

Fig. 1. Stability of normal and multiple solids milk at various storage temperatures. The numbers associated with the curves refer to the solids ratios to which the milk was concentrated. Body stability is expressed in terms of the protein solubility index, that is, the number of milliliters of deposit obtained on centrifuging 50 ml. of milk of approximately 13% solids content in a graduated tube under uniform conditions. Total destabilization or coagulation of the protein was indicated when as much as 16 ml. was deposited in the tube.

*Factors that affect
the stability of*

frozen concentrated milk*

By
R. W. BELL

*Dairy Products Section
Eastern Utilization Research and Development Division
Agricultural Research Service
U. S. Department of Agriculture
Washington, D. C.*

FROZEN storage is the only way by which whole and skimmilk can be preserved over a long period without essentially altering their fresh natural flavor. When these fluids are converted into relatively stable concentrates, such as sweetened condensed and evaporated milk and milk powder, they lose their identity and take on the flavor and other properties which are characteristic of each new product.

That frozen condensed whole or skimmilk is acceptable in the making of ice cream has been shown by several investigators, Openlander and Erb⁹, Reichart¹⁰, and more recently it has been reported¹¹ that an acceptable loaf of bread can be made from doughs that contain 6% milk solids-not-fat derived from thawed, destabilized, and reconstituted skimmilk, of even deteriorated flavor. Over a period of more than a year there was no substantial decrease in the baking quality of the frozen condensed skimmilk. As in nonfat dry milk, baking quality was related to the forewarming treatment that the skimmilk received.

A thawed concentrated milk that had the flavor and physical characteristics of fresh concentrated milk would be a satisfactory source of beverage milk, in so far as the quality is concerned, with or without mixing with a supply of fresh milk.

The problem of the preservation of concentrated whole and skimmilk in a frozen state can be divided

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into two parts, namely, the retention of a satisfactory body and an acceptable flavor.

Body

Retention of a good body in frozen milk revolves around the physical instability of the casein complex. Increasing the concentration of the solids by removal of water decreases the physical stability of milk, especially of this portion, as does high-heat treatment and homogenization. While cold homogenized, pasteurized whole milk can be held for days prior to freezing without influencing the rate of protein flocculation in frozen storage¹, concentrated milk should be cooled and frozen without delay in order to attain the maximum storage life¹⁶. This becomes an increasingly important factor as the solids level is raised above about 40%. Holding high solids milk after removal from the condensing equipment, particularly if it is first cooled, allows the casein micelles to aggregate into structures and may cause the formation of lactose nuclei which accelerate crystallization and thus could hasten protein aggregation. Opportunity for aggregation and crystallization and subsequent destabilization can be limited by prompt cooling and quick freezing.

Freezing, in itself, produces no measurable flocculation of the protein of concentrated skimmilk which is not immediately reversible on defrosting and reconstitution⁷. Frozen storage, however, causes a gradual change in the protein particles whereby, with time, the reversibility is lessened and a floc can be separated and washed by centrifugation. In the early stages this floc is redispersible with time or immediately by heat or agitation or both. In extended frozen storage the precipitated protein eventually may lose its reversible character completely.

The causes of protein flocculation in frozen stored milk are postulated by Sommer¹², who suggests that higher salt concentration, probable higher acidity, physical crowding of the protein micelles by ice crystals, and removal of water of hydration are the destabilizing factors, with the increasing viscosity as an opposing factor. The latter decreases the freedom of the particles to form aggregates. Webb and Hall¹³ are convinced that calcium caseinate, a weakly hydrated sol in milk, comprises the bulk of the flocculated protein and they believe that frozen storage further lessens the hydrophilic character of this protein so that on thawing it behaves more as a strictly hydrophobic substance.

Sodium citrate was found by Babcock² and by others⁵ to be of value as a body stabilizer in homogenized milk frozen and stored at 0° F. However, by far the most effective way to increase body stability is to lower the storage temperature. Relationships between solids concentration and storage temperature and the body stability of the product are shown by Bell and Mucha⁴ who concluded that -10° to -20° F. is a narrow temperature range above which neither milk nor its concentrate should be stored if prolonged preservation of a satisfactory body is desired and below which little additional stability is to be attained.

While satisfactory body stability usually can be obtained by storing at -10° to -20° F., such

facilities are not always available and, in any event, they cost more to maintain than comparable space at a higher temperature. Consequently, practical means for attaining the desired stability at a higher temperature are of interest and value or it may be important to know how to stabilize the product for a longer period at a given temperature. In these circumstances the addition of cane sugar¹⁶ or replacement of part of the calcium with sodium by ion exchange^{16, 8} could be resorted to.

An important contribution to our knowledge of the role that lactose plays in the body stability of concentrated frozen milk has been made by Tumerman and associates¹⁴. They concluded that

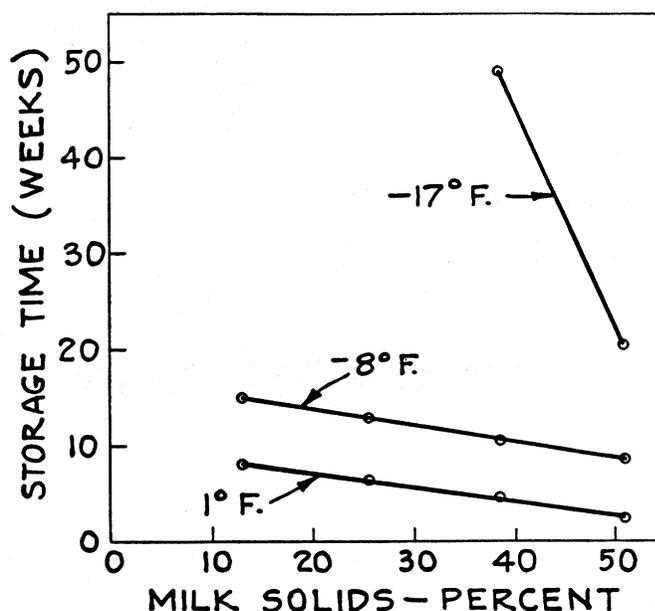


Fig. 2. Dependence of satisfactory storage life on solids content and storage temperature for milk stored at +1°, -8° and -17° F. A sample was considered stable if not more than 1.0 ml. of deposit formed on centrifuging 50 ml. of thawed and reconstituted milk.

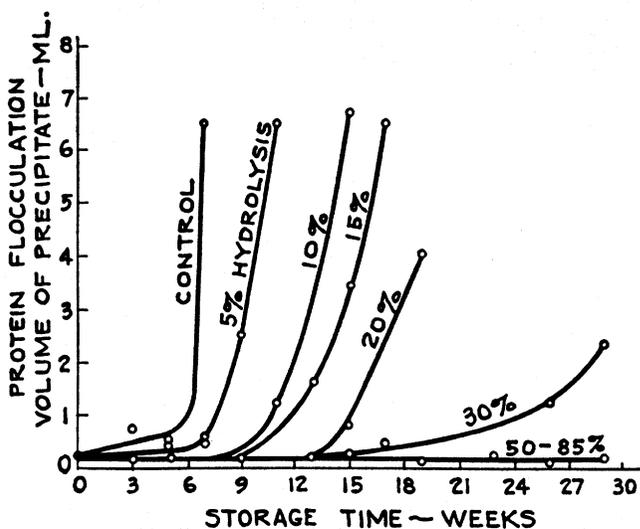


Fig. 3. Influence of enzymatic lactose hydrolysis on the protein stability of concentrated whole milk (35% solids-stored at 15° F.). (From data by Tumerman¹⁴.)

coagulation of casein during the storage of frozen concentrated milk is a direct consequence of lactose crystallization. Withdrawal of soluble lactose from the unfrozen phase by crystallization eliminates this sugar as a stabilizing factor and permits casein to flocculate. They explain the stabilizing effect of low temperatures by saying that flocculation of the casein is suppressed when over 90% of the water is frozen and the amorphous lactose phase becomes stable against crystallization. As proof of this Tumerman (Fig. 3) showed that the frozen concentrated product was stabilized in proportion to the degree of enzymatic lactose hydrolysis with the consequent retardation of lactose crystallization. From a body standpoint partial hydrolysis of lactose to monoses evidently increases the stability of concentrated frozen milk.

Another way to attain greater body stability, as pointed out by Tumerman, is to remove part of the lactose by forced crystallization and centrifuging and replace it with a more soluble sugar such as sucrose.

Temperatures which afford complete protection against protein destabilization (approximately -20° F.) coincide with almost total freezing of the water in the milk. At such temperatures the lactose is in a stable amorphous glass state, unable to crystallize without dilution. At temperatures between this stable temperature and -10° F., although a considerable amount of water remains unfrozen, the crystallization of lactose is significantly retarded and frozen concentrated milk is stable for periods longer than 15 weeks. The importance of maintaining a low temperature is thus evident. Fluctuation in temperature favors lactose crystallization and consequent protein destabilization. It is evident, also, that milk must be maintained at a lower temperature than most frozen foods, if long continued storage without significant deterioration in the form of protein denaturation is to be expected.

It is interesting to note that Wildasin and Doan¹⁰, by adding cane sugar, delayed the appearance of flocculated protein during frozen storage of concentrated skim milk even though lactose crystallization was evident in the early days of the storage period.

Flavor

While the use for which the product is intended is an important consideration, it can be said that the retention of a fresh flavor during frozen storage is the limiting factor in the preservation of milk and its concentrates at the lower solids levels. At some point, perhaps 40% solids, above which frozen concentrated whole and skim milk have a strong tendency to lose their fluidity and form a gel, the preservation of a satisfactory body rather than flavor, becomes the limiting factor.

Bell and Mucha³ were concerned with the extent of the heat treatment that milk could be given without impairing the flavor of the thawed product. Under the conditions of their experiments best results were obtained when the milk was heated at 160° F. for one minute and fortified with 50.0 mgm. of ascorbic acid per liter and at 170° F.

for one minute followed by brief boiling in a vacuum pan. The addition of ascorbic acid decreased the tendency of the milk to develop an oxidized flavor; boiling in the vacuum pan was considered a minor factor. The tendency of milk and of skim milk to develop an oxidized or cappy flavor makes it desirable to give the milk as severe a heat treatment as possible without imparting an undesirable flavor. In circumstances where a heated or cooked flavor is relatively unimportant, and therefore permissible, and the milk is processed accordingly, there is little likelihood that an oxidized flavor will develop in whole or skim milk during frozen storage. The body destabilizing effect of the heat can be offset by holding the milk at a lower storage temperature.

Possibilities of preserving milk solids in a frozen state over a long period were revealed by Tracy et al in 1950¹³. They found that samples of whole and concentrated milk remained in a satisfactory condition for at least a year at -5° to -15° F. They claimed better keeping qualities of the solids under these conditions than when comparable milk was dried and then stored at the same temperature. In preparing these products Tracy heated the milk at 170° F. for 20 minutes. Milk so heated does not develop an oxidized flavor in frozen storage.

Bell and Mucha⁴ have obtained variable results on the effect of lowering the storage temperature on the development of an oxidized flavor in whole milk. At -8° F. samples sometimes developed an oxidized flavor whereas at $+1^{\circ}$ F. comparable samples did not. However, at -17° F. the milk was less likely to become oxidized than at -8° F. and it was otherwise more stable in flavor.

As the solids content of milk and of skim milk is increased by the removal of water, the product becomes less susceptible to the development of an oxidized flavor during frozen storage. At a solids concentration of about 38% and above, an oxidized flavor is rarely detectable even after prolonged storage⁴. This effect was noted in 2:1 milk by Corbett and Tracy⁶. Bell and Mucha found that the effect was not permanent in that whole milk prepared from freshly concentrated milk by the addition of distilled water was as susceptible to the onset of an oxidized flavor during frozen storage as similar milk that was not concentrated.

From a practical standpoint this is of relatively minor importance because, as has been said, the development of an oxidized flavor in frozen whole milk can be prevented by suitable heating and it seldom occurs in 3 to 1 whole or skim condensed even when the heat treatment does not exceed that required by conventional pasteurization.

Why concentrated milk is more resistant to the onset of an oxidized flavor during frozen storage than unconcentrated milk has not been explained. It is not due to a lower Eh because the ratio of oxidants to reductants in milk is not changed by the evaporation of water. The oxidation-reduction potential of multiple strength milk is essentially the same as that of single strength milk.

Other defects that occur in frozen condensed milk are described as old and stale and a loss of flavor character. The best defense against them is the use of

heat just short of the point where it has an objectionable effect in itself, and storage at a low temperature such as -20° F. Regarding the former it should be remembered that a heated flavor tends to become less intense and even disappear with time. However, a flat or neutral flavor takes its place. From a market milk standpoint preservation of a satisfactory flavor is the limiting factor in the long time preservation of milk by freezing. Retention of a good body lends itself to solutions of which a low storage temperature is by far the most effective and practical.

A company with milk concentrating equipment and refrigeration facilities, but no dryer, can preserve surplus skimmilk for baking as well as for ice cream making and possibly other uses from the period of surplus milk production to the time when there is a shortage of this commodity. In special circumstances frozen concentrated whole milk may be advantageous as a source of beverage milk. Only recently have builders begun to equip ships with low temperature storage facilities and more and more of this kind of storage is becoming available all over the United States. Refrigerated warehouse operators as well as people in the dairy business are interested. When as much research has been done on frozen concentrated milk and its uses as on the major and long established dairy products the results may be encouraging to its future for retail as well as wholesale purposes.

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