OBSERVATIONS ON THE FREEZING POINT OF VACUUM TREATED MILK

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Non-steam injection vacuum flavor removal equipment used in conjunction with high-temperature, short-time pasteurizers results in an elevation of the freezing point at low levels and a depression at high levels of concentration of fluid milk. Regression lines calculated from data representing concentration and freezing point elevation or depression appear to be the best criterion at present to assist with the detection of water adulteration of vacuum treated milk.

The removal of off-flavors from milk is being accomplished, in part, by the use of non-steam injection vacuum equipment in conjunction with high-temperature, short-time (HTST) pasteurizers. The basic principle of this type of equipment is the removal of volatile flavor components by boiling the milk under reduced pressures. A concomitant loss of water results in a concentration of the milk, which increases as the amount of vacuum applied is increased (*5, 6*). The concentration results in a greater solute concentration; thus a depression of the freezing point value should be evidenced in the vacuum treated milk. However, the research of Roberts (*2*) and Henningson and Lazar (*1*) showed an elevation of the freezing point of vacuum treated milk. Preliminary studies by the present authors indicated that this elevation occurs at low levels of concentration of the product. Any deviation from the normal freezing point, when milk is subjected to vacuum treatment, could create a problem for regulatory officials attempting to detect adulterations of milk with water.

A realization of the importance of vacuum treatment equipment to the dairy industry and the apparent changes in freezing point values for milk, which has been vacuum treated, prompted the present study. The basic objective was to determine freezing point ranges for milk subjected to various levels of vacuum treatment in several non-stream injection vacuum systems. The results should be of assistance to regulatory officials attempting to detect watering of vacuum treated milk.

EXPERIMENTAL

Mixed herd milk was pasteurized at 172°F. for 16 sec. by a HTST pasteurizer engineered for 80% regeneration and a capacity of 3450 lbs. per hour. The milk was homogenized at 2200 p.s.i. after pasteurization and vacuum treatment. Vacuum treatment was accomplished using the following modifications of a vacuum installation[1]: (*a*) a single vacuum chamber (with and without a condenser) located between raw regeneration and the timing pump; (*b*) a single vacuum chamber located after the flow-diversion value; and (*c*) a double vacuum chamber with the first chamber located between raw regeneration and the timing pump and the second chamber after the flow-diversion valve. The condenser, in the single instance used, was a 2-in. diameter stainless steel tubular type 4 ft. long, cooled with city water circulating spirally in the outer water jacket. The condenser was installed at a rise of approximately 30° in the vacuum-vapor line as it left the top of the vacuum chamber. The flow of water through the condenser was adjusted to give a 3°F. drop in temperature of the vapors passing through the condenser.

In all installations studied the degrees of flash-cooling or degrees of treatment were determined by noting the temperature differential between the product entering and leaving the vacuum chamber. The degrees of flash-cooling were expressed as percent concentration or milk loss using previously reported formulas (*5*) for the conversion. This transformation to percent concentration was for the purpose of comparing the freezing point data with milk loss, a term which is of more importance to the dairy industry from an operating point of view.

The vacuum systems were operated as they normally would be under commercial conditions. The milk level in the vacuum chamber(s) was maintained by a valve in the product line before the chamber, and/or by adjusting the timing pump to maintain constant product level when viewed through the sight glass of the unit(s). During operation of the equipment a minimum of 15 minutes was allowed for the system to stabilize prior to collection of samples, when a change in degree of flash-cooling was made. In all cases the raw sample was obtained from the balance tank and a 2-min. time lapse allowed for the milk to

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[1]Vacu-Therm made by the DeLaval Separator Company, Poughkeepsie, New York.

travel through the system before collection of the treated sample, which was then taken from a valve in the line after the final cooling section.

Various increments of degrees of flash-cooling were used on each system to collect data over a range of operating levels. Four replicates with three to four different degrees of flash-cooling were studied on each modification. Duplicate freezing point determinations were made on the raw and treated milks with a model F Fiske cryoscope. Raw milk was used as a control instead of conventionally pasteurized milk. Preliminary studies and the work of other investigators (*1*, 3, *4*) indicated that HTST pasteurizers, when properly operated, do not affect the freezing point of milk. Thus the use of raw milk samples for the control in this research was considered valid.

The method of least squares was used for determining the linear relationship between freezing point difference and percent of milk loss. In cases where the relationship was curvilinear the square root of the percent of milk loss was used to make the relationship nearly linear. The standard error of estimate was used as a measure of accuracy of the line (*7*).

RESULTS

The flexibility of flavor removal equipment permits the selection of treatments (flash-cooling degrees) consistent with the intensity of off-flavors to be removed. This flexibility also may be accomplished by the use of various adaptations of the vacuum treatment system. Thus, it was considered advisable to collect freezing point data from four modifications.

Single vacuum chamber before pasteurization

The results of the effect of a single vacuum chamber located between raw regeneration and the timing pump upon the freezing point of milk are plotted in Figure 1. The term freezing point difference used in Figure 1 refers to the elevation or depression obtained from the difference in the freezing point between the raw (control) sample and the vacuum treated sample. The downward trend of freezing point difference with increasing percent concentration of the milk, was represented in the following equation, where Y denotes freezing point difference, and X the percent concentration: $Y=0.0045-0.0056X$. The standard error of estimate for the equation was 0.002°C. The linear regression of these variables was significant ($P<0.01$).

Single vacuum chamber before pasteurization and with a condenser

The equation derived for the effect of a single vacuum chamber located between raw regeneration and the timing pump and with a vapor line condenser on

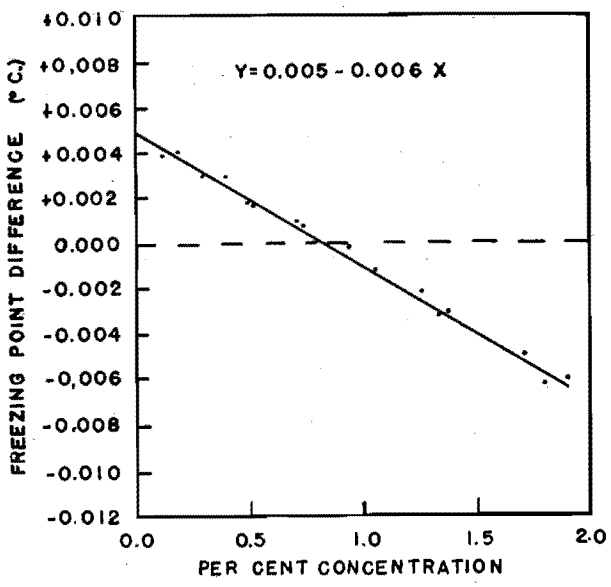

Figure 1. The effect of a single vacuum chamber after raw regeneration upon the freezing point of milk.

the freezing point of milk was $Y_1 = 0.114 - 0.064\,X$, with a standard error of estimate of 0.011°C., where Y_1 denotes the square root of the freezing point difference plus 0.007 ($Y_1 = \sqrt{Y + 0.007}$), Y the freezing point difference, and X the per cent concentration. The regression line, as shown in Figure 2, was plotted from the equation $Y = 0.0060 - 0.0146\,X + 0.0041\,X^2$ which was obtained from the equation for Y_1 by squaring and substracting 0.007. The nonlinear relationship with this vacuum system is due to the effect of the vapor line condenser. The con-

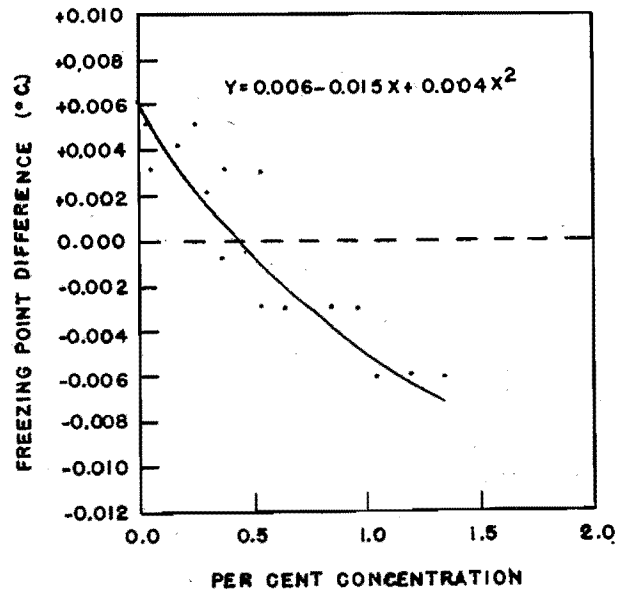

Figure 2. The effect of a single vacuum chamber with a condenser and after raw regeneration upon the freezing point of milk.

densing of some of the vapors and the returning of these condensed vapors to the product resulted in a non-linear relationship between the percent concentration and degrees of flash-cooling (5). Thus, a similar relationship would be expected in plotting such a non-linear concentration effect against freezing point difference.

Single vacuum chamber after final heating

The effect of a single vacuum chamber located after the flow-diversion valve upon the freezing point values of milk is shown in Figure 3. The derived equation was $Y = 0.0031 - 0.0052\,X$, where Y represents the freezing point difference and X the percent concentration. The standard error of estimate was 0.001°C. The freezing point difference associated with percent concentration was significant ($P<0.01$).

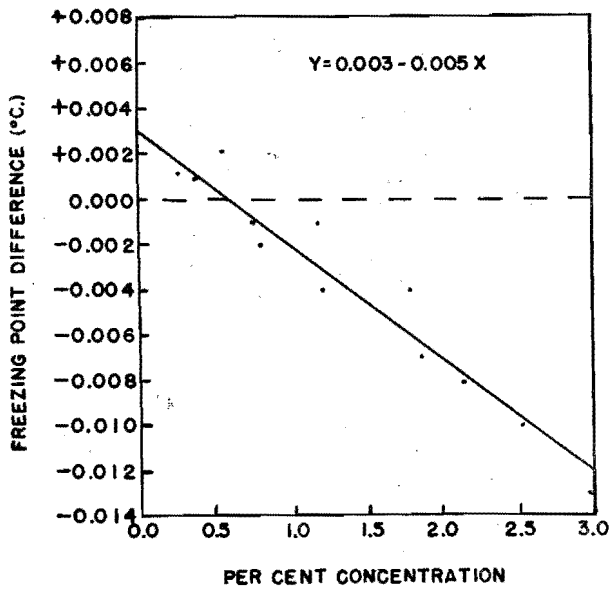

Figure 3. The effect of a single vacuum chamber after the flow-diversion value upon the freezing point of milk.

Double vacuum chamber

The double vacuum chamber unit regression line, as shown in Figure 4, is curvilinear because of the effect of the first vacuum chamber. The travel of vapors in this system is from the second chamber, where milk enters at a temperature controlled by the flow diversion value, to the first chamber, where some condensation of the vapors takes place, and thence to the vapor line to be exhausted. This results in a non-linear concentration effect and when such an effect is plotted against freezing point difference, a non-linear relationship would be expected (5). The regression equation for the double vacuum chamber unit was represented by $Y_1 = 0.157 - 0.044X$, with a standard error of estimate of 0.007°C., where Y_1 denotes the square root of the freezing point difference plus 0.017 ($Y_1 = \sqrt{Y + 0.017}$), Y the freezing point difference, and X the per cent concentration. The regression line, as shown in Figure 4, was plotted from the equation $Y = 0.0076 - 0.0138\,X + 0.0019\,X^2$ which was obtained from the equation for Y_1 by squaring and substracting 0.017.

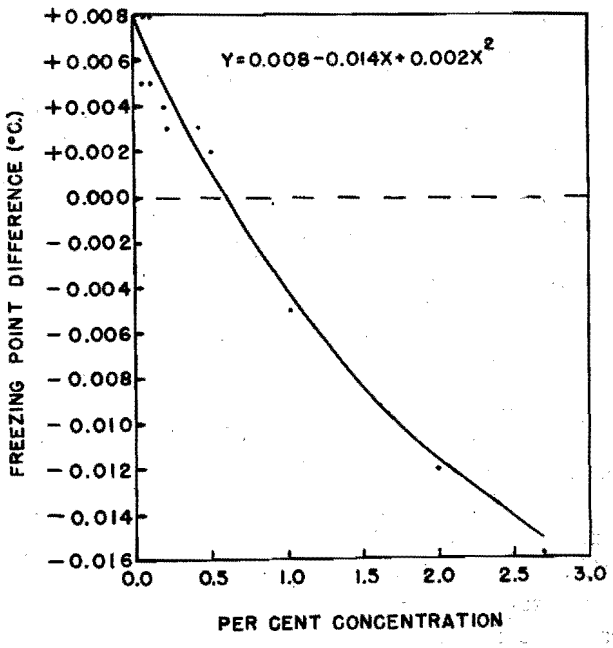

Figure 4. The effect of a double vacuum chamber upon the freezing point of milk.

DISCUSSION

It is apparent from inspection of Figures 1, 2, 3, and 4, that the elevation of the freezing point at zero concentration differs by 0.005°C. between the systems studied. These figures also indicate that the regression lines for the four systems studied are not identical. These variations could be due, in part, to the allowable error of the Fiske cryoscope. However, a more logical explanation is that each system is operated at a different temperature and vacuum for a given percent of milk loss. The theory that the removal of dissolved gases may be a factor in the freezing point elevation at low levels of concentration also offers an explanation for the variation between systems. Carbon dioxide should be removed to a greater or lesser extent depending on the temperature and vacuum applied and the carbon dioxide content of the original milk. The above discussion indicates that a common regression line for all vacuum systems would not be accurate.

In all of the systems studied (Figures 1, 2, 3, 4) the freezing point elevation was overcome as the per cent concentration increased. The levels of concentration which did result in the freezing point eleva-

tion were those most likely to occur in a commercial milk plant. The freezing point elevation creates a problem in that regulatory officials may determine a sample of vacuum treated milk to be adulterated when actually water has not been added to the product. This would be true particularly in cases where the original freezing point value of the raw milk is close to the adulteration point prior to vacuum processing. The realization that with the higher levels of concentration it would be possible to add water to the treated product and obtain a freezing point which approaches that of the original raw product is also important.

The availability of regression lines, similar to those in the Figures, for all types of vacuum systems, should assist in alleviating the problem of detecting small amounts of adulteration with water in vacuum treated milk. The fact that the raw product is not available to the regultory official when a violation is observed could be discounted, as the degrees of flash-cooling used during processing, if known, could be used to determine the percent concentration of the milk (5). The percent concentration could then be used to determine the freezing point elevation or depression from the regression line for the particular system being used. Thus, the freezing point of the vacuum treated sample could be adjusted by the amount of the elevation or depression to estimate the freezing point of the original raw product for the purpose of determining adulteration. Regression lines should be valid for different makes of non-steam injection equipment, since the placement of the unit in the high-temperature, short-time pasteurizing system is the important factor.

Further investigation is needed to more definitely establish the accuracy of freezing point regression lines for determining water in vacuum treated milk. If carbon dioxide is an important factor in the freezing point elevation of such milk, the variable carbon dioxide content of raw milk could result in its non-uniform removal and thus cause a variable elevation of the freezing point of vacuum treated milk. This would decrease the accuracy of detecting adulteration from freezing point regression lines for the purpose of litigation.

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