

## FREE FAT IN FOAM-DRIED WHOLE MILK

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### SUMMARY

Concentrates of 50% T. S. were foamed and dried in a vacuum shelf drier, and comminuted through a 20-mesh screen. Methods were developed for determination of free fat (by extraction with carbon tetrachloride) and of that fat readily separated by centrifugation of the reconstituted product.

Large increases in free fat (from 10 to over 90% of the total fat) resulted from aging and lactose crystallization of the concentrate. This effect was reversed by heating immediately before drying. Free fat decreased slightly with increasing homogenization pressure. Free fat contents less than 10% of the total fat were obtained by proper control of these factors.

Dispersibility of the dry milk decreased slightly with increase in free fat. The fat rise under centrifugal force in reconstituted milk was not affected by variation in free fat to about 50%. At high free fat levels, part of the fat was readily separated by centrifugation. Free fat increased with decrease in particle size. A fraction obtained by comminution through a 40-mesh screen, followed by removal of the finest particles by sieving on a 60-mesh screen, was similar in free fat, more dispersible, and much higher in bulk density when compared to the product obtained by comminution through a 20-mesh screen.

The fat in dried whole milk which is extracted by carbon tetrachloride was designated as free fat by Holm *et al.* (5). It was considered as "fat not protected by a protein film." In spray-dried milks the free fat increased slowly with increase in the total fat. Above 24% total fat a steep rise in free fat occurred. Homogenization decreased the amount of free fat and improved the keeping quality. King (6) concluded from microscopic examination of reconstituted milks that free fat in dried milks is demulsified fat. Carbon disulfide, ethyl ether, and carbon tetrachloride were found by Lampitt and Bushill (8) to extract similar quantities of fat from milk powder. They used carbon disulfide as a standard solvent for extraction. The free fat varied from 3 to 14% of the total fat in spray-dried milks, and from 92 to 96% in roller-dried milks. It varied from 58 to 92% in freeze-dried milk according to Nickerson *et al.* (10), but was reduced to from 35 to 75% by homogenization. In spray-dried milks from the same concentrates it varied from 12 to 19% of the total fat. Freeze-dried whole milks were difficult to reconstitute, due to the presence of free fat.

Shipstead (13) found that the free fat was oxidized first during storage of spray-dried milk, and concluded that the rest of the fat is protected from oxidation by solids-not-fat. King (6) stated that part of the free fat is on the surface of dried milk particles, thus making them more hydrophobic and less wettable. Coulter *et al.* (3) have shown that the amount and dispersion of fat in dried milk affect wettability. Stone *et al.* (15) demonstrated a sharp decrease in self-dispersion and ease of dispersion of dried whole milk, if solid fat was

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present. Results obtained by Litman and Ashworth (9) indicate that the free-fat content of dried milk may be related to the development of scum during storage. The development of a fat-protein complex was postulated.

The methods of determining free fat are empirical, and the specific properties of the fractions extracted with various organic solvents have not been defined. It is clear, however, that the amount extracted from dried whole milk varies between wide limits, depending upon many factors. The free fat has been associated both with poor dispersing properties and with oxidative deterioration during storage.

The present study was undertaken to obtain more information concerning the free fat in foam-dried whole milk, a new physical form with excellent dispersibility in water (14). This paper deals mainly with the effect of certain factors on the amount of free fat and with the relationship between free fat and dispersibility. Also, information was gathered on the significance of free fat in relation to some other properties of the dried milk.

### METHODS

*Determination of free fat.* After investigating several methods of mixing dry milk with carbon tetrachloride and recovering the extracted fat, the following method was adopted for routine analysis: At room temperature, 50 ml. of  $\text{CCl}_4$  is added to 10 g. of dry milk in a 125-ml. glass-stoppered Erlenmeyer flask. The mixture is then shaken for 30 min. on a Burrell wrist-action shaker at an intensity setting of 5. Filtering follows immediately through an 18.5-cm. (Reeve Angel No. 812) folded filter. A 25-ml. aliquot of the filtrate is transferred to an aluminum evaporating dish and the solvent removed first on a hot plate at  $135^\circ\text{C}$ ., then in a  $100^\circ\text{C}$ . vacuum oven. The fat residue is weighed and the weight multiplied by two. The free fat is expressed as a percentage of the total fat in the dry milk.

The reproducibility of this method was found to be 0.45% of the absolute value (95% confidence interval). Variation of temperature of extraction, amount of  $\text{CCl}_4$ , shaking time, and standing time before filtering did not affect the reproducibility for any given set of conditions. No sharp borderline was found between the free fat and the rest of the fat. The amount of free fat extracted increased with shaking time. A 30-min. period was chosen, since the rate of extraction after 30 min. was much less than in the region from 0 to 30 min. shaking time.

*Determination of fat rise under centrifugal force.* The method is based on the extent of fat rise in 50 ml. of reconstituted milk by centrifuging and recovering the top 5 ml. Errors due to analysis of an aliquot of an inhomogeneous top layer are prevented by using the whole top layer. First, acetone is added to denature the protein, then the fat is extracted with petroleum ether and weighed after removal of the solvent and drying. The procedure adopted is as follows: 15 g. of dry milk are added to 50 ml. of water in a 170-ml. separatory funnel. After shaking a few strokes to disperse the dry milk, another 50 ml. of water is added and the mixture shaken for 2 min. It is left for 5 min. and drained

slowly from the funnel to remove the scum. Tubes with 50 ml. of the reconstituted milk were centrifuged for from 10 to 40 min. at 1,000 and 2,000 r.p.m. in an International<sup>1</sup> centrifuge with a 16-in. diameter head. Thirty minutes at 1,000 r.p.m. is suitable for routine examination. The lower 45 ml. is carefully removed with a thin siphon tube. The top 5 ml. is first mixed with 10 ml. of acetone and then extracted with 10 ml. of petroleum ether. After centrifuging for 5 min. at 2,000 r.p.m., the top layer is transferred to a Mojonnier fat dish. Extraction is repeated twice, the last time with the addition of 2 ml. of acetone to the petroleum ether to facilitate separation. The fat residue is weighed after removal of solvent by the Mojonnier procedure as described under "free fat," and expressed as a percentage of the total fat in the reconstituted milk.

*Further analysis.* The dispersibility was determined by the method of Sinnamon *et al.* (14), using a stirring time of 1 min. in water at 40° F. The dried product was tempered to room temperature before dispersing. Total solids in the filtrate were determined by the Mojonnier procedure. Moisture was determined by the toluene distillation method (1), and  $\alpha$ -lactose according to Sharp and Doob's procedure (12). Observations of fat globule sizes were made by the Farrell index method (4).

#### EXPERIMENTAL AND RESULTS

*Preparation of foam-dried whole milk.* Mixed herd milk was standardized to a solids-not-fat to fat ratio of 2.75 and preheated in a hot water jacketed vat under continuous agitation at 145° F. for 30 min. The milk was condensed at 115° F. to 16° Bé and standardized to 50% T.S. The concentrate was homogenized at 135° F. in a two-stage Manton-Gaulin<sup>1</sup> homogenizer, as specified in each experiment. Nitrogen was incorporated in the concentrate during homogenization, or during a second homogenization at a lower pressure following the first homogenization, to obtain good foaming. Incorporation during homogenization was achieved by adding approximately 20 ml. of nitrogen (at room temperature and 1 atm. pressure) per liter of concentrate through a fine capillary in the line immediately prior to the homogenizer. Homogenization in the presence of nitrogen may decrease homogenization effectiveness. The fat was homogenized without the presence of nitrogen by incorporating the nitrogen during a second homogenization. The concentrate was cooled to 50° F., foamed in a vacuum shelf drier, and dried for 3 hr. at below 1-mm. pressure to about 2.5–3.0% moisture. The temperature of the product was kept below 120° F. Details of the drying procedure were described by Sinnamon *et al.* (14). Dry foams were comminuted through a 20-mesh screen.

#### FACTORS AFFECTING FREE-FAT

1. *Effect of homogenization.* In Trial A, concentrated milk, prepared as above described, was divided into three lots. These lots were homogenized at

<sup>1</sup>The use of trade names is for the purpose of identification only, and does not imply endorsement of the product or its manufacturer by the U. S. Department of Agriculture.

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0, 2,000, and 4,000 p.s.i. on the first stage and 500 p.s.i. on the second stage for the latter two lots. Nitrogen was incorporated in a second homogenization at 2,000 and 500 p.s.i., for the first and second stages, respectively.

In Trial B, two lots of the same concentrate were homogenized at 1,500 and 3,500 p.s.i. on the first stage, the second-stage pressure was 1,000 p.s.i., and the nitrogen was incorporated during homogenization. The results of both trials (Curves A and B, Figure 1) show a decrease in free fat with increase in homogenization pressure, which is in agreement with the results of Holm *et al.* (5). However, the over-all effect of homogenization was rather small, being about 1 to 2% difference in free fat per 1,000 p.s.i. pressure difference. The effect was probably lowered by an equalizing tendency of the nitrogen incorpora-

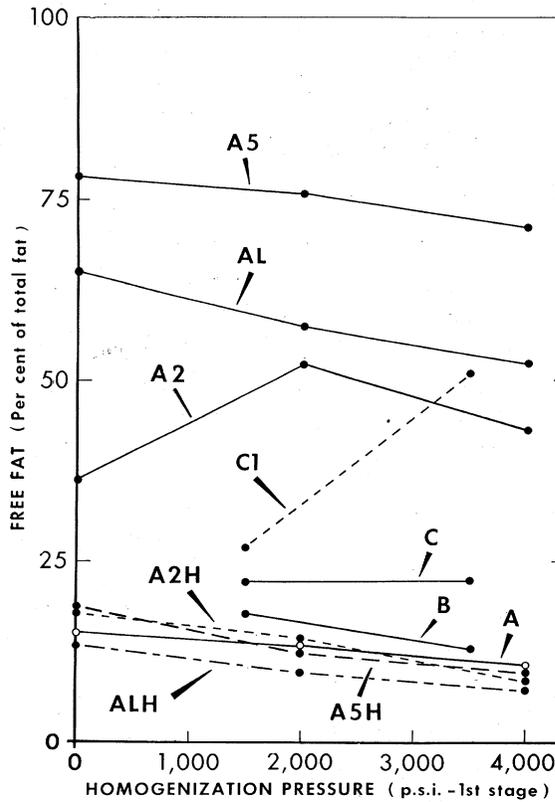


Fig. 1. Effect of homogenization pressure and temperature-time treatment of the concentrate on free fat in foam-dried whole milk.

- A = Cooled instantly in pans at 50° F.
- A2 = A concentrates held two days at 40° F.
- A5 = A concentrates held five days at 40° F.
- AL = A concentrates cooled rapidly on surface cooler to 50° F., seeded with 1% lactose, and dried immediately.
- A2H, A5H, ALH = A2, A5, AL concentrates heated to 140° F., held for 5 min., and cooled instantly in pans at 50° F.
- B = Cooled rapidly on surface cooler to 50° F.
- C = Slow cooling in ice-water under stirring.
- C1 = C concentrates held one day at 40° F.

tion procedure. Experiments, as in Trial B, where the nitrogen was incorporated during the only cycle through the homogenizer, resulted in excessive knocking when pressures above 1,500 p.s.i. were used. This produced less effective homogenization and was the reason for using a second cycle through the homogenizer for nitrogen incorporation in Trial A. An experiment without incorporation of nitrogen showed a larger effect (from 3 to 10% free fat per 1,000 p.s.i. pressure difference), but these results were of little value because of poor foams and less dispersible products.

The effect of homogenization indicates that superior homogenization will reduce free fat to a lower value. Superior homogenization can be obtained by repeated homogenization at high pressure or centrifugal removal of large fat globules (16). Centrifugal removal of large globules was tried for a 50% T.S. concentrate homogenized at 3,500 and 1,000 p.s.i., for first and second stages, respectively, using a De Laval<sup>1</sup> separator. After separating twice, which resulted in removal of 6% of the fat as cream, the free fat in the dry product was reduced to about three-fourths of that in a control standardized to the same composition.

2. *The effect of heating and aging of the concentrate, and lactose crystallization.* These factors are all inter-related, since the temperature and age of the concentrate greatly affect the degree of lactose crystallization. Each of the three lots in Trial A was further divided into three sub-lots, AL, A2, and A5. AL was cooled on a surface cooler, seeded with 1% lactose, and dried immediately. Sub-lots A2 and A5 were cooled on a surface cooler, then held two and five days, respectively, at 40° F. and dried without further treatment. The marked increase in free fat resulting from these treatments at all homogenization pressures is shown by comparing Curves A2, AL, and A5 with Curve A (in Figure 1). The complete reversibility of the effect of added lactose crystals, and aging of the concentrate, is shown by Curves ALH, A2H, and A5H. These concentrates were the same as AL, A2, and A5, respectively, except they were heated to 140° F., held for 5 min., and cooled immediately to 50° F. just prior to foam-drying. These results strongly indicate a relationship between crystallized lactose in the concentrate and free fat in the dried milk. A five-day-old concentrate (A5, Figure 1) contained 74% of the lactose in  $\alpha$ -form, as determined by Sharp and Doob's (12) method. Heating (A5H, Figure 1) lowered this percentage to 37. The equilibrium level without crystallization is 37%.

The effect of lactose crystallization on free fat can supersede the effect of homogenization, as demonstrated by Trial C in Figure 1. A batch of concentrate, prepared as described, was divided into two lots. These lots were homogenized at 1,500 and 3,500 p.s.i. on the first stage, and 1,000 p.s.i. on the second stage, and cooled slowly. Each of the two lots was divided into two sub-lots, C and C1. Sub-lots C were dried immediately, C1 after one day of holding at 40° F. No significant increase in  $\alpha$ -lactose was found in these samples, except for the sample C1, homogenized at 3,500 p.s.i., which had 46% of the lactose in the alpha form. Correspondingly, the free fat was highest in this same sample, which reversed the regular sequence: lower free fat with higher homogeni-

zation pressure. The same trend was already present in the C samples, as indicated by Curve C, which was almost horizontal instead of parallel to Curve B. Curve A2 (Figure 1) shows a similar behavior below 2,000 p.s.i. Lactose crystallization apparently had proceeded more in some samples by chance, due to conditions such as the presence of nuclei etc.

The effect of rate of cooling of the concentrate on free fat content is indicated by the results of Samples A, B, and C (in Figure 1). Slower cooling, which favors lactose crystallization, corresponded with higher free fat. No significant difference in  $\alpha$ -lactose was found in these samples by Sharp and Doob's method.

Figure 2 shows the relationship between excess  $\alpha$ -lactose and free fat, established with data collected from 12 samples, processed identically except for difference in seeding with lactose. A regression line of  $Y = 18.29 + 2.50 X$  was obtained for this relationship ( $Y =$  per cent free fat and  $X =$  excess  $\alpha$ -lactose), with a coefficient of correlation of  $+0.82$  ( $P = <0.01$ ). The standard error is 9.27. The excess of  $\alpha$ -lactose is expressed as the difference between the percentage of the lactose in the  $\alpha$ -form and the equilibrium value (37%): The regression coefficient, 2.50, shows that relatively small increases in excess  $\alpha$ -lactose resulted in large increases in free fat. The excess  $\alpha$ -lactose, however, may not represent the crystallized lactose, since Sharp and Doob's method yields the total  $\alpha$ -lactose. If crystallization proceeded more rapidly than the shift from  $\beta$ - to  $\alpha$ -lactose while holding the concentrate, or during drying, the amount of crystallized lactose would be greater than the excess  $\alpha$ -lactose (as shown in Figure 2). It will be noted that the free fat content for 0.0% of excess  $\alpha$ -lactose was 18.29, which is considerably higher than the minimum values from Figure 1. This indicates that other factors, including an indeterminate amount of crystallized

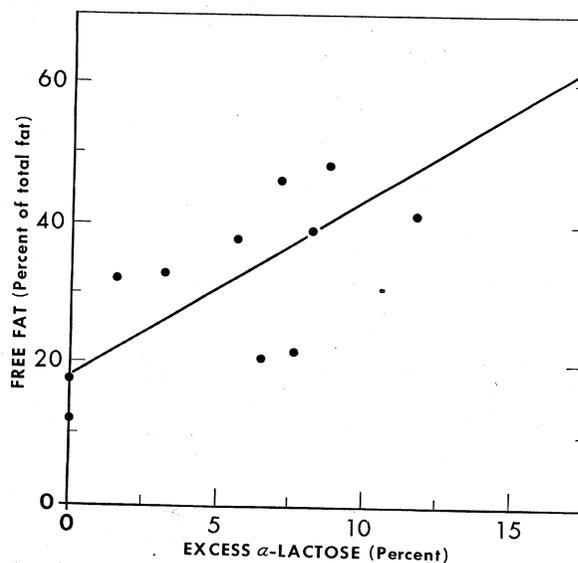


Fig. 2. Relationship between excess  $\alpha$ -lactose and free fat.

lactose, also may affect the free fat content. Choi (2) pointed out that variations in free fat may be affected by changes in the proteins.

3. *The effect of particle size.* A concentrated milk was homogenized at 3,500 and 1,000 p.s.i. for first and second stages, respectively. Nitrogen was incorporated during a second cycle through the homogenizer. The dry foam was comminuted through an 8-mesh screen, mixed to a uniform batch, and divided into Lots 1, 2, 3, and 4 (see Table 1). The first lot was further divided into three fractions as follows: Fraction 1 was used as such. The remainder was sieved on a 20-mesh screen into coarse and fine fractions, 1c and 1f, respectively. The second lot was comminuted through a 20-mesh screen and then further divided into three fractions. One-third (Fraction 2) was used as such, and the remainder sieved on a 40-mesh screen into coarse and fine fractions, 2c and 2f, respectively. Similar fractions were obtained with the third lot, using 40- and 60-mesh screens. The fourth lot was comminuted through a 60-mesh screen to give Fraction 4. The data in Table 1 shows an increase in free fat with decrease in particle size (and increase in surface area). These results indicate that free fat is present on the surface of the particles. This was further evidenced by fluorescence microscopy (7) of dry milk comminuted through a 60-mesh screen. The fat appeared as a layer around the particles.

The bulk density (tamped to a constant volume) increased with decrease in particle size resulting from comminution through a higher mesh screen [compare Fractions 1, 2, 3, and 4 (Table 1)]. Fine fractions varied somewhat in bulk density as compared with the original fraction in each series (compare 1 and 1f, 2 and 2f, 3 and 3f). Fractions obtained by sieving were much lower in bulk density than fractions obtained by comminuting through screens of the same size (1f-2, 2f-3, 3f-4). Some difference in form or size distribution of the particles, not reflected in the free fat, must be responsible. The bulk densities of the coarse fractions obtained by sieving in each series were not significantly different from those of the original fractions (compare 1c and 1, 2c and 2, 3c and 3, respectively). Coarse fractions, representing about 70% of the total, were readily dispersible. The dispersibilities for Fractions 2 (the standard product), 2c, and 3c are 88.5, 93.7, and 94.2%, respectively.

TABLE 1  
Free fat and bulk density of dry whole milk fractions differing in particle size

Fraction No.	Fractionating procedure		Bulk density ( <i>g. per ml.</i> )		Free fat (% of total fat)
	Comminuted mesh	Sieved mesh			
			Loose	Tamped	
1	8	no	0.09	0.12	12.6
1c	8	20 coarse	0.09	0.12	7.7
1f	8	20 fine	0.11	0.14	16.7
2	20	no	0.22	0.32	16.7
2c	20	40 coarse	0.25	0.31	12.7
2f	20	40 fine	0.20	0.39	25.2
3	40	no	0.31	0.45	23.7
3c	40	60 coarse	0.37	0.48	17.4
3f	40	60 fine	0.22	0.37	34.7
4	60	no	0.31	0.48	37.6

Utilization of coarse fractions may open a way to obtain good products with higher bulk density (compare 3c and 2, Table 1). The procedure would be comminution through a screen finer than 20 mesh and removal of the finest particles by sieving on a yet finer screen. Sinnamon *et al.* (14) found the dispersibility of powders comminuted through a 40-mesh screen to be lower than those comminuted through a 20-mesh screen.

*The effect of comminution temperature.* In the standard procedure the foam was comminuted at about 120° F. as it came from the drier with the fat in the liquid state. Four drying runs were made in which half of the foam was held overnight at 40° F. and then comminuted at 40° F. The other half was comminuted immediately by the standard (control) procedure. The free fat and dispersibility of the products comminuted cold are expressed as percentages of the corresponding control values. The free fat varied from 92 to 115%, with an average of 103%; the dispersibility varied from 105–114%, with an average of 108%. The data do not indicate a trend for the free fat, but a slight improvement in dispersibility resulted from the low temperature comminution.

*Melting point of the fat.* Recombined whole milk concentrates with 50% T.S. were made by homogenizing mixtures of fat and skim milk concentrate three times at 140° F., using 4,000 and 500 p.s.i. for the first and second stages, respectively. Dispersion of the fat as determined by microscopic observation was similar to that obtained to that obtained by homogenization of whole milk concentrates of 50% T.S. in the usual manner. Three fats were incorporated: whole milk fat, a fraction which was liquid at room temperature, and a more solid fraction remaining from milk fat, after removal of the liquid fraction. The resulting concentrates were processed by the standard procedure. The dry products had properties similar to those of regular foam-dried whole milk. The free fat contents and dispersibilities of the products are expressed as percentages of the values of the corresponding dry milks with whole milk fat. Five sets of dry milks were compared, each with a whole milk fat control and samples with liquid and solid milk fat fractions. The free fat content of the samples containing liquid fat varied from 71 to 154% (average, 111%) of the control; the dispersibilities from 101 to 106% (average 103%). The free fat in those containing solid fat varied from 86 to 166% (average, 132%) of the control; the dispersibilities from 95–101%, with an average of 98%. The data do not indicate trends for the free fat. The dispersibility of the samples containing liquid fat are slightly higher than those containing solid fat. This effect is similar to that reported by Stone *et al.* (15), who showed that milk powders are more easily dispersible at temperatures above the melting point of the fat.

*Effect of drying temperature.* Drying in the vacuum shelf drier for 6 hr. at 80° F., 4 hr. at 120° F., or 3 hr. at 160° F. shelf temperature did not change the amount of free fat. Drying at 160° F. caused some decrease in dispersibility.

*Effect of storage.* The initial free fat and dispersibility data of 27 air-packed samples were compared with the data obtained after from 3 to 5 mo. of storage

at room temperature. The samples included the variations in processing previously discussed. The results after storage, expressed as percentages of the original values, varied from 80–115 (average of 105) for the free fat, and from 87 to 109 (average of 102) for the dispersibility. No trends due to storage are indicated by these data. In microscopic observations of Whittenberger (17), no increase in free fat was observed on storage.

Although the experiments reported herein were not designed for taste analysis, some preliminary organoleptic results were obtained. The stored samples developed tallowy flavors. Improved keeping quality was indicated for samples high in crystallized lactose (also high in free fat content), and for samples prepared from aged or lactose-seeded concentrates which were reheated to 140° F. just prior to drying. The effect of lactose is similar to the results reported by Sharp (11). The organoleptic results were obtained by five judges, using a paired comparison preference method of taste testing. Further studies of the effect of heat treatment, and of lactose crystallization in the concentrate, are currently in progress.

*Relationship between free fat and dispersibility.* The relationship between free fat and dispersibility for 92 samples of foam-dried whole milk is shown (Figure 3). The equation of the regression line is  $Y = 97.26 - 0.111X$ , the correlation coefficient is  $-.54$ , and the standard error of estimate is  $\pm 2.54$  ( $P = < 0.01$ ). The data show that the dispersibility decreased only about 0.11% for each 1.0% increase in free fat. Although the data for this group of samples were combined from many experiments which included variations in several processing steps (heat treatment, homogenization pressure, lactose crystallization, particle size, etc.), the regression coefficient is significant at the .01 level ( $P = < .01$ ).

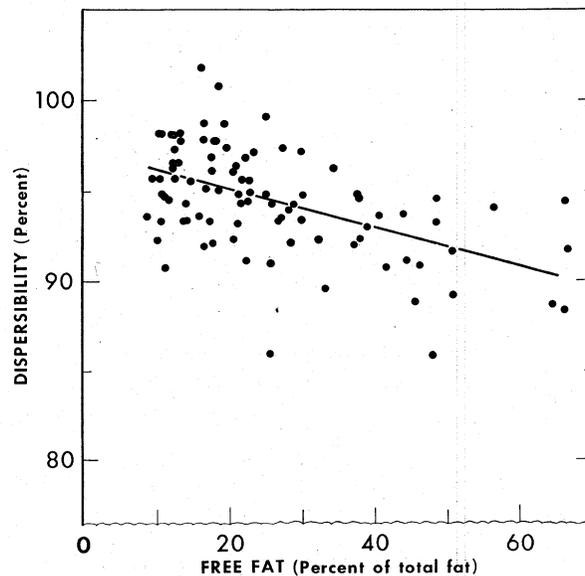


Fig. 3. Relationship between free fat and dispersibility.

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Linear regression analysis of 12 of the samples, from experiments in which the only variable was in seeding with lactose, gave a regression equation of  $Y = 97.19 - 0.118X$  (approximately the same as for the combined data), a correlation coefficient of  $-0.71$ , and a standard error of  $\pm 1.91$  ( $P = < .05$ ).

*Fat rise under centrifugal force at different free fat levels.* Fat rise was determined in five samples of reconstituted foam-dried milks selected for free fat difference. Results (Figure 4) were about the same at 1,000 r.p.m., except for Sample 3, which showed much more fat rise. At 2,000 r.p.m. (prime series), the extent of fat rise correlated with the size of the fat globules (homogenization effectiveness) rather than with difference in free fat. The data (Figure 4) show that the fat rise under centrifugal force after reconstitution is not affected significantly by the difference in free fat from 26.7–50.6%, as presented in Samples 1, 2, 4, and 5. Variation in this range is probably caused by differences in

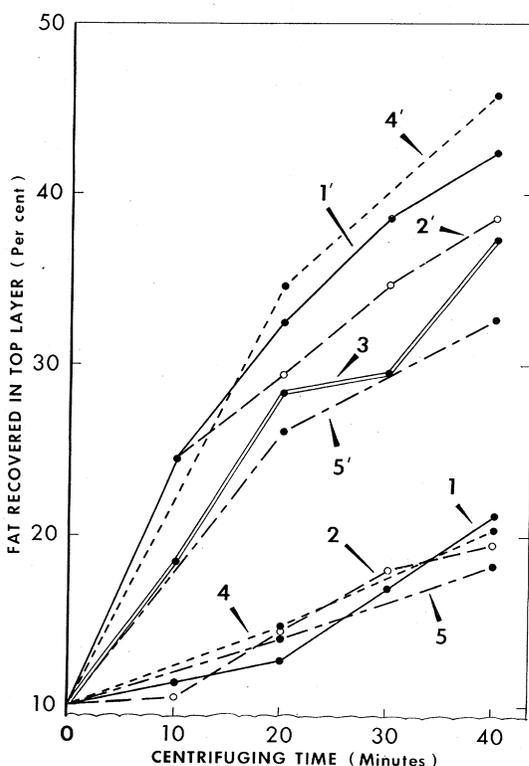


Fig. 4. Fat rise in reconstituted foam-dried milks.

No.	Homo. pressure (p.s.i.)	Free fat (%)
1	2,500	29.8
2	2,500	44.2
3	2,500	92.0
4	1,500	26.7
5	3,500	50.6

Plain series centrifuged at 1,000 r.p.m.

Prime series centrifuged at 2,000 r.p.m.

the dry particles, such as the presence or absence of cracks affecting solvent penetration. At very high free fat levels the fat rise under centrifugal force shows major increase (Sample 3), indicating a change in the state of the fat.

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