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FAT SEPARATION IN EVAPORATED MILK. III. GRAVITY SEPARATION AND HEAT STABILITY[1]

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Fat separation is a continuous problem in the production, handling, and storage of evaporated milk. This defect becomes most objectionable during prolonged quiescent storage at high temperatures. The butterfat rises to the upper surface forming a viscous, leathery layer, which may prevent pouring of the milk from a relatively small opening.

To retard fat separation, the manufacturer attempts to obtain effective homogenization and sufficient coagulation of the proteins during sterilization to give the product a high viscosity or "heavy body" *(1, 12, 14)*. These practical applications are aimed at three fundamental considerations: a reduction in the size of the fat globules, an increase in the viscosity of the suspending phase, and perhaps an alteration in the density difference between the fat globule mass and the suspending medium *(6)*.

A consideration of the fundamental factors controlling fat separation by necessity would include the phenomenon of heat stability, which might conceivably influence both the viscosity and the density difference between the fat particles and the suspending medium.

The effectiveness of homogenization is known to influence the heat stability *(2, 5)*. The interaction of these phenomena, therefore, made it necessary to consider heat stability simultaneously with the over-all problem of fat separation.

When milk is heated sufficiently, the protein undergoes a gradual but complete coagulation. During the process of sterilization of evaporated milk, this gradual coagulation of proteins, especially when carried out in a quiescent state, tends to form a gel structure, which accounts for the increased viscosity of sterilized evaporated milk as compared to the unsterilized product *(5)*. The manufacturing practice, therefore, has been designed to obtain a partial heat coagulation of the proteins. The individual factors that influence heat stability have been reviewed by Hunziker *(5)*. He considers the following factors to be of importance: *(a)* acidity, *(b)* albumin and globulin content, *(c)* concentration, *(d)* forewarming treatment, *(e)* homogenization, *(f)* possible actual differences in casein, *(g)* relative concentration of ions, *(h)* rennet forming organisms, and *(i)* total salt concentration.

Homogenization is less detrimental to heat stability in normal concentrated milk than in concentrated milk that is otherwise relatively unstable, e.g., milk

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with developed acidity. The destabilizing effect becomes greater as the pressure of homogenization is increased *(5)*. Webb and Bell *(11)* have shown that the temperature of homogenization (98.6-176° F.) of forewarmed concentrated milk has very little effect on the final heat stability.

A plausible mechanism for the destabilizing effect of homogenization was originally suggested by Tracy and Ruehe *(10)*. They attributed the destabilization to the adsorption of casein or phosphates on the newly created fat surfaces. More recently, Sommer *(8)* suggested that the destabilization might result from the adsorption of either cations or anions, resulting in a disruption of the ionic equilibria. The present commercial practice, therefore, must be adjusted to meet the two problems: maintenance of sufficient heat stability to permit sterilization and the prevention of excessive fat separation.

The primary purpose of this work was to determine the efficacy of the various fundamental factors in retarding fat separation in a commercial product. Thus, the size of the fat globules, viscosity, and density difference received direct consideration and heat stability received indirect consideration.

EXPERIMENTAL PROCEDURE

A general description of the method used for preparing the evaporated milk is given below. Where experiments involved departure from the normal process, the details are given with each such experiment.

Raw mixed milk from the University dairy plant was tested for butterfat and total solids by the Mojonnier technique and adjusted to a ratio of 0.44 fat to solids-not-fat by the use of cream containing 19% butterfat. The milk was forewarmed in steam-jacketed stainless steel open hot wells, which held approximately 10 gal. and which were equipped with small mechanical agitators. In all experiments, the forewarming temperature was 206° F.; only the holding time was varied to obtain relatively high heat stability. When the desired holding time had been completed, cold water was turned into the jackets to cool the milk to 180° F. within 2-3 minutes.

A nickel vacuum pan was used to concentrate the milk to slightly less than half its original volume. The temperature of evaporation was 130-135° F., and the water was removed at the rate of approximately 20 gal. per hour. Thus, the process of condensing lasted 30 minutes or less. The concentrated product was then homogenized in a Creamery Package homogenizer which had a rated capacity of 125 gal. per hour. A new, single service (Multi-Flo) valve was used each time the machine was assembled. The homogenized, concentrated milk was stored at 32-35° F.

A sample of the milk was tested for butterfat by the Mojonnier technique. Sufficient distilled water then was added to the milk to adjust the fat content to 7.95%, except that enough water was withheld for later addition so that it could serve as a carrier of "correction." The milk was filled into 14½ oz. cans, which had previously received an addition of correction, consisting of 0 to 4 oz. of calcium acetate, or 0 to 10 oz. of anhydrous disodium phosphate per 1,000 lb. of concentrated milk.

The sealed cans were placed into a sterilizer that had individual recesses 3 in. from the center of the reel. Thus, the cans were permitted to roll in a manner analogous to that of cans in an Anderson-Barngrover sterilizer. The speed of the reel was fixed at 11.5 r.p.m., and the reel was allowed to run throughout the coming-up process but was run only for the first 3 minutes of the holding period. The heating schedule during the coming-up process was as follows: 5 minutes to reach 212° F., 5 minutes holding at 212° F., and 5 minutes to reach 242° F. After holding at 242° F. for various times, cooling was accomplished by injecting a spray of cold water directly onto the cans with the reel revolving.

The cans of milk were opened immediately after cooling and examined for coagulation by viewing the milk in a thin film on the side of a glass beaker by transillumination. The appearance of visible grain particles was taken to be the most advanced degree of coagulation that would permit sterilization for subsequent observations. A minimum heat treatment of 14 minutes at 242° F. was considered to be essential for commercial sterility. Thus, from the series of samples it was possible to determine the quantity of correction, if any, that was necessary to prevent excessive coagulation in the milk that was to be stored for observations on fat separation. Where it was necessary to add correction to the milk, the details are given along with each experiment.

The heat stability of each batch of concentrated, homogenized milk was determined by varying only the time of holding in the sterilizer at 242° F. The entire process of sterilization was repeated a sufficient number of times to determine the heat stability in minutes, the end point being taken as the appearance of small grain particles.

Fat separation was determined by storing cans of the sterilized evaporated milk for 7 days at 100° F. and then removing a 25-ml. sample from the upper surface for determining the butterfat content *(6)*. The viscosity measurements were made at 100° F. with a modified Gardner mobilometer *(6)*. The method for calculating average viscosity was given in detail in a previous publication *(7)*.

EXPERIMENTAL PROCEDURE

Sterilizing time. The viscosity of different samples of unsterilized evaporated milk is low and uniform, but during the process of sterilization there is a gradual increase in the viscosity. It has been shown that a high viscosity in the finished product tends to retard fat separation *(1, 6, 12, 14)*. Observations by previous workers, however, were made on different samples of milk. Therefore, it was not possible to evaluate the total effect of sterilization on fat separation.

Evaporated milk was prepared by the general procedure up to the time of sterilization. Separate batches of the canned milk were then sterilized for varying lengths of time while the temperature was maintained at 242° F. A representative set of the results is given in Table 1. From these data, it is apparent that an increase in sterilizing time causes an increase in viscosity and a decrease in fat separation. This relation can be expressed simply by the equation $VN = k$, where V is velocity, N is viscosity, and k is a constant. If the

TABLE 1
The effect of sterilizing time on viscosity and fat separation

Forewarming time	Sterilizing time	Viscosity After 1 hr.	Viscosity After 7 days	Viscosity Calculated average	Fat separation
(min.)	*(min.)*	*(cps.)*	*(cps.)*	*(cps.)*	*(% enrichment)*
15	14	7.3	6.0	6.2	39.6
15	18	8.8	7.0	7.3	27.0
15	22	11.0	9.3	9.6	12.4
15	26	14.3	10.4	11.1	8.3

velocity of rise of the fat globules is expressed by the test for fat separation *(6)*, and the k is taken as a constant in harmony with the conditions of the experiment, then fat separation multiplied by viscosity should give a constant value.

From the data in Table 1, it can be shown that viscosity multiplied by fat separation gives a progressively smaller value as the time of sterilization is increased. Thus, the indications are that fat separation is retarded more by an increase in time of sterilization than would be expected solely from the resulting increase in viscosity. It seems necessary, therefore, to consider other factors for an explanation of this phenomenon. One such factor might be the difference in density between the fat globule mass and the suspending medium.

Homogenization prior to forewarming. If there had been an increase in the density of the fat globule mass due to adsorption of proteins during sterilization, forewarming should produce a similar effect, but to a lesser extent. However, any material added to the fat globule during the normal process probably would be dislodged during homogenization, since homogenization is applied after forewarming. On the other hand, homogenization prior to forewarming would give an increased surface area of the butterfat, permitting additional adsorption.

Raw mixed milk from the University dairy plant was standardized and then heated to the desired temperature for homogenization. Immediately after homogenization at 2,000 lb. pressure, the milk was transferred to the hot wells and forewarmed. The forewarmed milk was concentrated and subsequently standardized to the normal composition of evaporated milk.

One major difficulty was encountered when the milk was homogenized prior to forewarming. Homogenization at low temperatures (93-135° F.) resulted in an exceedingly unstable concentrated product. To permit sterilization, it was necessary to homogenize the unconcentrated milk at 170° F., or above, or to rehomogenize the concentrated product at approximately 130° F. Therefore, it was not possible to carry out the experiment so that none of the albumin and globulin would be coagulated before homogenization.

The data in Table 2 show that the temperature of homogenization of unconcentrated milk has a marked influence on the heat stability of the forewarmed, concentrated product. As the temperature of homogenization was reduced from 175° F., there seemed to be a progressive reduction in the heat stability, even though forewarming at 206° F. was applied immediately following homogenization. When the unconcentrated milk was homogenized at a sufficiently high

TABLE 2

The heat stability of evaporated milk homogenized prior to forewarming

Milk sample	Temperature of homogenization	Forewarming time	Heat stability: Not rehomogenized	Heat stability: Rehomogenized at 1,000 lb.	Heat stability: Rehomogenized at 2,000 lb.
	(° F.)	*(min.)*	*(min.)*	*(min.)*	*(min.)*
A	170	25	20	—	—
B	164	0	11	—	13
B	164	10	13	—	17
B	164	20	13	—	17
C	130	20	2	2	2
C	175	20	15	21	20
D	130	20	2	2	2
D	175	20	15	22	22

temperature to produce a relatively stable concentrated product, the heat stability could be improved by rehomogenizing the concentrated milk at 130-135° F. This improvement in heat stability could be obtained by rehomogenizing the concentrated milk at either 1,000 or 2,000 lb. pressure.

As for fat separation observations, it was possible to utilize only a limited number of the samples that were homogenized prior to forewarming. The results indicated the possibility of producing evaporated milk with low fat separation tendencies when the temperature of homogenization was near 160° F.; on the other hand, when the homogenization temperature was 175° F., the fat separation was extensive.

Homogenization of cream. Since it was not possible to obtain direct information on the effect of coagulating albumin and globulin in the homogenized milk, an indirect approach seemed desirable. If the butterfat were not present during the forewarming process, there would be no adsorption of albumin and globulin onto the fat globules. Therefore, fat separation should be more rapid than under the normal process of manufacturing evaporated milk.

The above procedure has definite practical possibilities because the milk could be separated, allowing only the cream to pass through the homogenizer. The cream and skimmilk could be combined at any convenient stage in the process. Thus, the working capacity of the homogenizer would be increased threefold or more, and the separator would function also as a clarifier. However, this procedure might accentuate the problem of heat stability, since cream is extremely susceptible to the destabilizing effect of homogenization *(3, 10, 13)*.

With these considerations in mind, cream containing approximately 20% butterfat was heated to 136° F. and homogenized at 2,000 lb. pressure. Skimmilk was forewarmed at 206° F. for 25 minutes, then condensed to slightly less than half the original volume. These products were blended to give a mixture with the exact composition of evaporated milk. A part of the blended product was heated to 136° F. and rehomogenized at 2,000 lb. pressure. Each of the samples, with and without rehomogenization, was sterilized and stored for fat separation observations. A representative set of the results is given in Table 3.

Fat separation is apparently greater when the butterfat is withheld from

TABLE 3
Fat separation in evaporated milk made from homogenized cream and concentrated skimmilk

Rehomogenized	Viscosity			Fat separation
	After 1 hour	After 7 days	Calculated average	
	(cps.)	*(cps.)*	*(cps.)*	*(% enrichment)*
No	14.3	8.7	9.8	66.2
Yes	10.4	7.2	7.8	16.7

the normal forewarming process and homogenized in the form of cream. These results could be attributed to an adsorption of albumin and globulin during the normal forewarming process or to less effective homogenization of the high butterfat product. At any rate, fat separation was sufficiently pronounced to cast considerable doubt on the feasibility of separating the milk for fractional homogenization as a commercial process.

The heat stability of the above samples was sufficiently high to permit sterilization; it could be improved, however, by rehomogenizing the blended product at 136° F. This increase in the heat stability amounted to 4-5 minutes at 242° F. and was probably associated with an increased subdivision of the fat globules.

Repeated homogenization. To determine the effect of repeated homogenization on fat separation, evaporated milk was prepared according to the general procedure through the process of condensing. After standardization, the product was homogenized at 130-135° F. from one to seven times by repeatedly passing the milk through the homogenizer at 2,000 lb. pressure. The individual batches were sterilized and stored for fat separation observations. A representative set of the results is given in Table 4.

TABLE 4
The effect of repeated homogenization on fat separation

No. of times homogenized	Viscosity			Fat separation	Heat stability
	After 1 hour	After 7 days	Calculated average		
	(cps.)	*(cps.)*	*(cps.)*	*(% enrichment)*	*(min.)*
1	18.2	12.2	13.3	5.7	20
3	19.1	13.5	14.6	1.9	20
5	18.7	13.5	14.5	2.4	20
7	25.0	17.3	18.8	0.9	20

The data indicate that repeated homogenization under the conditions of this experiment has little effect on fat separation. This was especially true where homogenization was highly effective on the first passage of the milk through the machine. In addition, repeated homogenization did not alter the heat stability.

Temperature of homogenization of concentrated milk. Whole milk was standardized to the proper ratio of fat to solids-not-fat, forewarmed for 15 minutes, condensed, and adjusted to the desired temperature for homogenization. After homogenization, each batch of the milk was standardized to 7.95% butterfat.

TABLE 5
The effect of temperature of homogenization on fat separation

Temperature of homogenization	Viscosity			Fat separation	Heat stability
	After 1 hour	After 7 days	Calculated average		
(° F.)	*(cps.)*	*(cps.)*	*(cps.)*	*(% enrichment)*	*(min.)*
110	10.2	7.1	7.8	30.3	23
146	10.2	7.2	7.8	21.4	23
170	9.7	7.1	7.6	14.2	23

The remainder of the process was carried out according to the general procedure, which has been outlined previously. A representative set of the results is given in Table 5.

The data indicate that an increase in the temperature of homogenization results in less fat separation during subsequent storage. This could probably be attributed to an increase in the effectiveness of homogenization or to an increase in the extent of adsorption at the higher temperatures.

The heat stability was not altered by changes in the temperature of homogenization.

The effect of soybean lecithin on the efficiency of homogenization and fat separation. Holm *(4)* has pointed out that one of the forces to be overcome by homogenization is interfacial tension between the fat and the serum. Furthermore, Sommer *(9)* states that the size of fat globules attained in skimmilk with a given mechanical emulsifying action tends to vary inversely with the quantity of phospholipid that is added to butter oil. Thus, fat separation should be reduced through the addition of soybean lecithin to butter oil, since the emulsifying properties of soybean lecithin should alter the state of dispersion of the butterfat.

The previous experiments indicated that the dispersion of the fat globules exerted considerable influence on the final heat stability. Accordingly, the phospholipids of milk, through their emulsifying properties, might have an effect on heat stability.

The experiment was conducted as follows: butter from the University creamery was oiled off at 140° F. and filtered through cotton to obtain a clear butter oil. Sufficient soybean lecithin was dissolved in a part of the butter oil to represent 8% of the total weight. Skimmilk was forewarmed at 206° F. for 20 minutes, then condensed to slightly less than half its original volume. The condensed skimmilk and distilled water were blended with butter oil containing soybean lecithin and with butter oil alone to give products with the composition of evaporated milk. Each batch was mixed at 140° F. for 10 minutes in a motor driven emulsifier (Creamaid), and the resulting product was homogenized at 2,000 lb. pressure. The remainder of the process was carried out according to the general procedure.

The data, an example of which is given in Table 6, indicate fat separation was not materially altered by the addition of soybean lecithin. The results should be considered as quite significant, however, since the viscosity of the samples without added lecithin was much higher, thereby retarding fat separa-

TABLE 6
The effect of soybean lecithin on fat separation and heat stability

Per cent fat as soybean lecithin	Viscosity			Fat separation	Heat stability
	After 1 hour	After 7 days	Calculated average		
	(cps.)	*(cps.)*	*(cps.)*	*(% enrichment)*	*(min.)*
0.0	22.9	16.6	17.8	14.6	15
8.0	8.6	7.2	7.5	11.3	60

tion. Undoubtedly, the primary effect of the soybean lecithin was to increase the efficiency of homogenization. Microscopic observations showed a greater subdivision of the fat in the homogenized samples which contained soybean lecithin.

The soybean lecithin had a marked influence on the heat stability. Thus the results support the previous experiments where the state of dispersion of the butterfat had an influence on the heat stability. On the other hand, there is a possibility that the increase in heat stability resulted from a physico-chemical effect of the soybean lecithin.

DISCUSSION

In light of the present knowledge, there are three fundamental factors that control the rate of fat separation in evaporated milk. These factors are viscosity, size of the fat particles (effectiveness of homogenization), and density difference between the fat particles and the suspending medium.

The difference in density between the fat particles and the suspending medium is the factor which has the greatest appeal to potential investigators, because a reduction of this difference to zero is one way of completely eliminating fat separation. An increase in viscosity or effectiveness of homogenization can only prolong the time before objectionable fat separation would become apparent. Unfortunately, the means of increasing the relative density of the fat particles are extremely limited. Apparently this would depend on a closer association of materials like casein with the fat particles and in greater quantities than hitherto possible. With the present knowledge, this phenomenon can be accomplished only to a limited degree during the process of sterilization. Nevertheless, an advanced degree of coagulation gives a higher viscosity to retard fat separation. A high viscosity combined with effective homogenization is a relatively effective means of retarding fat separation. In commercial practice it seems advisable, therefore, to carry the coagulation process as far as possible without impairing the desirable properties of the finished product. This, of course, depends on the heat stability.

The method that was chosen for determining heat stability in this work had as its end point the appearance of small grain particles. It should be recalled, therefore, that the results are a measure of the resistance of the sample to coagulation by heat but do not indicate the maximum viscosity which could be produced in the evaporated milk. For example, one batch of evaporated milk showed

a heat stability of 20 minutes, and after sterilization for 19 minutes, had a viscosity of 18.2 cps.; another batch of evaporated milk showed a heat stability of 17 minutes and after sterilization for 15 minutes had a viscosity of 54.5 cps.

The increase in viscosity during sterilization is limited by the appearance of grain particles which, presumably, result from flocculation of the proteins. Since agitation of the cans of evaporated milk during sterilization hastens grain formation, it is logical to suspect that the hastening of grain formation comes from breaking of the gel structure, which permits flocculation of the proteins. It seems equally logical to expect that points of weakness within the gel structure would permit more rapid flocculation of the proteins and thereby enhance grain formation. Thus, the heat stability of evaporated milk, as determined in this work, would be reduced by the presence of weak points in the gel structure.

These weak points in the gel structure could conceivably arise either directly or indirectly from clumps of fat globules. If the weak points arise directly from clumps of fat globules, the result would be considered as physical, whereby there is a disruption in continuity of the protein phase. On the other hand, if the weak points arise indirectly from clumps of fat globules, the result would be considered as electro-chemical. The proteins that are enmeshed in the clumps and surrounding the clumps could be influenced by the electrical charge on the fat globules. Whether the proteins are either more heat stable or less heat stable than proteins that are free of the clumps, the effect would be the same. Consequently, there would not be a uniform rate of coagulation of the proteins throughout the milk, and weak points in the gel structure would result.

Thus, grain formation would be enhanced by the presence of clumps of fat globules. The concept that clumps formed during homogenization influence the heat stability of the finished product aids materially in explaining the results obtained in this work on heat stability. This is especially true where the heat stability of the milk could be improved by a second homogenization. In addition, the results extend the work reported by Doan *(3)*, who showed that increased fat clumping in cream is accompanied by a decreased stability of the cream toward coagulation by alcohol and by heat. The astonishing thing, however, is that heat stability should be influenced materially by the slight clumping which is visible in unsterilized evaporated milk.

The above comments on heat stability are intended merely as additions to the postulation presented by Tracy and Ruehe *(10)* and extended by Sommer *(8)*.

SUMMARY

The fundamental factors governing fat separation are considered in light of the normal commercial processing methods.

During sterilization there is an increase in viscosity; this is desirable for preventing fat separation during storage. Sterilization produces an additional reaction, which gives an increased retardation of fat separation. This phenomenon may be explained by assuming an increase in the density of the fat globule

mass. In harmony with the preceding supposition, there is considerable doubt about the feasibility of separation of milk for homogenizing the cream alone.

The most important factor governing fat separation is effective homogenization. In this respect the condition and temperature of the medium are extremely critical. At higher temperatures, homogenization is more effective. The maximum effectiveness of the homogenizer can be obtained if all known factors are adjusted to ensure complete dispersion of the butterfat.

The state of dispersion of the butterfat is of additional importance, since it affects the heat stability during sterilization. This work also shows that under certain conditions of homogenization, there is an increase in the heat stability.

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