the wash water to have a freezing point depression of 0 C.

Freezing point determinations were made using a standard Hortvet cryoscope.

Conductivity readings were made at 1 C using an Aminco[1] conductivity cell and a Model RC 16B conductivity bridge from Industrial Instruments, Inc.[1]

Total solids and total fat were determined using the standard Mojonnier method (5).

Nitrogen was determined by a micro-Kjeldahl method described by the A.O.A.C. (2). Nitrogen values were then converted to protein by use of the factor 6.25.

Lactose was determined polarimetrically using the reagents described by the A.O.A.C. (1). Polarimetric analyses were made with a Beckman[1] DU Spectrophotometer, using a standard Model D Keston[1] polarimeter attachment.

[1] Reference to certain products or companies does not imply an endorsement by the Department over others not mentioned.

RESULTS AND DISCUSSION

When the method of Bockian et al. (3) was employed in the analysis of the orientation of the components of the milk powder particles, it was difficult to prevent channeling in the powder bed. Therefore, the powder was dispersed in a porous hydrophilic supporting agent, to avoid all evidence of channeling during washing. It became apparent that no criteria, other than the absence of channeling, had been established to demonstrate when a true washing of the powder particles was occurring.

A true washing technique requires that, using the same volume of wash water, each successive washing must remove the same fraction of the remaining powder, provided that the powder is homogeneous with respect to composition and structural rigidity.

An analytical expression for this criterion was developed as follows:

let q = initial quantity of powder

a = fraction of powder removed by each washing

W = quantity of powder removed by each washing

N = number of washing

when $N = 1$

amount removed, $W_1 = aq$

amount left $= q - aq = q\,(1 - a)$

when $N = 2$

amount removed, $W_2 = aq\,(1 - a)$

amount left $= q - aq - aq\,(1 - a)$
$= q\,(1 - a)^2$

when $N = k$

amount removed,

$$W_k = aq\,(1 - a)^{k-1} \tag{1}$$

$$\text{amount left} = q\,(1 - a)^k \tag{2}$$

taking logarithms of each side of Equation (1)

$$\log W_k = \log aq + (N - 1) \log (1 - a) \tag{3}$$

According to Equation (3), plotting log W versus the number of the washing less one $(N - 1)$ should give a straight-line graph with slope of $\log (1 - a)$ and intercept equal to $\log aq$. This equation was shown to be valid by determination of the solids content of the washing fractions of crystalline lactose and other crystalline materials of known homogeneity.

Results obtained by washing a sample of foam-dried whole milk powder are presented in Figure 1. Numbers on the lines represent

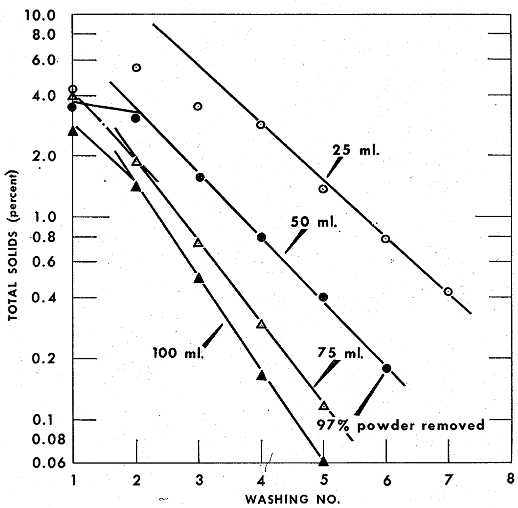

FIG. 1. Effect of varying the volumes of wash water on the per cent total solids in successive washings of foam-dried whole milk powder.

TABLE 1

Per cent powder removed by the end of the fourth washing

Powder	% Powder removed
Foam-dried whole milk powder	91.9
Spray-dried whole milk powder (1)	82.4
Spray-dried whole milk powder (2)	70.3
Foam-dried nonfat milk powder	92.1
Spray-dried nonfat milk powder	92.5
Instant nonfat milk powders A	85.7
B	84.2
C	97.6
D	92.3

the volume in milliliters of the aliquots of wash water. For clarity, in Figure 1, the per cent concentration of solids in the wash water is plotted on the ordinate axis instead of the quantity of powder removed. The slopes of the lines are the same in either case, but by plotting only per cent concentration, the lines are displaced vertically from one another. The amount of this displacement is equal to the logarithms of the volume of the aliquots of wash water.

The log (per cent solids) versus $(N-1)$ for successive washings was found to be linear when volumes of wash water of 50, 75, and 100 ml were used, with the exception of the data for the first washing. The log plot of the data for 25 ml of wash water indicated that this was the minimum volume of wash water that should be used with an apparatus of the dimensions used in this experiment, because the data obtained began to deviate from linearity at this point. An aliquot of 50 ml per wash was selected for routine use, since it permitted distribution of solids through a sufficient number of washings to make detection of layering in the particles possible (see Table 1).

The ability of this technique to detect layering of chemical constituents within powder granules was checked by washing sugar-coated milk powder particles. The particles were prepared by mixing foam-dried whole milk powder with confectionery sugar in the ratio of 4 g milk powder to 1 g sugar, equilibrating the mixture against an atmosphere of 80% relative humidity for 15 hr, then drying the mixture at 40 C under 28 in. vacuum for 72 hr. Results obtained by washing this material are shown graphically in Figure 2. The data for total solids removed show that the first washings removed a much more soluble material than was present in succeeding washings. The freezing point depression of the solution of the first washing was 0.389 C, whereas the freezing point depression of a solution of foam-dried whole milk solids at the same concentration was 0.262 C. The freezing point depression data show that the material removed in the first washings consists largely of powdered sugar on the surface of the milk powder.

Results obtained from the chemical analyses of the washings of various milk powders are presented in Table 2. Data presented in Table 1 have shown that the material removed by

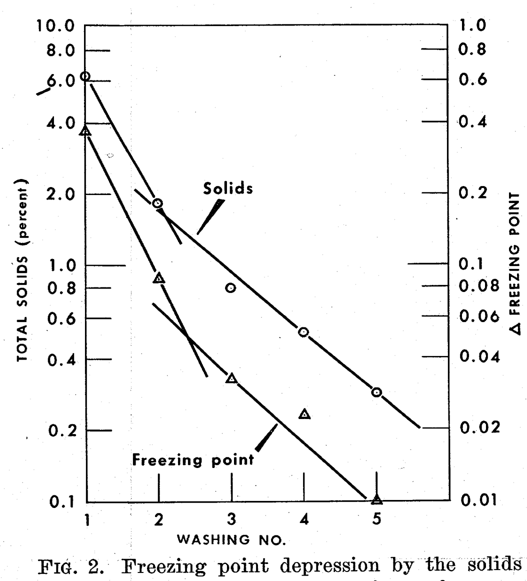

FIG. 2. Freezing point depression by the solids successively washed from the surface of sugar-coated foam-dried whole milk powder (1:4 W/W) by use of 50-ml aliquots of wash water.

TABLE 2
Chemical analysis of the washing fractions of the various powders

Ratio	Washing no. 1	2	3	4
		Foam-dried whole milk powder		
Fat/Total Solids (T.S.)	.24	.28	.31	.35
Lactose/T.S.	.35	.31	.29	.28
Protein/T.S.	.24	.28	.32	.31
Salts/T.S.[a]	.17	.13	.08	.06
Lactose/Protein	1.46	1.11	.91	.90
		Spray-dried whole milk powder (1)		
Fat/T.S.	.17	.26	.39	.40
Lactose/T.S.	.44	.37	.31	.22
Protein/T.S.	.21	.28	.31	.30
Salts/T.S.	.18	.09	..	.08
Lactose/Protein	2.10	1.32	1.00	.73
		Spray-dried whole milk powder (2)		
Fat/T.S.	.21	.25	.26	.34
Lactose/T.S.	.48	.37	.36	.25
Protein/T.S.	.20	.23	.24	.31
Salts/T.S.	.11	.15	.14	.10
Lactose/Protein	2.40	1.61	1.50	.81
		Foam-dried nonfat milk powder		
Lactose/T.S.	.70	.66	.60	.83
Protein/T.S.	.37	.41	.38	.42
Salts/T.S.	....	....	.02	....
Lactose/Protein	1.89	1.61	1.58	1.98
		Spray-dried nonfat milk powder		
Lactose/T.S.	.62	.54	.57	.42
Protein/T.S.	.31	.39	.43	.46
Salts/T.S.	.07	.07	....	.12
Lactose/Protein	2.00	1.38	1.33	.91

Instantized nonfat milk powders[b]

Ratio	Washing no. 1	2	3	4
		Instantized Powder A		
Lactose/T.S.	.65	.58	.50	.50
Protein/T.S.	.29	.36	.41	.37
Salts/T.S.	.06	.06	.09	.13
Lactose/Protein	2.24	1.61	1.22	1.35
		Instantized Powder B		
Lactose/T.S.	.60	.54	.55	.58
Protein/T.S.	.32	.34	.39	.40
Salts/T.S.	.08	.12	.06	.02
Lactose/Protein	1.88	1.59	1.41	1.45
		Instantized Powder C		
Lactose/T.S.	.47	.40	.34	....
Protein/T.S.	.34	.35	.37	....
Salts/T.S.	.19	.23	.29	....
Lactose/Protein	1.38	1.14	.92	....
		Instantized Powder D		
Lactose/T.S.	.51	.44	.34	.30
Protein/T.S.	.33	.39	.46	.45
Salts/T.S.	.16	.17	.20	.25
Lactose/Protein	1.55	1.13	.74	.67

[a] Salts/T.S. = 1.00 − (Lactose/T.S. + Protein/T.S.).
[b] These powders are commercial products purchased through regular trade channels.

four successive washings represents the bulk of the material in the powder granules. A comparison of the ratios of the concentrations of the various components in each wash shows where evidence for layering in the granules exists. Table 3 shows results obtained from the freezing point and conductivity determinations. These data indicate the extent to which the

TABLE 3

Freezing Point Depression (F.P.) per per cent Total Solids (T.S.) and Conductivity ($\lambda \times 10^4$) per per cent total solids of the washing fractions of the various powders

	Powder									
	Foam-dried whole-milk		Spray-dried whole milk (1)		Spray-dried whole milk (2)		Foam-dried nonfat milk		Spray-dried nonfat milk	
Wash no.	FP/% TS	Cond./ % TS	FP/% TS	Cond./ % TS	FP/% TS	Cond./ % TS	FP/% TS	Cond./ % TS	FP/% TS	Cond./ % TS
	(C)	*($\lambda \times 10^4$)*	*(C)*	*($\lambda \times 10^4$)*	*(C)*	*($\lambda \times 10^4$)*	*(C)*	*($\lambda \times 10^4$)*	*(C)*	*($\lambda \times 10^4$)*
1	.0515	4.17	.060	3.93	.0598	5.18	.0574	3.47	.0652	3.93
2	.0412	2.95	.0459	5.00	.0469	3.40	.0571	4.13	.0568	3.96
3	.0703	2.58	.0267	2.68	.0387	3.46	.0518	4.82	.0493	4.28
4	.0358	2.59	.0226	3.34	.0268	3.50	.0483	6.19	.0264	4.27
	Instantized nonfat milk powders									
	A		B		C		D			
1	.069	5.22	.071	5.22	.060	3.77	.0642	4.18		
2	.062	4.92	.057	4.26	.058	4.35	.0568	3.96		
3	.055	4.36	.053	4.50	.0327	4.48	.0459	4.62		
4	.043	4.00	.036	4.33	.0417	5.00	.0468	4.65		

osmotically active constituents present in the various powders tend to concentrate in the surface zone. Table 4 shows the per cent dispersibility of each of the powders used in this study.

TABLE 4

Dispersibility of the various powders studied

Powder	Dispersibility
	(%)
Foam-dried whole milk powder	96.0
Spray-dried whole milk powder (1)	65.7
Spray-dried whole milk powder (2)	82.0
Foam-dried nonfat milk powder	98.0
Spray-dried nonfat milk powder	82.1
Instantized nonfat milk powder A	92.0
B	98.0
C	93.0
D	89.6

From Tables 2 and 3, it can be seen that there is greater spatial orientation of the milk constituents in spray-dried whole milk powder than in foam-dried whole milk powder. Foam-dried skimmilk powders show much greater spatial orientation of the constituents than foam-dried whole milk powders. The converse is true of spray-dried nonfat solids. Instantized Powders A and B have a greater portion of low molecular weight components at or near their surfaces than Powders C and D. However, the two sets are similar within sets. Upon comparison with spray-dried skimmilk powder, the instantized Powders A and B have a spatial orientation similar to that of the unagglomerated spray-dried skimmilk powder. Powders C and D are more uniform in composition than the regular spray-dried powder, and have component distribution characteristics similar to those of the foam-dried whole milk powder.

On the basis of these analyses, it may also be noted that in the foam-drying process, presence of fat in the milk results in a more uniform distribution of milk components within the powder granule. No similar effect is observed in spray-dried materials.

The higher homogenization pressures used in the foam-drying process may be responsible for the above effect. A previous report shows that high-pressure homogenization of high solids concentrates promotes the formation of complexes having high protein-to-fat ratios (2). Presence of these complexes in the foam-dried whole milk powder could possibly explain the reduced rate of migration of low molecular weight constituents toward the drying surface.

Comparison of data in Tables 2, 3, and 4 shows no relationship between constituent orientation and observed dispersibility. It appears that some factor or factors other than spatial orientation are responsible for the enhanced solubility of instantized and foam-dried powders.

Our data for some of the instantized powders are at variance with those reported by Bockian et al. (3). However, these powders may have been manufactured by methods other than those used in the prior study.

REFERENCES

(1) ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS. Official and Tentative Methods of Analysis. 6th ed., p. 309. Washington, D. C. 1945.

(2) ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS. Official and Tentative Methods of Analysis. 6th ed., p. 763. Washington, D. C. 1945.

(3) BOCKIAN, A. H., STEWART, G. F., AND TAPPEL, A. L. Factors Affecting the Dispersibility of ''Instantly Dissolving'' Dry Milks. Food Research, 22: 69. 1957.

(4) FOX, K. K., HOLSINGER, V. H., CAHA, J., AND PALLANSCH, M. J. Formation of a Fat Protein Complex in Milk by Homogenization. J. Dairy Sci., 43: 1396. 1960.

(5) MOJONNIER, J., AND TROY, H. C. The Technical Control of Dairy Products. 1st ed., pp. 93-129. Mojonnier Bros. Co., Chicago, Illinois. 1922.

(6) SINNAMON, H. I., ACETO, N. C., ESKEW, R. K., AND SCHOPPET, E. F. Dry Whole Milk. I. A New Physical Form. J. Dairy Sci., 40: 1036. 1957.

(7) VAN KREVELD, A., AND VERHOOG, J. H. A New Solubility Test for Whole-Milk Powder. Netherlands Milk Dairy J., 17: 209. 1963.