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FAT SEPARATION IN EVAPORATED MILK

I. HOMOGENIZATION, SEPARATION, AND VISCOSITY TESTS¹

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The rising of fat in evaporated milk on prolonged storage will inevitably occur since the density of the fat particles is lower than that of the concentrated milk plasma in which they are suspended. It has been shown that the velocity of rise of individual fat globules in milk plasma is in good agreement with the velocity as predicated by Stokes' law (5). Troy and Sharp (7) also found the velocity of rise of globule clusters in general agreement with this law. Their observations on clusters were less precise, however, because of inherent complications arising from the irregularities in the shapes of clusters and the occlusion of plasma within the clusters.

Accordingly, it must be expected that the factors controlling fat separation in evaporated milk are: the size of the fat particles, the difference in density between the fat particles and their suspending medium, and the viscosity of the suspending medium.

The size of the fat globules is obviously of first importance, since the velocity of rise is inversely proportional to the square of the globule radius. The importance of smallness is further accentuated through density considerations.

The difference in density between the fat particles and the medium in which they are suspended does not lend itself to direct control in a product of defined composition such as evaporated milk. This factor may, nevertheless, be important to an understanding of differences in fat separation. Insofar as heavier components become associated with the surface of fat globules by adsorption or coagulation, the density of the fat particles is increased. Through this effect the density of the fat particle can approach, equal, or even exceed that of the suspending medium, as smaller globule sizes are obtained, or as the amount of associated material is increased by processing conditions. Smallness of globules is important in this connection because the ratio of area to volume varies in inverse proportion to the radius of the globules.

The density of the suspending medium in evaporated milk is not positively fixed, even though the ratio of solids-not-fat to water is closely controlled. In aqueous solution the density of the three main classes of components of solids-not-fat ranges from about 1.35 for proteins to 1.63 for lactose and 2 to 3 for the

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milk salts. In normal milk the content of salts and lactose is relatively constant. On the other hand, the casein content is directly related to the quantity of butterfat. Since evaporated milk is standardized to a definite ratio of solids-not-fat to fat, the quantity of casein would be greater in evaporated milk made from milk with a high butterfat content than from milk with a low butterfat content. Thus, the evaporated milk with a high casein content would show a low density of the suspending medium. Even though this consideration has theoretical significance, it is not obvious how density should be considered for a nonhomogeneous medium comprising particles that are relatively large.

In connection with the density factor, it is interesting to note that fat separation in unconcentrated, homogenized, sterilized milk is less troublesome than in evaporated milk, presumably because of the lower density of the suspending medium. This condition exists even though the apparent viscosity is somewhat less than in evaporated milk.

The viscosity factor in Stokes' law relates to the viscosity of the suspending medium. For ideal application, the medium should be homogeneous and should exhibit true viscous flow characteristics. Neither of these conditions obtains in the concentrated milk plasma of evaporated milk. In unheated skimmilk, however, fat globules rise in good agreement with Stokes' law; this suspending medium is not perfectly homogeneous, containing casein particles up to $0.780\ \mu$ in diameter (4). As the fat globule size is decreased by homogenization, and as the protein particles are increased in size by coagulation to sizes that may exceed the size of the fat globules, the system becomes more difficult to interpret in relation to Stokes' law.

The "viscosity" of evaporated milk decreases rapidly on vigorous agitation and slowly during quiescent aging. "Age thinning" of evaporated milk is retarded significantly by cool storage temperatures (3). These facts indicate that the suspending medium in evaporated milk does not exhibit true viscous flow characteristics.

Industry practices in seeking to minimize fat separation in evaporated milk have been: (a) efficient homogenization, (b) attainment of maximum viscosity, just short of "graininess," (c) cool storage, and (d) periodic inversion of the cans during storage.

In connection with a study of fat separation in evaporated milk, the following tests were selected or developed, and evaluated.

METHODS

A. A Test to Rate the Effectiveness of Homogenization.

It is generally acknowledged that careful control of the pressure of homogenization alone does not afford assurance that the fat break-up and dispersion will be adequate. The other essential factors, such as valve design and condition, do not lend themselves to brief, descriptive specification and, in fact, have not been fully resolved. Assurance of proper homogenization must, therefore, be sought

through objective tests applied to the homogenized product, rather than through specification of homogenizing conditions.

Microscopic observation of the globule sizes in the homogenized product is the obvious and quick method of checking on the effectiveness of homogenization. This approach, however, lacks strict objectivity; its main use is in yielding a quick general impression; it does not lend itself to the recording of an "effectiveness rating" for later consideration in relation to the various degrees of fat separation that occur in the stored products. Attempts to make this approach objective are laborious, entailing the observation of a number of microscopic fields and complete globule counts by size groups; even then the personal factor enters, and the final rating of the data is arbitrary. It was for these reasons that the effectiveness of homogenization in the U.S.P.H.S. definition for homogenized milk was specified in terms of relative freedom from fat separation on 48-hour quiescent storage.

The evaluation of homogenization by means of a centrifugally accelerated fat separation test has received attention by several manufacturers of evaporated milk. In addition, Snyder and Sommer (6) described a test of this type in which the diluted product was centrifuged in a Babcock milk test bottle and the cream layer was read in terms of the Babcock test scale. A difficulty of this test is that the cream layer frequently lacks distinctness, especially when the fat is lightly pigmented. In the test described below, the fat separation is measured by fat tests to determine the fat enrichment that has occurred in a selected upper layer as a result of centrifuging.

Procedure. In this work 50 ml. of the homogenized milk at room temperature was measured into a 50-ml. centrifuge tube (round bottom, ungraduated). For evaporated milk, the test was applied to the concentrated milk before sterilization, diluting it with an equal volume of water, and measuring 50 ml. of this diluted product into the centrifuge tube. The samples were centrifuged for 10 minutes at 1,500 r.p.m. (International Centrifuge, Head No. 240, radius 8 in. to the bottom of the extended pocket). With continuous, gentle suction applied through an assembly as illustrated in Figure 1, the surface of the centrifuged milk was scanned with the suction inlet barely (or intermittently) in contact with the surface. This was continued until 10 ml. had been collected, as measured in the graduated receiving tube. After thorough mixing of the 10-ml. sample its fat content was determined.⁴ From the known percentage fat content of the uncentrifuged sample (*A*) and the found percentage fat content of the 10-ml. sample (*B*) the per cent enrichment was computed as follows:

$$\text{Per cent enrichment} = \frac{(B - A)}{A} \times 100$$

⁴ In the present study the fat content was determined by the Babcock test, applied to an 8.8-ml. sample in an 8% Babcock test bottle. No water was added to compensate for the reduced volume of the bottle charge before adding the sulfuric acid. Ten ml. of sulfuric acid was used, added in three separate portions with vigorous agitation for 2 minutes after each addition. The 8.8-ml. sample was considered to be a "half sample," and the Babcock reading was multiplied by 2.

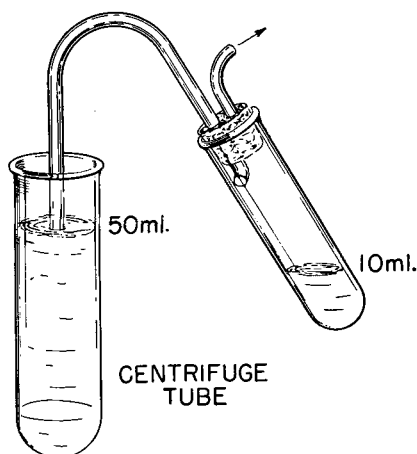


FIG. 1. Assembly for removing the upper 10 ml. for determining the effectiveness of homogenization.

The per cent enrichment might be considered as an effectiveness rating that parallels the tendency for separation and is inversely related to the size of the fat particles. The effectiveness rating may range from 0 for perfectly homogenized milk to approximately 400 for unhomogenized milk containing 4.0% fat.

B. Gravity Fat Separation Test

If the centrifugal test, as just described, is to measure differences in the effectiveness of homogenization, it must be applied before the other factors that affect fat separation have been altered by additional processing. In evaporated milk the centrifugal test should be applied before sterilization of the product. On the other hand, any fat separation test applied after sterilization will reflect not only the effectiveness of homogenization but also the consequences of protein coagulation and increases in viscosity. The inclusion of these additional factors, however, is precisely what is desired in a test to determine how effectively fat separation during storage of the product has been precluded.

An accelerated test for the latter purpose should recognize that "age-thinning" of evaporated milk during storage is part of the fat separation problem. For this reason the test that was adopted to measure fat separation tendencies included the acceleration of "age-thinning" through an elevated temperature.

Procedure. The test was performed in duplicate by placing two 14½ oz. cans of the evaporated milk in an upright position in an incubator at 100° F. immediately after the sterilization of the product. The cans remained undisturbed for exactly 7 days. Then, with careful handling to avoid mixing, the top of each can was cut out and a 25-ml. sample was removed from the upper surface. This removal and measurement was conveniently accomplished by the use of a 25-ml. pipette with its inlet tip bent upward. This tip was inserted in the center of the can so that the rim of the tip was parallel to and just below the surface.

By means of very gentle suction the pipette was slowly filled to its mark, keeping the inlet tip just below the surface but otherwise stationary. The fat content of the 25-ml. portion was determined by means of the Mojonnier fat test. The known fat content of the evaporated milk was subtracted from the found fat content in the 25-ml. sample and the difference was expressed as "per cent enrichment" by calculation as in the preceding test. This also may be spoken of as a fat separation rating.

C. Viscosity Measurement

Insofar as the viscosity measurement is to indicate the probable rate of fat rise in the product, the measurement ideally should be confined to the viscosity of the medium in which the fat particles, or fat-containing particles, are suspended. It is obviously impossible to attain this ideal; the measurement is of necessity confined to the rheological properties of the product as it exists.

The "viscosity" of the evaporated milk decreases on vigorous agitation, even after the weak, continuous gel, which may be evident immediately after sterilization, has been disrupted. In other words, evaporated milk exhibits plastic flow characteristics, which presumably should be recorded in terms of "yield value" and "consistency." It is, however, questionable whether the "yield value" has any reality in terms of the product as a whole. After the gel has been disrupted, the "yield value" most probably reflects the structural stability of suspended particles of indeterminate size and shape. The medium in which the particles are suspended undoubtedly exhibits true viscous flow.

In order to measure plastic flow characteristics, the flow rates of each sample must be measured at a number of different pressures or applied forces. Such a procedure might be used to provide information as to the structural stability of suspended particles in evaporated milk. It is, however, not apparent how such data should be interpreted in relation to the fat separation problem. At any rate, in the study in which the present methods were applied the flow of evaporated milk samples was measured under a single set of conditions, and the observed flow rates were expressed in terms of viscosity units. The plastic flow characteristics are tacitly acknowledged, and the results are kept on a basis that justifies comparisons between samples, in that the same set of conditions was used for all samples.

The modified Gardner Mobilometer. The instrument that was used for measuring the viscosity of evaporated milk was a modified Gardner Mobilometer (1). With this instrument the viscosity was measured by timing the descent of a disc or piston through the liquid in a vertical cylinder of uniform bore. A stem, attached to the piston, served to control the positioning, release, and timing of this disc or piston. The applied force could be varied by placing weights on a small pan at the top of the stem.

It has been found that this type of instrument, when used for liquids that exhibit true viscous flow, yields flow rates that are directly proportional to the applied forces over a wide range of forces; deviation finally occurs, as in all

viscosity methods, when the velocity is sufficiently high to produce turbulence. This type of instrument was originally adopted in this laboratory for cream viscosity measurements to obviate erratic results that will occur with capillary efflux instruments when the size of suspended particles approaches or exceeds the bore of the capillary.

The modifications consist of the following features: (*a*) a leveling stand for holding the removable cylinder, the entire assembly being of suitable design for placement in a water bath for convenient and accurate temperature control; (*b*) a stem guide to fit the top of the cylinder; and (*c*) a piston-like plunger of suitably chosen clearance in relation to the cylinder diameter, but with four small high points on the piston rim to serve as centering guides with a minimum of friction against the cylinder wall. The hollow piston-like plunger is illustrated in Figure 2, and the entire assembly, in Figure 3. By using a piston, instead of a disc of limited thickness, the length of the flow channel, between two parallel surfaces, is increased, and the clearance can be correspondingly increased. Except for very limited flow through the air escape vent, all of the flow occurs between two parallel surfaces, viz., the piston clearance in relation to the cylinder wall.

The cylinder was made out of a 10-in. length of 2-in. stainless steel sanitary tubing. In use it was filled uniformly to a height of 21 cm. Piston travel was timed through a distance of 10 cm. by sighting the passage of suitably placed marks on the stem in relation to the top of the stem guide. The position of the

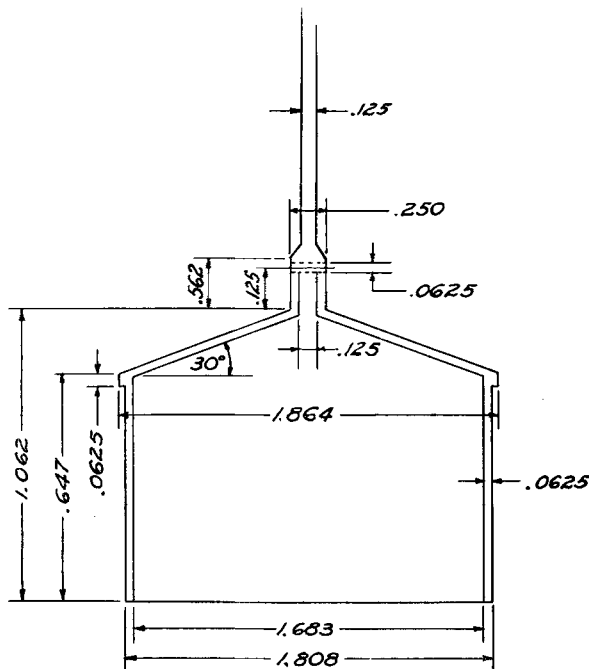


FIG. 2. The plunger of the modified Gardner Mobilometer.

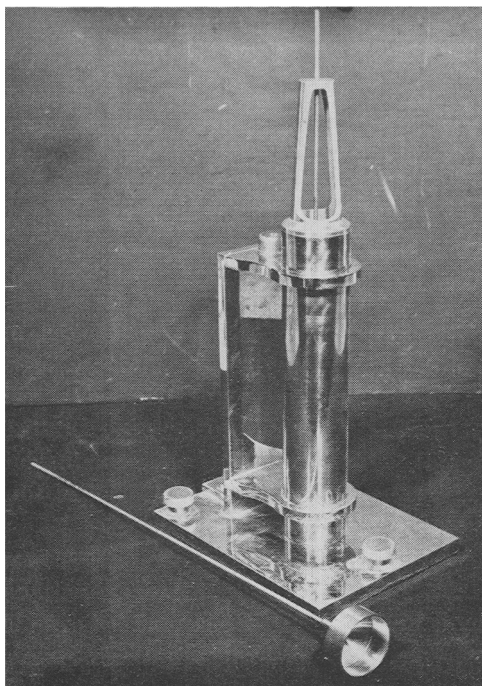


FIG. 3. The modified Gardner Mobilometer.

marks was established to allow at least 5 cm. travel for acceleration before starting the timing and to terminate the timing 2.5 cm. from the bottom to avoid a possible "end effect."

Procedure, standardizing the instrument. To standardize the instrument so that timing observations can be translated into viscosity units, it was necessary to determine the flow rate of a liquid of accurately known viscosity, using an accurately known applied force.

For solutions of known viscosity, sucrose solutions are conveniently used, referring to detailed tables that have been published for this purpose by the U. S. Bureau of Standards (2).

The applied force is the total weight of the traveling parts minus the buoyant force of the displaced liquid. During a measurement the piston is completely immersed, but the stem immersion varies. Accordingly, the volume of the piston and stem is to be determined to a point midway between the immersed stem length at the start and at the end of the timing interval. The displacement volume to this point can be most conveniently determined by weighing the piston and stem (*a*) suspended in air, and (*b*) suspended in water to the selected point on the stem. A torsion balance, accurate to 0.01 g., is suitable for this purpose. The difference between *a* and *b* in grams, divided by the density of the water in grams per cubic centimeter represents the displacement in cubic centimeters.

The instrument used in this study was calibrated in accordance with the above principles, and the essential data are as follows:

Weight of piston and stem in air	71.32 g. ⁵
Weight of piston and stem ⁶ in water, 35° C.....	63.79 g. ⁵
Weight of water displaced	7.53 g.
Density of water at 35° C.	0.99403 g./cc.
Volume of water displaced	7.58 cc.

⁵ Net weight, i.e., corrected for the weight of the suspension wire used in connection with the weighings.

⁶ Immersed to the selected point on the stem as explained.

Therefore, the effective force (in grams) employed in timing the descent of the piston through the liquid in the cylinder is:

$$71.32 - (7.58 \times \text{the density of the liquid in g./cc.}).$$

If the pan and weights are applied at the top of the stem, their weight is added directly to the effective force.

The constant (k) for the instrument was determined by utilizing a 60% sucrose solution at temperatures shown in Table 1, proceeding as directed below for viscosity measurement. The instrument constant (k) reflects the piston clearance and the distance of travel. With these factors fixed, the time (t) in seconds will be directly proportional to the viscosity of the liquid (n) in centipoises, and inversely proportional to the effective force (f) in grams. Thus, the relationship can be expressed by the formula $kt = n/f$. The fixed factors are therefore taken into consideration through the instrument constant (k).

With n and f known, and with t determined by experimental observations, k can be computed by means of the preceding formula. The data pertaining to the determination of the instrument constant are presented in Table 1. The five found values for the instrument constant are in satisfactory agreement; their average value is 0.01318.

Procedure, viscosity measurement. The cans of evaporated milk were placed

TABLE 1
Determining the instrument constant for the modified Gardner Mobilometer

Solution	Bureau of standards values ^a		Calculated effective force ^b	Observed time ^c	Instrument constant
	Viscosity	Density			
	(cp.)	(g./cc.)	(g.)	(sec.)	
60% sucrose solution, 5° C.	156.0	1.29334	61.516	190.76	0.01329
60% sucrose solution, 10° C.	109.8	1.29114	61.533	134.12	0.01330
60% sucrose solution, 20° C.	56.5	1.28640	61.569	70.26	0.01306
60% sucrose solution, 25° C.	43.86	1.28396	61.588	53.86	0.01322
60% sucrose solution, 35° C.	26.52	1.27876	61.627	32.98	0.01305

^a The values were converted to g./cc. by multiplying by 0.999973.

^b Weight of piston and stem, 71.32 g.; displacement volume, 7.58 cc.

^c The observed time represents the mean of 5 determinations in each case.

in a water bath at the chosen temperature ⁷ for 1 hour. The viscosity stand and cylinder also were placed in the water bath and the leveling screws were adjusted so that the stand was level and the cylinder was perpendicular. Each can was opened just prior to the viscosity test, and the contents were transferred to the cylinder with a minimum of agitation. The piston was inserted to such depth as to eliminate the air pocket from the hollow piston, allowing the air to escape through the vent for that purpose. The stem guide was then placed into position. With the eyes on a level with the upper surface of the stem guide, the piston was released and its time of descent was determined by sighting the stem marks in relation to the guide.

The observed time in seconds was converted to centipoises by substituting the proper values for f , k , and t in the formula: $n = fkt$.

In using this procedure with the sucrose solution, or any other fluid that exhibits true viscous flow, replicate determinations can be made with the same cylinder contents. This cannot be done, however, with evaporated milk; repeat determinations lead to successively lower readings, apparently because structures are being disrupted.

EVALUATION OF METHODS

Homogenization effectiveness test. The centrifugal procedure was evaluated by applying it to unconcentrated, homogenized milk with known admixtures of unhomogenized milk and to milk that had been homogenized at different pressures. Typical results are given in Table 2. The procedure also was evaluated in relation to evaporated milk manufacture, and these findings are reported in connection with the gravity fat separation and viscosity determinations in Table 3.

The results demonstrate that the procedure, as outlined above, will detect small admixtures of unhomogenized milk or small differences in the thoroughness of homogenization. These results were obtained with the fat contents measured by the Babcock test; greater precision probably could be achieved by using gravimetric fat determinations.

Composite evaluation of the tests. To evaluate the homogenization effectiveness test in conjunction with the viscosity test in relation to the gravity fat separation test, the three tests were applied to 27 samples of evaporated milk from the commercial operations of five different evaporated milk plants. For each sample one can of unsterilized milk and three cans of the same milk after sterilization were obtained. The homogenization effectiveness test was applied to the unsterilized product, diluted with an equal volume of water; one can of the sterilized product was used for the viscosity determination, and the other two cans were stored for the gravity fat separation test in duplicate. The results are given in Table 3.

⁷ In this study the viscosity of evaporated milk samples was measured at 37.78° C. (100°F.) using a piston and stem weight of 71.32 g., and assuming the density of the samples to be 1.06. The effective force was $71.32 - (7.58 \times 1.06)$ or 63.285 g. Because the effective force (f) was considered to be constant from sample to sample, this factor was consolidated with k ; $fk = 63.285 \times 0.01318$ or 0.834. Thus, $n = 0.834 \times t$.

TABLE 2
The sensitivity of the homogenization effectiveness test

Description of milk sample	Fat content of top 10 ml.		Homogenization effectiveness (% enrichment)
100 ml. homo. milk ^a	5.1	5.1	47.8
98 ml. homo. milk + 2 ml. unhomo. milk ^b	5.5	5.6	60.7
96 ml. homo. milk + 4 ml. unhomo. milk	5.8	5.8	67.8
94 ml. homo. milk + 6 ml. unhomo. milk	6.0	—	73.5
Milk ^a homogenized at 1,750 psi.	5.1	5.2	49.3
Milk homogenized at 2,000 psi.	4.8	4.7	37.7
Milk homogenized at 2,250 psi.	4.6	4.6	33.3
Milk homogenized at 2,500 psi.	4.4	4.4	27.5

^a Fat content 3.45%

^b Fat content 3.6%

TABLE 3
Gravity fat separation in evaporated milk in relation to the homogenization effectiveness and viscosity tests

Sample No. and Plant No.	Gravity fat separation	Homogenization effectiveness (% enrichment)	Viscosity (cp.)	HE/V ^b
1 - 3	1.7	17.4	30.0	0.58
2 - 3	2.0	19.3	27.4	0.70
3 - 5	2.1	20.6	40.7	0.51
4 - 4	2.3	19.3	37.0	0.52
5 - 4	2.5	26.3	39.9	0.66
6 - 5	2.8	21.8	37.4	0.58
7 - 2	3.2	22.5	26.9	0.84
8 - 3	3.8	12.4	22.9	0.54
9 - 2	3.9	21.2	22.3	0.95
10 - 1	4.3	30.0	31.8	0.94
11 - 5	5.1	18.7	40.9	0.46
12 - 1	6.6	23.1	22.1	1.04
13 - 5	7.2	21.8	25.0	0.87
14 - 1	8.8	32.6	23.4	1.39
15 - 1	10.0	23.4	20.5	1.14
16 - 3	10.2	25.0	18.1	1.38
17 - 3	10.6	22.5	15.3	1.47
18 - 2	11.8	21.7	15.3	1.42
19 - 4	13.7	31.3	17.6	1.78
20 - 4	18.1	28.8	11.9	2.42
21 - 2	19.8	23.7	14.7	1.61
22 - 4	23.5	29.6	13.8	2.14
23 - 1	26.8	46.4	14.7	3.16
24 - 3	30.5	24.4	10.8	2.26
25 - 2	37.5	24.4	9.0	2.71
26 - 1 ^a	76.9	70.5	9.8	7.19
27 - 1 ^a	124.0	106.0	9.2	11.50

^a Specially prepared samples.

^b Homogenization effectiveness rating divided by viscosity.

These data show that there is less gravity fat separation in the samples with a high viscosity. Likewise, there is less gravity fat separation as the effectiveness of homogenization is increased, obtaining a lower homogenization effectiveness rating.

Since the rate of gravity fat separation is directly related to the size of the

fat globules and inversely related to the viscosity of the suspending medium, these latter terms should somehow be combined. This may be accomplished by dividing the effectiveness of homogenization by the viscosity of the sample. Thus, a fourth term is obtained and this figure is directly related to the extent of gravity fat separation. This relationship is positive and is linear over the range of samples in this trial. The precision of the relationship between fat separation and the term reflecting homogenization effectiveness and viscosity can best be expressed by the statistical term coefficient of correlation. The data in Table 3 show a positive coefficient of correlation of 0.989.

DISCUSSION

It is logical to expect that the general phenomenon of fat separation can be viewed in light of the fundamental concepts relating to the rise of fat particles in unconcentrated milk. This permits the division of the problem into a number of separate entities, e.g., viscosity, size of the fat particles, and the density difference between the fat particles and the suspending medium. It is then necessary to adopt or devise methods for measuring the individual factors which can be related to the general phenomenon. For example, the rate of rise of the individual fat particles (likewise fat separation) would be directly related to the square of the radii of the fat particles.

From the theoretical standpoint the size of the individual fat particles is of interest, but from a practical standpoint the interest is primarily in the combined effect of the various sizes. The total effect of the various sizes will determine the extent of fat separation.

The total effect of the various sizes is the basis of the methods developed in this work for effectiveness of homogenization and gravity separation. This may be explained by the following reasoning: The sample to be tested is in a container of fixed height and the quantity removed from the upper layer is equivalent to a fixed height, or a fixed proportion of the total sample. The size of the fat particles influences the rate at which they travel. For example, a 2μ fat particle would travel x distance under the conditions of the experiment, and a 6μ fat particle would travel $9x$ distance, since the rate of travel is directly proportional to the square of the radius. Thus, the larger globules would travel a greater distance, and a greater number would travel from the lower section to the upper section to be removed in the sample for enrichment. It therefore follows that the tendency for enrichment is directly related to the size of the particles. On the basis of this reasoning, the previously described tests are fundamentally sound and measure the tendency for concentration in the upper layer, reflecting practical conditions.

The general phenomenon involves another factor, viscosity, which may be isolated and measured. The importance of viscosity can be determined by its influence on the rate of rise of the individual globules or the over-all problem of fat separation. With our present knowledge, however, the effectiveness of the method for measuring viscosity can be evaluated only in terms of the other tests, as a total relationship.

The over-all evaluation of methods using commercial samples showed a good correlation between gravity fat separation and the combined value for effectiveness of homogenization and viscosity. These results substantiate the efficacy of the methods and establish the relative importance of the individual factors of the general problem of fat separation.

SUMMARY

The problem of fat separation in evaporated milk can be divided into at least three factors. The extent of fat separation is inversely related to the viscosity, directly related to the square of the radii of the fat particles and directly related to the density difference between the fat particles and the suspending phase.

A suggested procedure is given for determining the effectiveness of homogenization. This method is based on the total effect, or the result of the rate of travel, of the fat particles. Extending the same logic, a method is given for determining the extent of gravity fat separation during storage. In addition, the procedure and description are given for a modified Gardner Mobilometer as a means of measuring viscosity.

These methods were applied to samples of commercially prepared evaporated milk, and the correlation between the tests was highly significant.

ACKNOWLEDGMENTS

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