

A long-range research program was recently initiated at this Laboratory, with the ultimate goal of developing a dried whole milk of easy dispersibility, good flavor, and storage stability. As presently conceived, this program will be carried forward in three phases: The first phase will be concerned with the preparation by batch methods of a dried whole milk which is initially easy to disperse and of good flavor. The second phase will include a study of the storage stability of the material and factors which influence stability. The third phase will be a translation of the essential processing conditions to a commercially feasible operation. The purpose of this paper, which deals with the first phase of the program, is to report the properties of whole milk dried in a new physical form and possessing excellent flavor and easy dispersibility in ice water. The processing variables found to be essential in its preparation are discussed.

CHOICE OF THE DRYING METHOD

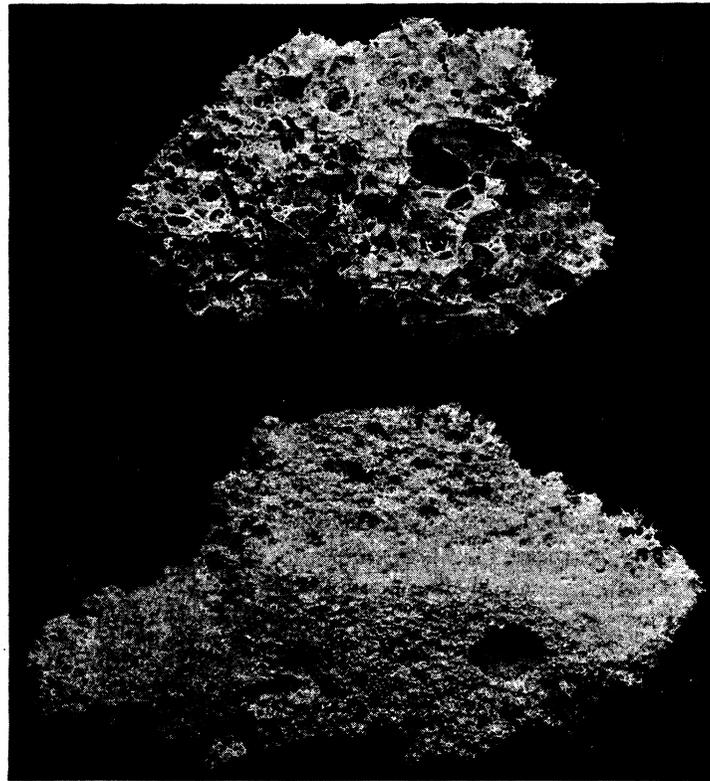
Puff-drying methods were investigated, because the authors believe them to be superior to spray- or roller-drying. Puff-drying has been successfully applied to heat-sensitive citrus juices and other food products in both batch and continuous processes (2-5, 7). Therefore, a systematic engineering study was made of the factors responsible for producing a product that would be easy to disperse and have good flavor.

In general, puff-drying may be defined as the formation of a highly expanded, sponge-like structure of dried material from a thin film of liquid concentrate, under conditions of high vacuum and low temperature. The product would be expected to disperse rapidly because of its large surface area per unit weight, and to possess natural flavor because of the high-vacuum, low-temperature drying conditions employed. As applied to whole milk, drying at low temperatures

would also be expected to inhibit protein destabilization, and dehydrating at low oxygen concentrations at low temperature would preclude atmospheric oxidation.

BASIC CONSIDERATIONS IN PUFF-DRYING WHOLE MILK

Puffed structures can be developed by various devices and, for some materials, such as those containing large amounts of sugars, the type of structure may not be of prime importance for rapid dispersibility. With whole milk, however, the structure has been found to influence markedly the dispersibility rate of the dried product. Two possible structures are illustrated (Figure 1). The puffed form on the top was developed by the evolution of water-vapor from a deaerated concentrate. This is characterized by large, nonuniform bubbles and a preponderance of unpuffed, intercellular material. Product obtained from such a form disperses very poorly compared to that shown (bottom of Figure 1). The latter was formed by the expansion of entrained gas of low solubility in the concentrated milk, and is characterized by small, uniform bubbles and a minimum of unexpanded intercellular material. To differentiate, the latter is hereafter referred to as a foam.



The mere use of an entrained gas does not insure a form that will disperse readily. In order for entrained gases to be effective in forming a fine-grained foam, the concentrate must be held at a low temperature during the initial drying stage. At temperatures above 55° F., and under the vacuums necessary for foaming, the concentrate will boil before it has expanded sufficiently, and boiling at this point will remove the gas from the unexpanded material. The dried structure will then be similar to a puff formed by water-vapor from a deaerated concentrate. Initial experiments using only dissolved CO₂ or N₂O indicate that these gases, because of their high solubility, give structures similar to the water-vapor type.

When the absolute pressure within the drier reaches the vapor pressure of the concentrate, vigorous agitation of the expanded structure results from the flashing of water-vapor. If the desirable form is to survive, it must be rigid enough to withstand this action. Here again, low temperatures are desirable because of the added viscosity. High solids content is another factor which increases the viscosity of the concentrate, and it has been found that materials containing up to about 50% solids can be readily dried as a foam. Beyond this concentration a gel forms which becomes difficult to spread evenly for drying.

The conditions essential for producing a material of good dispersibility have been determined by experiments in a pilot-plant vacuum shelf drier. Exploratory studies conducted cooperatively with the Chain-Belt Company² of Milwaukee, Wisconsin, have shown, moreover, that these essential conditions can most probably be translated to a continuous method and thus, that foam-drying of whole milk may have a future in commerce. A public service patent is pending covering these principles.

PREPARATION OF THE DRIED WHOLE MILK

Fresh pasteurized, homogenized milk was obtained from a local dairy for these studies. It had been homogenized at 145° F. and 2,500 p.s.i. and then pasteurized at 162° F. for 16 seconds. It contained 3.6 to 3.7% fat and about 8.7% solids-not-fat. The milk was first concentrated to from 47 to 50% total solids at a batch temperature of 85 to 100° F. in a high vacuum, falling-film evaporator. It was then heated to 135° F. and homogenized, first at 4,000 p.s.i. and then at 500 p.s.i., through a single-stage homogenizer of the pulsator type. Immediately prior to homogenization, nitrogen was bubbled through the concentrate from a fritted-glass sparger dispersing entrained gas through the material. The concentrated milk was then flowed over stainless steel drying pans to an average depth of 1/16-inch, chilled to 55° F. or below, and dried as a foam in a vacuum shelf drier. The resulting dried mass was crushed lightly through stainless steel screens. A typical drying cycle is illustrated (Figure 2).

DRY WHOLE MILK. I.

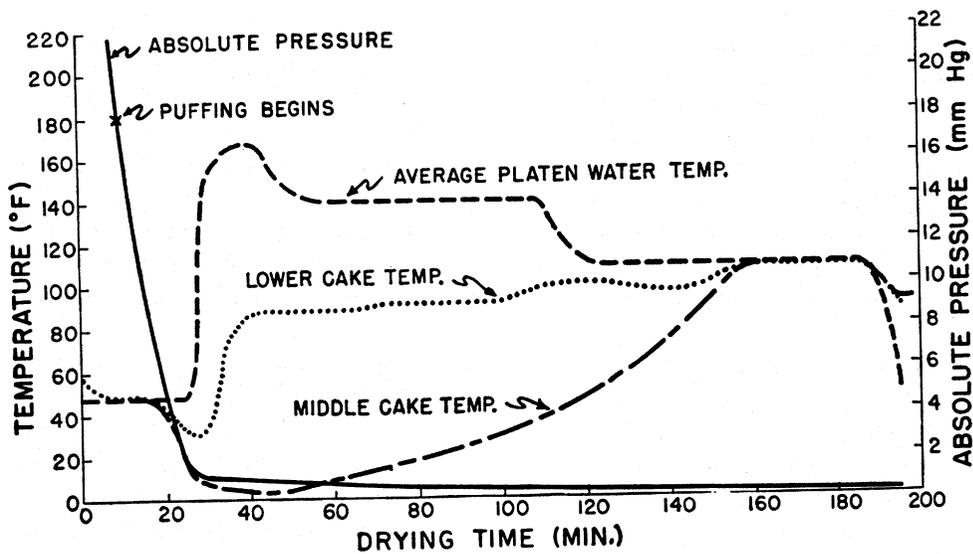


FIG. 2. Vacuum shelf drying cycle for whole milk.

It has been found that variations in foam structure were possible, depending on the rate of vacuum application. For instance, when the pressure inside the drying chamber was reduced from 20 to 2 mm. in five minutes, the concentrate expanded rapidly to about 24 times its original volume, and then became sufficiently rigid to prevent further expansion or collapse. The resulting structure was composed of tiny bubbles approximately 1/16-inch in diameter and had a relatively large proportion of unexpanded intercellular material. When the drying chamber pressure was reduced from 20 to 2 mm. in about 20 minutes, on the other hand, the concentrate expanded slowly to a volumetric increase of from 32- to 96-fold before it stabilized. Bubble sizes were larger in these structures, and the proportion of unexpanded intercellular material was substantially reduced. Where slow-vacuum application was used, variations in volume appeared to be caused by the viscosity of the concentrate. In general, concentrates of higher viscosity contained more entrained gas and expanded to a greater extent. Where vacuum was applied rapidly, on the other hand, concentrates of different viscosities all expanded to about the same degree, indicating that in those cases the rapid application of vacuum was the controlling variable.

RATE-OF-DISPERSIBILITY TEST

Because the structure could be altered by varying the degree of expansion of the concentrate, a reliable method was required for determining the rates at which the several products would disperse. To fill this need, the ease-of-dispersion method of Stone *et al.* (6) was studied and modified. In the original method, 15 gm. of material were added to 90 ml. of water at from 70 to 75° F. in a 250-ml. beaker. The mixture was stirred by hand with a teaspoon for a designated time-interval, dispersing as much of the material as possible without

splashing the dried milk or water out of the beaker. After stirring, the mixture was filtered through a coarse (40 to 60 microns) fritted-glass Buchner funnel, with the aid of about 16 inches of vacuum. The filtrate was then diluted to 200 ml. and grams of total solids were determined in a 20-ml. aliquot. When multiplied by ten, this weight represented the dispersion value for the corresponding stirring time.

Several modifications in this test were made, in an effort to more nearly standardize the filtration step from one material to another, to detect smaller differences among dried milks, and to improve the precision of the method. In the original test it was found that some materials bound the fritted-glass filter, whereas others did not. This condition, in turn, varied the holdup time in filtering from a few seconds for readily dispersible materials to more than two minutes for materials of poorer dispersibility. This wide variation is obviously undesirable. Two modifications in the procedure brought the filtration time for all materials to within about three to 15 seconds. The first was the use of scalper filters, consisting of cones of 100-mesh screen (140 microns) and 150-mesh screen (103 microns), just ahead of the vacuum filter to eliminate filter binding. The second provided that instead of collecting all the filtrate for analysis, only 35 ml. should be used.

Collecting only the first 35 ml. of filtrate for analysis improved the precision of the method in two other ways. First, it was questionable that all of the filtrate could have been collected, and second, the initial fraction of the filtrate was probably more representative of the solution immediately after stirring than the total filtrate. Thus, by removing only the minimum of filtrate necessary for analysis, the solution was separated from the undispersed material more quickly, and any further dispersing of solids after stirring was minimized.

Danger of splashing during stirring was reduced by substituting a 300-ml. tall-form beaker for the 250-ml. beaker used in the original test.

In order to detect smaller differences among dried milks, the filter screens and beaker were chilled in ice water prior to use and the temperature of the stirring water was reduced to 38° F. The variation in dispersibility caused by changing the water temperature from 75 to 38° F. is illustrated (Figure 3). For this experiment, materials from the same respective batches were used. A comparison of the curves at the two temperatures for either material illustrates the effect of temperature on dispersibility rate, and a comparison of the curves for the two materials at 38° F. and then at 75° F. illustrates that smaller differences in dispersibility can be detected by using water at 38° F.

In summation, the rate-of-dispersibility test as used in this work was as follows: Fifteen grams of powder at room temperature were added to 90 ml. of water at 38° F. in a 300-ml. tall-form beaker. The mixture was stirred with a teaspoon for a predetermined time-interval, dispersing as much of the powder as possible without splashing. The mixture was then poured through a 100-mesh cone contained in a glass funnel, dropped through a 150-mesh cone contained in another glass funnel, and finally dropped onto a coarse fritted-glass Buchner

DRY WHOLE MILK. I.

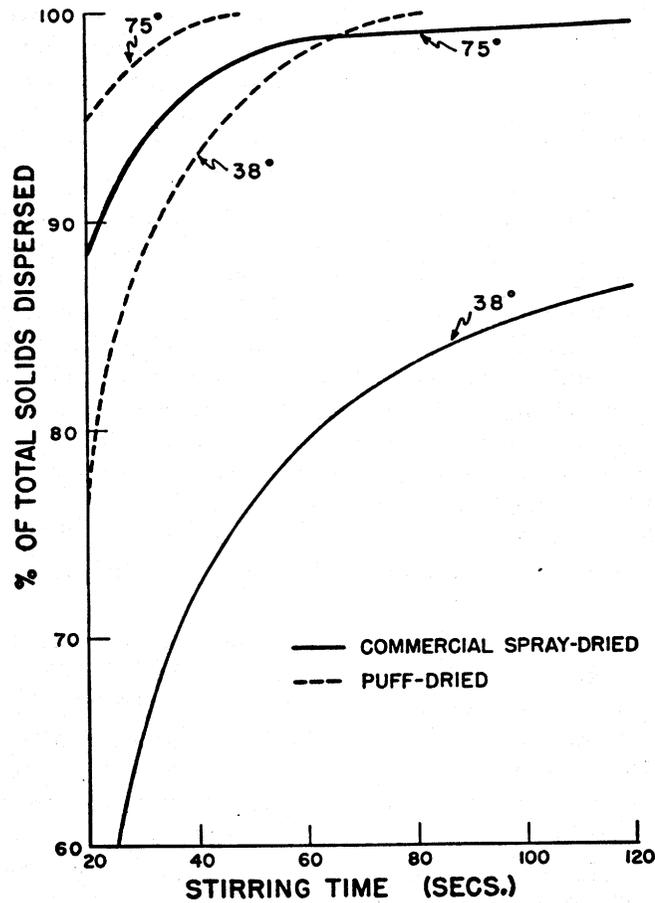


Fig. 3. Dispersibility rates for dried whole milk and effect of water temperature.

suction filter with about 16 inches of vacuum on the filtering flask. After 35 ml. of filtrate had been collected, the filtration was discontinued. An aliquot of 25 ml. was added to an aluminum pan of known weight and concentrated to a plastic state in a Brabender Moisture Tester,² using an air current at 275° F. The material was then brought to dryness overnight, in a vacuum oven operating at about 194° F., 0.4-inch absolute pressure, and with a bleed of dry air. Percentage moisture in the filtrate was then determined, and dispersibility was calculated as the percentage of the total solids in the dried milk which had dispersed sufficiently in the given time to pass through the filters. This calculation was carried out as follows:

- Let x = % solids of the dried milk
- y = % moisture of the filtrate
- F = Grams of filterable solution (assuming the 35 ml. collected was representative)

$G = \text{Grams of solids contained in } F$
 $T = \text{Grams of solids m.f.b in the 15-gram sample}$
 $S = \% \text{ of the total solids in the dried milk that had dispersed}$
 Then, $F = (90/y) 100$
 $G = F - 90$
 and $T = \frac{15 \times}{100}$
 Thus, $S = \frac{G}{T} \times 100$

Six stirring times were employed and each stirring time was run in triplicate. The filtering system is sketched (Figure 4).

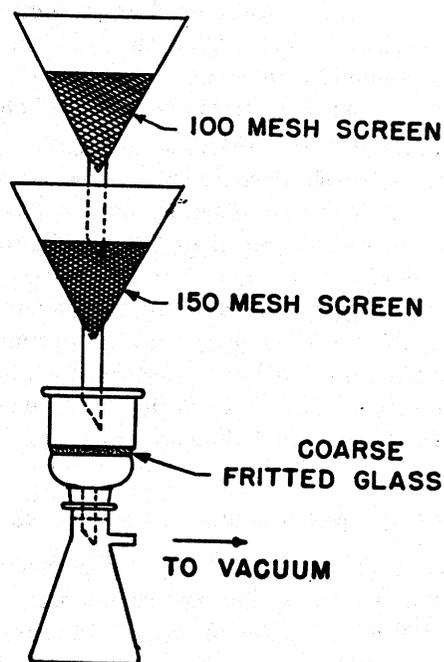


Fig. 4. Filtering system for dispersibility-rate test.

In an experiment designed to test the precision of the dispersibility-rate procedure used in this work, three different dried milks were run through the dispersibility-rate test in random order, using stirring times of 20, 40, 60, 80, 100, and 120 seconds. The data (see Table 1) for solids content of the filtrates were analyzed statistically, using the analysis of variance technique.

The coefficient of variability for this experiment was 1.88%. Differences between treatment means of 0.22% solids were significant at the 0.01 level of probability. Expressed as percentage of the total solids in the dried milk which have dispersed, a mean difference of 0.22% solids in the filtrate corresponds to plus or minus 1.8% total solids at the point of maximum dispersion. Thus, this is a precise method for measuring rates of dispersibility.

TABLE 1

Solids contents of dried whole milk filtrates, as measure of dispersibility rate

Material.....	A	B	C
Stirring time (sec.)	Total solids*		
20	10.03	10.31	10.26
40	12.03	12.27	12.47
60	12.46	13.11	13.11
80	12.67	13.45	13.50
100	13.13	13.51	13.67
120	12.97	13.87	13.65

* Mean of three replicates.

DISPERSIBILITY RATES OF FOAM-DRIED WHOLE MILKS

Batches of material of the various types of foam structure developed by entrained gas were crushed lightly through 20-mesh (0.034-inch) and 40-mesh (0.015-inch) screens, and dispersibility rates were determined, using the modified test described above. Data from these tests are shown (Table 2).

Statistical comparisons of the data were made, using the analysis of variance technique. These showed that 20-mesh screening was superior to 40-mesh in all cases, that there was no real difference apparent between 32- and 72-fold expansion, that 96-fold expansion was poorer than 32- and 72-folds, that 24-fold expansion was poorer than the other three, and that differences caused by degree of expansion, screen size, and stirring time were all significant beyond the 0.01 probability level. In essence, these results show that the optimum dispersibility rate for foam-dried material is unaffected by the degree of expansion of the concentrate over the wide range from 32-fold to at least 72-fold; that there are, however, upper and lower limits, and that 20-mesh screening is more desirable than 40-mesh.

BULK DENSITIES OF FOAM-DRIED WHOLE MILKS

Bulk densities of the materials listed in Table 2 were determined, in order to exhibit the effects of volumetric expansion and screen size. These data were found by carefully placing 100 ml. of material in a 250-ml. graduated cylinder, weighing the material, and calculating its bulk density untamped. Then the graduate was tapped briskly upon a table top, until the material reached a con-

TABLE 2

Mean percentage dispersibilities of dried whole milks

Volumetric expansion (fold).....	24		32		72		96	
Screen size (mesh).....	20	40	20	40	20	40	20	40
Stirring time (sec.)	Total solids							
10	41.0	20.7	54.8	35.6	49.0	24.6	—	—
25	71.6	59.3	83.6	67.4	80.2	69.8	79.7	70.2
40	83.9	79.3	92.2	81.0	88.7	83.5	91.4	80.0
55	91.5	85.2	97.2	90.2	96.3	90.0	89.2	85.0
70	93.4	89.0	99.2	92.8	96.2	91.0	96.2	92.8
120	97.2	97.2	101.9	99.9	100.1	98.0	98.9	99.5

TABLE 3
Bulk densities of dried whole milks, gm. per cc.

Volumetric expansion (fold).....	24		32		72		96		Commercial product ^a
Screen size (mesh).....	20	40	20	40	20	40	20	40	
Density									
Untamped	0.17	0.19	0.18	0.30	0.15	0.20	0.13	0.28	0.54
Tamped	0.27	0.34	0.25	0.42	0.25	0.35	0.22	0.50	0.72

^a A spray-dried product available commercially.

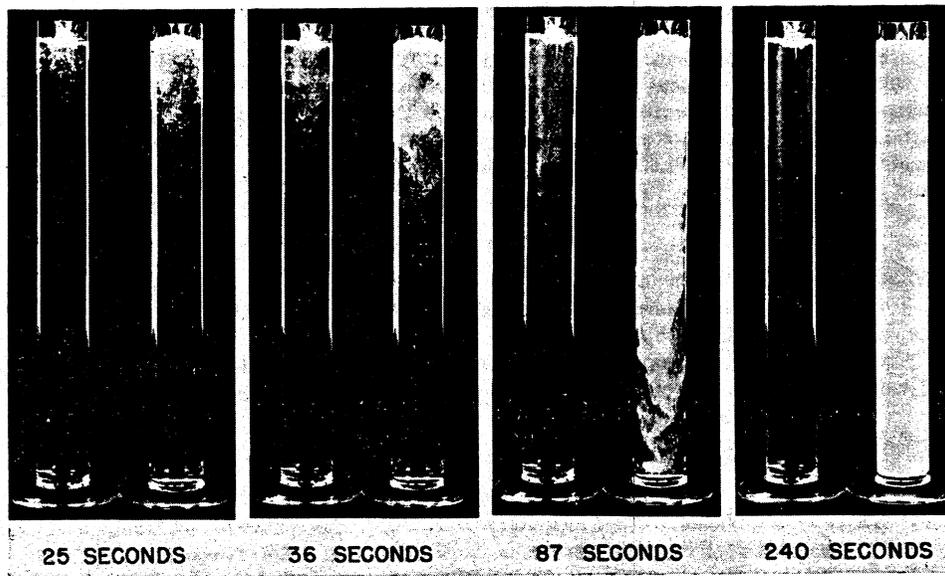
stant level in the graduate, and its new volume was determined as a basis for calculating its bulk density tamped. These data are shown (Table 3).

There is no consistent trend in these data which would indicate a variation of bulk density with degree of expansion of the concentrate, either at 20- or 40-mesh screening. As would be expected, however, the powders run through the 20-mesh screen are more bulky than those run through the 40-mesh screen. Assuming an averaged tamped bulk density of 0.25 gm/cc for 20-mesh, and 0.43 gm/cc for 40-mesh, a unit volume of material would yield 2 and 3½ volumes, respectively, of reconstituted fluid milk.

OTHER CHARACTERISTICS OF FOAM-DRIED WHOLE MILKS

Foam-dried whole milks contained only trace amounts of undispersed material when run through the solubility index test (1).

Comparisons of the whey proteins of fresh whole milk and freshly prepared



—USDA photo by M. C. Audsley

foam-dried material were made. In these studies, the dried milk was reconstituted to three times the concentration of fluid milk, the fat separated by centrifugation, the casein precipitated by acidification, the resulting whey solution dialyzed against a phosphate buffer solution of pH 7.5, and ultracentrifugal inspection carried out at 25° C. The fresh milk was treated similarly, except that concentration of the material was carried out after the casein had been precipitated. Ultracentrifugal patterns showed no difference between the whey proteins of the fresh milk and the foam-dried material.

A striking demonstration of the ease of dispersion of a foam-dried whole milk is given (Figure 5). For this test, two 250-ml. clear, graduated cylinders were filled with water of about 38° F. The water surface of the right-hand graduate was carefully covered with foam-dried material and the other with a spray-dried commercial product. Photographs were taken after 25, 36, 87, and 240 seconds. The superior dispersing characteristics of the foam-dried whole milk are evident.

SUMMARY

A dried whole milk of excellent fresh flavor and rapid dispersibility in cold water has been prepared in a vacuum shelf drier, using special foam-drying techniques. The important variables studied in the batchwise drier can probably be translated to a continuous method.

A modified test for determining the rates at which dried whole milks disperse has been shown to have good precision. Using this test, foam-dried whole milks will completely disperse in water of 38° F. within 100 seconds with manual stirring, and in water of 75° F. within 50 seconds.

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