EFFECTS OF VISCOSITY AND TEMPERATURE ON THE FOAMING CHARACTERISTICS OF CONCENTRATED WHOLE MILK

T. F. HOLDEN, N. C. ACETO, AND E. F. SCHOPPET

Eastern Regional Research Laboratory, USDA, Philadelphia, Pennsylvania

SUMMARY

To better understand continuous vacuum foam-drying of whole milk, a study of milk concentrate foams was undertaken. An apparatus was developed for measuring two independent parameters, foaming ability and stability. Foaming ability was defined as the initial height of foams immediately after formation in a column and stability was defined as the rate of subsidence of these foams. Foam determinations were made on several concentrate batches of $44 \pm 1.5\%$ solids between 50 and 95 F. Variations in concentrate viscosity were caused by temperature changes and intrinsic batch differences and ranged from 70 to 800 centipoises. Foam stability was well correlated with concentrate viscosity. Foaming ability of each batch was correlated with temperature and minima were 70 F. A complex correlation of viscosity and temperature described foaming ability for all batches. These experiments confirmed the importance of viscosity and temperature as control parameters in the vacuum foam-drying process and also showed that final foam structure is established early in the drying operation.

In the course of developing a continuous vacuum foam-drying process for whole milk (1), it was found that the foaming characteristics of whole milk concentrates play an almost predominant role in controlling the rate of drying and product quality. Consequently, an investigation was initiated to gain information concerning factors which affect the foaming of whole milk concentrates.

Very little information has been gathered concerning milk concentrate foams and not much has been learned about any dairy product from the standpoint of determining how foaming is affected by physical properties. Other workers (10), using a variety of foam tests, have investigated relationships existing between the foaming properties of dairy products and the various protein fractions, fat levels, and minor chemical constituents encountered in these products. Most of these investigations have been carried out either on unconcentrated fluid milk or on water solutions containing specific milk constituents near the levels in which they appear in fluid milk. The effects of physical properties on foams also have been studied. However, these studies have been confined in most cases to such systems as detergent-water solutions (5), which are not complicated by the great profusion of chemical constituents found in dairy products.

Even though the experiments presented in this paper were undertaken to gain information for direct application to the continuous vacuum

foam-drying process, any information obtained about milk concentrate foams should be applicable to other dairy operations where foaming of milk concentrate is encountered. In fact, the information also could assist, at least qualitatively, in understanding the properties of foams formed from any proteinaceous material of high viscosity.

EXPERIMENTAL PROCEDURE

Apparatus. The apparatus developed for measuring milk concentrate foaming characteristics is shown in Figure 1. It is superficially similar to static foam testing equipment employed by others in the measurement of fluid milk foam properties (6). However, certain modifications of prior fluid milk foam tests of this type have been made, principally to take account of the particular rheological properties of milk concentrates and to better define the parameters which describe foaming characteristics.

The apparatus is designed to force a fixed volume of gas (nitrogen) through a sintered glass disc into a layer of liquid contained in the bottom of a vertical column. Observations then are made on the volumes and subsidence rates of the foams produced.

Procedure and measurements. To investigate the foaming characteristics of milk concentrate at a constant temperature, a 350-cc sample of the concentrate is placed in the tempering bath. When the sample reaches the control temperature it is transferred to the column, producing
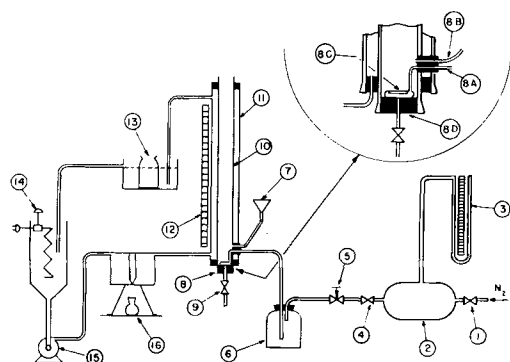
FIG. 1. Foam determination apparatus. (1) Accumulator charging valve; (2) accumulator; (3) mercury manometer; (4) accumulator discharge valve; (5) flow regulator (needle) valve; (6) trap; (7) sample inlet funnel; (8) sparging section; (8a) gas inlet port; (8b) sample inlet port; (8c) side entering 30-mm EC fritted glass sparging disc; (8d) rubber stopper; (9) drain valve; (10) 3-in. id by 48-in. column; (11) jacket; (12) measuring rod; (13) sample tempering bath; (14) thermostatically controlled constant temperature bath; (15) circulating pump; (16) jacketed viscosimeter.

an initial concentrate level of 2.65 in. above the sparging disc. Concurrently, the viscosity of a 50-cc aliquot is measured at the control temperature in a Saybolt fural tip viscometer.[1] A separate viscosity check was also made at room temperature before and after each run, to insure that concentrate age thickening had not occurred during the course of a run.

In the meantime, the accumulator has been pressurized to 30 in. of mercury (gauge) and, on sparging, the pressure is dropped to 25 in. This delivers 5,550 cc $\pm$ 3% of nitrogen at standard temperature and pressure to the sample. Sparging times were dependent to a certain degree on the viscosity of the sample being foamed and a variation in over-all sparging time of 60 to 90 sec was observed during these experiments. It should be noted that these sparging times were not recorded as experimental data, since foaming, in a test such as this, is virtually independent of sparging time (3, 8). Immediately after the foam is formed, its initial height is recorded and a stopwatch is started. The foam height is recorded at 30-sec intervals until either 600 sec have passed or the foam has entirely subsided.

Burcik (5) points out that any foam test designed to compare the quality of foams consisting of large numbers of bubbles should

[1] Reference to certain products or companies does not imply an endorsement by the Department over others not mentioned.

clearly differentiate between the ability of a solution to form foam and the stability of the foam once it is formed.

A reasonable criterion of foaming ability is obviously the maximum height to which the foam is expanded immediately after the sparging gas is shut off. Therefore, this is used in these tests and will be referred to as Initial Foam Height (H_i).

In many foam tests of this type, a problem seems to exist in the selection of a suitable parameter for the description of foam stability. Heretofore, half-volume time, i.e., the time for the foam to collapse half way, has been employed to define stability. This, by definition, does not separate foam stability from foaming ability, because of the dependence of half-volume time on the initial foam height. If the foam would subside at a linear rate with time, the problem could easily be resolved by assigning the slope of the foam height-time line as the criterion of foam stability. Fortunately, this was found to be the case for milk concentrate foamed in the apparatus described. Therefore, for each run, the foam height is measured at 30-sec intervals and a linear regression is run on the data thus obtained. This regression yields a straight line of the form:

$$H = m \Theta + I$$

where:

H = foam height in inches

Θ = time in minutes

m = regression coefficient, i.e., the slope of the straight line

I = the H intercept.

The slope, m, is used as the independent criterion of foam stability and is referred to as the Rate of Foam Subsidence (R_{fs}) and is in units of inches per minute. Figure 2 shows an example of a foam height-time plot with the regression line drawn in. Similar determinations (giving different slopes) were made on other foams at different conditions in collecting the data for this paper. In all of the foam determinations made in this paper, the probability (F test) that a straight line will describe the data for each run exceeded 95%.

Material. Foam determinations were made on whole milk concentrates prepared in a recirculating falling film vacuum batch evaporator. The feed milk was obtained as a pooled market milk from a local dairy and consisted of homogenized whole milk diluted with enough nonfat skimmilk so that the resultant blend of 12% solids contained 26% milk fat on a dry solids basis (3.12% fat, 8.88% SNF). The evaporator was operated at a batch tempera-

ture of 88 F at an absolute pressure of 25 mm of mercury and reduced the fluid milk to $44 \pm 1.5\%$ solids concentrate in approximately 90 min. The solids content of the concentrates employed was dictated in these experiments by the nature of the feed to the vacuum foam drier.

After evaporation the milk concentrate was warmed from 88 to 135 F in approximately 5 min, homogenized at 2,750-500 psi, cooled to 70 F in about 30 min and was ready for foam determinations.

RESULTS

Viscosity-temperature. Figure 3 shows the relationship observed between the two factors, viscosity and temperature of unfoamed milk concentrate, whose effects on foams were studied. Each set of data points on the graph represents an individual concentrate batch and is identified by an experiment number, e.g., FS-7. All of the curves drawn through the individual batch data points are described by the appropriately designated equation tabulated in the legend.
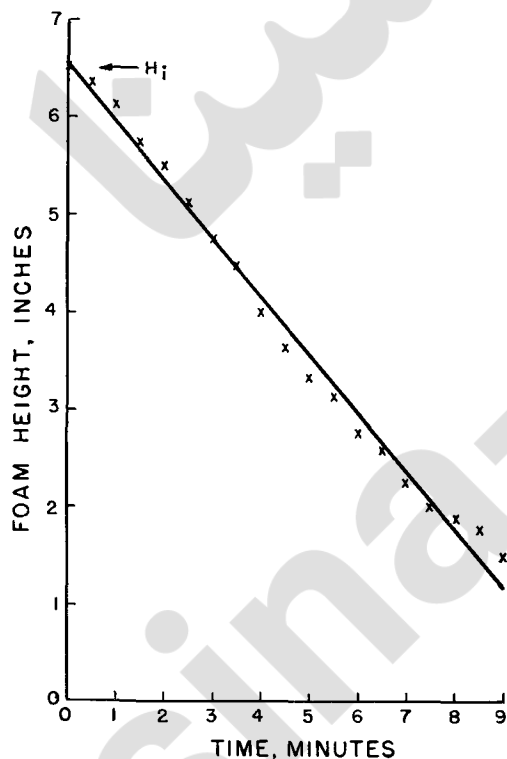

FIG. 2. A foam height-time line showing a typical rate of foam subsidence analysis. The equation from the linear regression analysis is: $H = 6.55 - 0.596\Theta$. In this case R_{fs} is 0.596 in. per minute. Viscosity = 104.5 centipoise. Temperature = 70 F. Run No. FS-9.
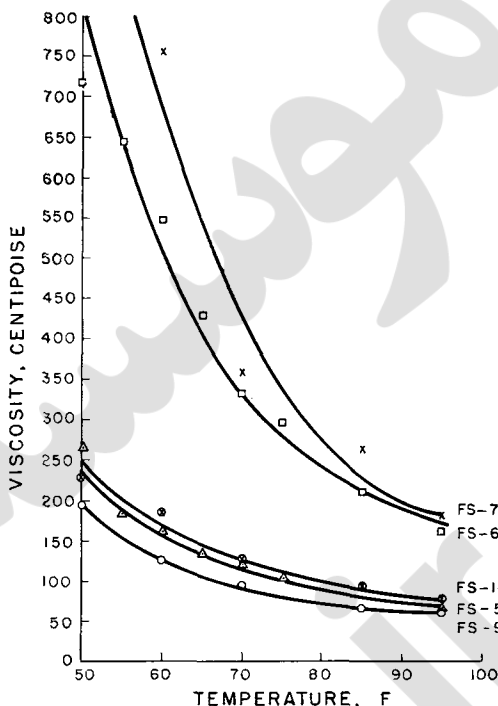

FIG. 3. Viscosity-temperature relationship for whole milk concentrate. The curvilinear regression equations for each run are as follows:

FS-7, $\mu = 411{,}353\,(T\text{-}27)^{-1.83414}$
FS-6, $\mu = 42{,}438\,(T\text{-}27)^{-1.28167}$
FS-10, $\mu = 6{,}844\,(T\text{-}27)^{-1.05430}$
FS-5, $\mu = 8{,}261\,(T\text{-}29)^{-1.14614}$
FS-9, $\mu = 4{,}141\,(T\text{-}29)^{-1.01544}$

It is quite obvious that no simple viscosity-temperature relationship can be derived to describe all of these points and a family of curves is, therefore, employed. However, if no simple relationship correlates these points, at least they were found to be adequately described by one simple mathematical model. The following general exponential model was employed:

$$\eta = a_i\,(T - a_j)^{n_k}$$

where:

η = viscosity of the unfoamed concentrate, centipoise.

T = temperature, F.

n_k and a_i = constants for the individual correlation equations obtained from the curvilinear regression analysis, $[\log\,\eta = \log\,a_i + n_k\,\log\,(T - a_j)]$.

a_j = constant obtained by trial and error so as to give the maximum probability that the equation describes the data to which it is applied (F test).

It was found that, for each concentrate batch, the probability was greater than 95% that the particular functions described the data to which they were applied.

The credibility of these equations is further strengthened by an examination of the equations themselves. Physically, the equations indicate that when concentrate temperatures are lowered to levels near the value of the constant, a_j, the viscosity of the concentrate will approach infinity; a_j is, therefore, that temperature at which the concentrate viscosity is infinity and is an indication of the freezing point of the concentrate. It should be noted that, in all cases, a_j agrees quite well with the observed freezing point of milk concentrate in the range of 44% solids.

A further inspection of the a_j values tabulated in the legend of Figure 3 shows that, for the high viscosity curves, a_j is lower in value than for the low viscosity curves. Since the high viscosity curves may well represent concentrates of higher general solids content (on the + side of the 44% ± 1.5% solids content), the decrease in a_j easily could be indicative of a depression in freezing point in the concentrate batches brought about by slight increases in solids content. This reason might be put forth to explain why a family of curves is necessary to describe the temperature-viscosity relationships in milk concentrate, nominally of the same solids content. However, slight differences in the working of the non-Newtonian milk concentrates and day-to-day variation in chemical constituents also could be contributing factors.

Foam stability-viscosity. Figure 4 shows the foam stability of whole milk concentrate, defined by R_{fs}, as a function of viscosity. The R_{fs} determinations shown on this graph were made on the same concentrate batches shown in

Figure 3. Again, statistical evaluation indicates that the probability that the equation in the legend of Figure 4 describes stability-viscosity data is greater than 95%.

The equation presented as describing the data is another empirically derived exponential similar in form to those equations employed to describe the viscosity temperature relationship of Figure 3. However, in correlating the rate of foam subsidence with viscosity only one equation, not a family of equations, is necessary.

Foaming ability–temperature, viscosity. The foaming ability of whole milk concentrate, described by the initial foam height, is shown in Figure 5 as a function of temperature. Again, each set of points on this graph was determined in conjunction with the viscosity-temperature data displayed in Figure 3 and the temperature determinations are identical on each graph.

An inspection of these data clearly indicates
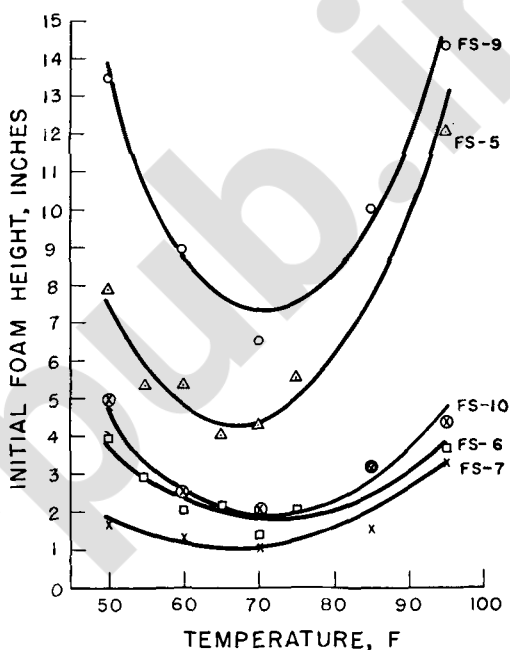
FIG. 5. The effect of temperature on the initial foam height of whole milk concentrate. The parabolic multiple regression equations for each run are:

FS-9, $H_i = 77.477 - 1.960T + 13.650T^2 \times 10^{-3}$;
                   $T_{min} = 71.79$ F
FS-5, $H_i = 49.936 - 1.352T + 10.042T^2 \times 10^{-3}$;
                   $T_{min} = 67.33$ F
FS-10, $H_i = 29.988 - 0.774T + 5.330T^2 \times 10^{-3}$;
                   $T_{min} = 72.61$ F
FS-6, $H_i = 21.924 - 0.559T + 3.900T^2 \times 10^{-3}$;
                   $T_{min} = 71.67$ F
FS-7, $H_i = 13.162 - 0.361T + 2.689T^2 \times 10^{-3}$;
                   $T_{min} = 67.12$ F
              Avg. $T_{min} = 70.10 \pm 3.30$ F

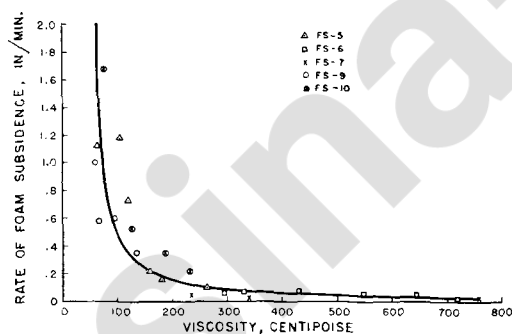FIG. 4. Rate of foam subsidence as a function of viscosity for whole milk concentrate. The curvilinear regression equation is:

$$R_{fs} = 28.24 \, (\mu - 50)^{-1.03555}$$

that the foaming ability of milk concentrate cannot be considered dependent on a single physical property of the concentrate as was the dependence demonstrated between foam stability and concentrate viscosity. Initial foam height as a function of temperature can be analyzed in a manner similar to the concentrate viscosity-temperature relationships, i.e., a family of individual curves. Here again is a case where the apparent slight differences in the concentrate batches have markedly influenced the behavior of the parameters under investigation.

A cursory inspection of the initial foam height–temperature data noted the pronounced tendency for the initial foam height to approach a minimum in the neighborhood of 70 F for each concentrate batch. This factor led to the correlation of the data by means of a mathematical model based on a general parabolic equation of the form

$$H_i = b_i - b_j T + b_k T^2 \text{ (for each batch)}.$$

The data for each batch were, therefore, analyzed as a multiple regression on the above model. Each equation had a better than 95% probability of fitting the data points to which it was applied. The average temperatures corresponding to minimum foam height, obtained by setting the derivative, $\dfrac{dH_i}{dT}$, of each equation equal to zero, is also shown in the legend of Figure 5. It is interesting to note that Rogers (8) has reported a minimum in the tendency for unconcentrated whole milk to foam at temperatures between 68 and 86 F. El-Rafey and Richardson (6) also found a minimum in their foam parameter, half-volume time, in the neighborhood of 70 F. However, half-volume time is generally used as a measure of foam stability but, as pointed out in the measurements section, half-volume time is dependent by definition on initial foam height, i.e., foaming ability.

To describe initial foam height over the entire spectrum of viscosities and temperatures encountered in these experiments, a bivariable mathematical model was derived. This model was based on a relationship observed, at constant temperature, between the individual batch viscosities and the regression coefficients of the parabolas shown in Figure 5. When the model was applied as a multiple regression on all of the initial foam height, viscosity, and temperature data collected, the following equation resulted:

$$H_i = \left[\frac{(206.422T - 3.985T^2 + 0.025T^3 - 2,876.339)}{\eta^{(0.01T + 0.25)}} \right] - 0.714$$

This final equation was found to be meaningful with a confidence level greater than 95%.

DISCUSSION

Experimental results have shown that the apparatus employed to compare the quality of milk concentrate foams clearly described two separate foaming parameters; namely, foaming ability and foam stability. Foam stability for milk concentrate was shown to be an exponential function of the concentrate viscosity, whereas the foaming ability must be described by a complex function of viscosity and temperature.

It has been pointed out (2, 4) that the factors that affect the foam stability of simple surface active systems are: (1) The difference between the surface tension of the solution and that of the pure solvent; (2) the rate of surface tension lowering of a freshly formed surface, and (3) the viscosity of the solution. However, it was found that the foam stability of milk concentrates could be described accurately by considering only how it is affected by viscosity. Therefore, within the scope of these experiments, the effects of surface tension and surface tension lowering must have been negligible when compared to the effects of viscosity. Apparently, the viscosity of the films that form the foam bubble was so high that it controlled the manner in which these films tended to resist the deforming forces that lead to foam collapse.

Experimental results show that viscosity is also an important factor in describing the ability of milk concentrate to form a foam. However, temperature, over and above temperature-induced viscosity effects, must be taken into account, to accurately describe this parameter. Of course, temperature is not a physical property, and it is of interest to speculate what temperature-dependent physical properties other than viscosity are the factors which complete the description of foaming ability. In simple surface-active systems such as detergent-water systems it has been pointed out (2) that the solution surface tension is the property other than viscosity that affects foaming ability. Attempts were made during the course of these experiments to measure concentrate surface tension and include this factor in the correlations. It was found, however, that in the great majority of cases no reproducible measurements of surface tension could be made on milk concentrate, at least with the type of surface-tension-measuring apparatus employed (ring tensiometer, with both liquid-air and liquid-liquid interfaces). However, it should be emphasized

with regard to surface-tension measurements that theories of foam stability applicable to such simple systems as detergent-water often do not apply to films which contain a second liquid or solid present as discrete particles (7, 11), such as encountered in milk with its emulsified fat. In fact, it has been pointed out that the effect of temperature on the foaming properties of unconcentrated fluid milk may be explained not by surface tension but in terms of molecular size, degree of protein hydration, protein solubility, and molecular orientation (6).

The test developed should be a valuable tool for determining the effect that various naturally occurring constituents and surface-active agents have on whole milk concentrate foaming. In fact, studies of this nature are now being undertaken and will be reported upon in later publications.

Significance to the vacuum foam-drying process. Some of these findings can be utilized in understanding and in further developing the process for vacuum drying foamed milk concentrates (1). In this process, the final foam structure greatly influences rate of drying and product quality, especially dispersibility and bulk density. Foams should not be so stable that voluminous structures which require prolonged drying survive.

When the exponential correlation between foam stability and viscosity is considered (Figure 4), it is apparent that a comparatively small zone on the belt adjacent to the nozzle will be critical in determining the final foam structure to be dried. The phenomenon which characterizes the desired foam behavior in this critical zone has been termed controlled subsidence.

It can be seen, from both the foaming ability and foam stability correlations, that viscosity of the concentrate will play a most important role in determining the nature of the foams en-

countered in the feed nozzle and on the belt. Consequently, viscosity is an essential parameter to be controlled in this process.

Since concentrate temperature affects foaming ability and foam stability (insofar as temperature influences viscosity), chamber pressure is of paramount importance in achieving the desired foam behavior and product quality.

REFERENCES

(1) ACETO, N. C., SINNAMON, H. I., SCHOPPET, E. F., AND ESKEW, R. K. Continuous Vacuum Drying of Whole Milk Foam. J. Dairy Sci., 45: 501. 1962.

(2) BARTSCH, O. On Foaming Ability and Surface Tension. Kolloid-Ztschr., 38: 177. 1926 and Chem. Abstrs., 20: 3110. 1926.

(3) BIKERMAN, J. J. The Unit of Foaminess. Trans. Faraday Soc., 34: 634. 1938.

(4) BIKERMAN, J. J. Surface Chemistry for Industrial Research. p. 141. Academic Press, Inc., New York. 1948.

(5) BURCIK, E. J. The Rate of Surface Tension Lowering and Its Role in Foaming. J. Colloid Sci., 5: 421. 1950.

(6) EL-RAFEY, M. S., AND RICHARDSON, G. A. The Role of Surface-Active Constituents Involved in the Foaming of Milk and Certain Milk Products. I. Milk Proteins. J. Dairy Sci., 27: 1. 1944.

(7) PRESTON, W. C., AND RICHARDSON, A. S. The Surface Properties of Soap Solutions. J. Phys. Chem., 33: 1142. 1929.

(8) ROGERS, L. A. Fundamentals of Dairy Science. p. 171-174. Chemical Catalog Co., New York. 1928.

(9) ROSS, S. Foam and Emulsion Stabilities. J. Phys. Chem., 47: 266. 1943.

(10) SANMANN, F. P., AND RUEHE, H. A. Some Factors Influencing the Volume of Foam on Milk. J. Dairy Sci., 13: 48. 1930.

(11) WILSON, R. E., AND RIES, E. D. National Symposium on Colloid Chemistry, First, Colloid Symposium Monograph, 145, University of Wisconsin, Madison. 1923.