THE EFFECT OF SOME SALTS AND OAT FLOUR UPON LACTOSE CRYSTALLIZATION IN ICE CREAM

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INTRODUCTION

The purpose of this experiment was to study the effect of some salts and of oat flour upon lactose crystallization in ice cream. Experimental work by Keith (4) indicates that the magnitude of the electrical charge on the active ions in a mix may be an important factor in controlling sandiness in ice cream. The work with oat flour was undertaken in an attempt to check experimental work at the Oklahoma Agricultural Experiment Station, which had indicated that oat flour aggravated lactose crystallization in ice cream.

Before proceeding further, it may be well to consider a brief history of our knowledge of lactose, a few of its properties, its relation to sandiness, and the importance of a smooth texture in ice cream.

In 1619 Bartolettus (16), a philosopher and physician of Mantau, Italy, mentioned butterfat, casein, and whey as the only constituents of milk. In 1628, however, he spoke of a "manna seri" (glass-like food) which he obtained by the evaporation of whey. In 1688, Ettmuller (16) described the evaporation of whey and the purification of the crude lactose by recrystallization.

Ludovico Testi of Venice (16) advertised lactose as an invention of his own and sol d it as a medicine under the name of "saccharum lactis". The identity of the "saccharum lactis" of Testi with the "manna seri" of Bartolettus as pointed out by Fick (16) a few years later.

In 1870 Scheele (9) proved that lactose was a true sugar. This discovery was the first proof of the fact that a true sugar existed in milk. It is present in the milk of all mammals, with the possible exception of that of the whale (9). Uncorroborated reports indicate that it is present in two tropical fruits (9). The lactose content of milk varies from 1.8 per cent in rabbits' milk to 7.56 per cent in mares' milk (16). Normal cows' milk contains about 4.88 per cent. Lactose constitutes about 50 per cent of milk solids-not-fat (13). Experiments conducted by Bert (16) indicate that lactose is formed in the mammary gland by the condensation and internal rearrangement of two molecules of glucose. This internal rearrangement consists of a change of a glucose molecule to a galactose structure, for upon hydrolysis in the laboratory lactose yields a molecule of glucose and a molecule of galactose for every molecule of lactose present (16).

Lactose (9) is a disaccharide sugar with the empirical formula $C_{12}H_{24}O_{12}$, which is the same empirical formula as maltose and sucrose. The disaccharide sugars differ only in the internal arrangement of the atoms; this factor, however, is responsible for several important differences in properties, one of which is the low solubility of lactose compared to other sugars.

If lactose hydrate is added to water at 30° C., it immediately becomes saturated with alpha-lactose. The quantity which dissolves at this point is called the initial solubility (15). Solution, however, is not complete at this stage, for some of the alpha-lactose changes to beta-lactose. This factor disturbs the alpha-lactose equilibrium and more of it enters into solution. This process goes on until there are 1.58 parts beta- to 1 part alpha-lactose (9). At this stage there is an equilibrium between the two forms of lactose and the point which Hudson (15) calls the final solubility of lactose.

If a solution in which lactose has reached its final solubility were cooled to 28° C., one would expect some of the lactose to crystallize out; crystals, however, do not form but rather a state of supersaturation is reached. Ostwald (15) calls this state the meta-stable area. Lactose probably occurs in this state in ordinary commercial ice cream. If the solution were cooled further, crystals of lactose would probably form. Ostwald (15) calls this area the labile area. The line which divides the meta-stable area and the labile area cannot be definitely pointed out in ice cream mixes.

Perhaps crystals which gave the physical appearance of sand in ice cream had been observed years ago, but it was not until recently that the identification of these crystals was definitely established. In 1920, Bothell worked with some ice cream which contained hard crystals that did not dissolve at once in the mouth (1). Although he offered no proof, he stated that these crystals were lactose crystals. His statement was corroborated by Zoller and Williams (17) in 1921. These investigators conceived the idea of isolating the crystals by centrifuging the melted ice cream at high speeds (1). This they did and identified the rhombic prisms as almost pure lactose. Thus it became definitely established that lactose crystallization produces a coarse texture in ice cream.

The American Dairy Science Association score card for ice cream allots 25 points for body and texture. Body and texture are so closely related that it is difficult to differentiate between the two. Sommer (10) states that texture is the attribute of a substance relating to its finer structure - the size, shape, and arrangement of the small particles. The same author defines body as the attribute of a substance relating to the mass as a whole - its consistency or firmness and, in the case of ice cream, its melting resistance. Turnbow and Raffetto (14) state that coarse, snowy, buttery, or sandy texture seriously impairs the flavor of a frozen product. To an average consumer body and texture are fully as important as flavor (14). Off-flavors often elude the most skillful judges, but any one can detect a coarse, sandy texture. Ice cream manufacturers report that texture, more than any other factor on the score card, is a source of complaint (14). A smooth texture is an indispensible attribute to high quality ice cream; any factor which tends to produce a coarse, sandy product becomes a factor which the experienced manufacturer strives to eliminate.

There are several defects which texture may undergo. Among the most important of these defects Sommer (10) mentions coarse or icy texture, snowy or flaky texture, buttery texture, and sandy texture. Only the latter defect is treated in this report.

In the hardened ice cream made of the average commercial ice cream mix, the solution of lactose-in-water always passes the point of final solubility (10). This has been found to be true even in ice cream with 6 per cent serum solids and 30 per cent total solids (10).

Very small crystals which cannot be detected in the mouth frequently form in ice cream. This type of crystal is not objectionable; occasionally, however, the crystals become large and leave a gritty sensation in the mouth as though the ice cream contained sand. These crystals linger on the palate and between the teeth due to the difficulty with which lactose enters into solution. This defect is appropriately designated by the term "sandiness". Although sandiness is not a serious defect, it impairs the texture of the ice cream and is objected to by the consumer; therefore, every precaution should be taken to prevent it.

Hunziker (3) states that the diameter of lactose crystals in sweetened condensed milk must not exceed 12 microns if the product is to have a smooth texture. According to this author crystals which exceed 30 microns in diameter produce an objectionably sandy texture. A similar condition with respect to ice and lactose crystals probably exists in ice cream.

In ordinary commercial ice cream mixes there are about 62 per cent water and 38 per cent total solids. Lactose crystallization cannot occur in the mix due to the high per cent of water which is present. Williams and Peters (15), however, have shown that the final solubility of lactose is diminished in sucrose solutions, and all ice cream mixes contain about 14 per cent sucrose. Whitaker (15), however, states that some of the ionized salts may increase the solubility of lactose or may, by their electrical charges, prevent crystallization. At the Oklahoma Agricultural Experiment Station ice cream with 12 per cent serum solids seldom develops sandiness, but in other localities sandiness is a common occurrence in ice cream made from mixes with 12 per cent serum solids. These mixes apparently have exactly the same composition, with a possible exception of the salt content. These observations, together with conflicting experimental results on sandiness in nut ice cream, by Keith (4), Reid (6), and Dahle (2) have led the author to make this study upon the effect of some salts.

REVIEW OF LITERATURE

The literature indicates that much experimental work has been undertaken in attempts to control lactose crystallization in ice cream, since Bothell's (1) discovery in 1920. Some of the literature which has been encountered while pursuing this study is briefly discussed in the following pages.

Bothell (15) was the first to call attention to the fact that sandiness may be controlled by limiting the serum solids content of the mix. Although Sommer (10) has shown that in a mix with only 6 per cent serum solids and 30 per cent total solids the solution of lactose-in-water always passes the point of final solubility after the mix is frozen, the serum solids concentration is an important factor in controlling sandiness. At the Oklahoma Agricultural Experiment Station ice cream with 12 per cent serum solids seldom develops sandiness, but the author was able to secure a sandy product in a few days with ice cream containing 14 per cent serum solids.

It is impossible to state a definite per cent of serum solids at which sandiness will appear under all conditions. The percentage of milk solids-not-fat must have a definite relationship to the free water present (13). Dahle (13) states that the percentage of lactose in free water cannot exceed 8.7 per cent based upon a total solids content of 36.5 per cent in the mix.

The treatment of the mix should be varied to some extent with the treatment given the ice cream. Whitaker (15) suggests that the composition of the mix be varied with treatment as is shown in the following table.

Maximum percentage of milk-solids-not-fat to avoid sand defect under various conditions

Treatment	Per cent solids-not-fat to use with 12% fat and 15% sugar	Lactose to water ratio
Rapid turn over-sold in a few days	11.3	1 : 11
Held 2 weeks	10.5	1 : 11.9
Held 4 weeks	10.0	1 : 12.6

In calculating the per cent serum solids for a mix, it is always assumed that the skimmilk which is added contains 9 per cent serum solids. Sommer (11) points out that the solids-not-fat content of milk varies with age of the cow, breed, and period of lactation. The per cent serum solids of milk is always higher in the winter months, and this fact should receive some consideration in calculating the winter ice cream mix (12).

Hunziker (3) has shown that sucrose in sweetened condensed milk decreases the solubility of lactose. This condition is probably true in ice cream. According to the fundamental laws of physical chemistry, fat and gelatin tend to produce sandiness inasmuch as they increase the concentration of total solids. On the other hand, they increase the viscosity and reduce the kinetic motion of particles within the mix; therefore, they are important factors in preventing sandiness (10).

In the average commercial ice cream mix there is about 62 per cent water and 38 per cent total solids. Mixes do not become sandy due to the high per cent of water which is present. In the freezer, however, some of the water is frozen into ice. This increases the concentration of total solids and of lactose in the remaining water. Whitaker (15), however, points out that crystallization of lactose seldom occurs in the freezer because the point of final solubility of lactose is not reached above 21° F. With freezer temperatures below 21° F., crystallization of lactose may occur in the freezer.

In the freezer also there are other factors which should not be overlooked. If the mix has to be agitated a long time to secure the proper overrun, the tendency for crystallization is greater since agitation promotes nuclei formation. Whitaker (15) has demonstrated this fact experimentally and recorded the results in the following table.

Influence of temperature and length of time of whipping ice cream mix in freezer on rate of appearance of lactose crystals

Temperature of ice cream in freezer	Time ice cream was removed from freezer: Immediately on reaching desired temperature	After whipping for 5 minutes at desired temperature	After whipping for 10 minutes at desired temperature
° F.	Days to crystallize	Days to crystallize	Days to crystallize
24	20	11	14
23	25	17	14
22	25	14	10
21	8	8	6

Influence of Rate of Hardening on Lactose Crystallization

Ice cream is drawn from the freezer at 22-24° F. (-5.5 to -4.4° C.). When it is placed in the hardening room, the temperature decreases to 0° F. (-18° C.) or less. The time required to reach this temperature depends upon the amount of water frozen into ice in the freezer, type of container, and method of stacking (15). Bayer (15) showed that quick hardening tended to retard the crystallization of lactose.

The influence of rate of hardening of ice cream on lactose crystallization

Hours to reach 0° F.	Days to first lactose crystals	
	Experiment 1	Experiment 2
1	51	32
15	44	25
28	37	19

In order for crystallization to occur, the molecules of lactose must move together in the ice cream. This kinetic motion of molecules is greatly reduced at low temperatures due to the loss of translational energy and increased viscosity of the solution.

Influence of Method of Packaging on the Development of Sandiness

Whitaker (15) prepared three different types of packages of ice cream, namely: five-gallon cans, small packages which were filled by hand, and freezer drawn packages. He found that bulk ice cream showed sandiness in 27 days, hand packaged in 11 days, and machine packaged in 18 days.

The probable explanation of this fact is that the bulk ice cream experienced less temperature fluctuation. The bulk goods also hardened more slowly. But these factors were more than off-set by the temperature fluctuation and agitation incident to hand packaging.

Crystallization in Containers

Crystallization is usually uniform throughout the container. Occasionally, however, it may occur only in the center due to slow cooling of the ice cream, or it may appear only on the outside due to the effect of temperature fluctuations (15).

Influence of Temperature Fluctuation on the Development of Sandiness

This factor is often called "heat shocking", but the term is misleading since it implies a sudden temperature change. Temperature fluctuation is a better term. Raising and lowering the temperature tends to produce lactose crystallization. This factor was quite a problem with the old salt and ice cabinet. The electric cabinet, however, has eliminated this factor to some extent. When the temperature of ice cream is raised, some of the ice melts. If lactose crystals are present, the smallest ones may completely dissolve in this water. If the temperature is lowered, the water again goes into ice. The lactose which went into solution crystallizes again, but this time it may be deposited on larger crystals which did not completely dissolve. Thus the lactose crystals grow.

Effect of Mechanical Shocks on Lactose Crystallization in Ice Cream

Any sudden shock due to bumping, shaking, falling on the floor, or hauling in trucks or cabinets tends to hasten the development of sandiness (15). A shock results in a sudden movement of the liquid in the frozen mass which may result in collision of lactose crystals or molecules.

Effect of Nuclei on the Rate of Lactose Crystallization

The presence of nuclei in ice cream promotes lactose crystallization. Nuclei with sharp angular structures are the best seeding agents. This factor has been demonstrated experimentally by Whitaker (15). (See table below).

Influence of insoluble foreign material on rate of lactose crystallization in ice cream

Material added	Days to first appearance of lactose crystals	
	Cabinet 1	Cabinet 2
	days	days
Nothing	None at 28	None at 28
Asbestine	21	lost
Sand	23	21
Kaolin	23	17
Ground glass	17	13
Carborundrum	23	17

Nuclei serve as centers on which the lactose molecules may crystallize. Occasionally crystals of lactose and of inorganic salts in milk powder and condensed milk may not completely dissolve in processing the mix. Such crystals may serve as nuclei in the ice cream and precipitate an outbreak of sandiness.

It is generally agreed that ice cream containing nut meats develops sandiness more frequently than any other flavor. This peculiarity has drawn the attention of Reid (6), Dahle (2), Keith, Fouts, and Weaver (4). Reid (7) believes that the nuts serve as nuclei which induce premature lactose crystallization. He delayed sandiness in ice cream by washing the nuts in warm water and autoclaving under steam pressure and also by soaking the nuts in a gelatin solution prior to using in the ice cream (6). Dahle (2) delayed sandiness by washing the nuts in a sugar syrup before using in the ice cream. Reid (7) maintained that this had little or no effect.

This controversy attracted the attention of Keith, Fouts, and Weaver (4). They treated nuts with hot and cold sugar syrup solutions and altered the salt balance of the mixes, using calcium chloride and sodium citrate. In cases where calcium chloride was added, all samples were sandy. These findings are in accord with those of Reid. On the other hand, when sodium citrate was added, mixed results were obtained. Sandiness did not appear in the ice cream which contained no salts. These results bear out Dahle's work. The salt balance apparently influenced the development of sandiness.

Whitaker (15) believes that dry nuts cause sandiness because they imbibe water, thereby increasing the lactose in water concentration. This factor automatically hastens the development of sandiness.

Effect of Fat Clumping on Development of Sandiness in Ice Cream

Sommer (10) states that mixes in which the fat globules have clumped have a higher viscosity than mixes in which the fat globules have not clumped. The kinetic movement of the unfrozen materials in ice cream must be greatly influenced by the viscosity of the mix. In an exceedingly viscous mix, one would expect the movement to be retarded. In order for ice cream to become objectionably sandy the molecules or small crystals of lactose must move together; therefore, fat clumping should tend to prevent sandiness.

Whitaker (15) has demonstrated this experimentally. A mix which showed fat clumping on microscopic examination and one of the same composition, but which showed no fat globule clumping, were frozen and stored in electric cabinets under identical conditions. The data collected in one such experiment are shown in the following table.

Influence of fat globule clumping on the rate of lactose crystallization in ice cream

Fat globules	Viscosity	Time of appearance of lactose crystallization	
		Cabinet 1	Cabinet 2
	sec.	days	days
Not clumped	$16\frac{1}{2}$	11	17
Clumped	161	31	31

Lucas and Spitzer (5) have found that lactose crystallization is neither prevented nor affected so far as can be determined by high or low pasteurizing temperatures.

Nothing was encountered in the literature which indicated that neutralizers, per cent overrun, per cent acidity, or protein stability had any effect upon the development of sandiness.

PLAN OF STUDY

All dairy products used in this experiment were of a uniformly high quality. The skimmilk powder, which was made by the spray process of manufacture, was about two months old. The skimmilk and cream were less than two days old at time of use and were free from pronounced off-flavors and odors and extraneous matter. All dairy products except the milk powder were secured from the college dairy herd.

The sugar used was finely granulated sucrose, free from foreign materials. The gelatin was of a high quality, free from lumps and foreign substances. In cases where oat flour was added, Avenex No. 7 was employed. This flour was made by grinding the oat kernel, after the hull had been removed.

In the course of this study four trials were made at weekly intervals in order to check the results secured from each trial. In each trial two 40-pound batches (numbered batch I and batch II) of mix were made and each batch was divided into three 12-pound lots. The mixes of both batches were numbered 1, 2, 3, 4, 5, 6. Batch No. I was always divided into mixes 1, 2, 3; and batch No. II was always divided into mixes 4, 5, 6.

The composition of each batch was calculated so as to correct the dilution error which resulted when water, salt solutions, and oat flour were added. The total solids content was the same in the frozen ice cream of all mixes. A volume of water exactly equal to the volume of salt solution was added to each control mix. The oat flour was used to replace part of the serum solids content in the mixes where it was used. The increased concentration due to the salts which were added was such a small factor that it was disregarded.

The composition of each frozen mix in the four trials is shown in the table below.

Table I

Composition of mixes

Mix No.	% Fat Trial I	% Fat Trials II, III, IV	% Serum Solids Trial I	% Serum Solids Trials II, III, IV	% Sugar Trials I, II, III, IV	% Gelatin Trials I, II, III, IV	% Oat Flour Trials I, II, III, IV
1	10	12	14	12	14	.2	
2	10	12	14	12	14	.2	
3	10	12	14	12	14	.2	
4	10	12	13.6	11.6	14	.2	.4
5	10	12	13.6	11.6	14	.2	.4
4	10	12	13.6	11.6	14	.2	.4

The per cent of fat and the per cent of serum solids were changed in trials II, III, and IV, because sandiness developed within three days after freezing in the ice cream of the first trial. The observation which led to this discovery was not a part of the original plan of the study.

Two salts, calcium chloride ($CaCl_2$) and sodium citrate ($2Na_3C_6H_5O_7 \cdot 11H_2O$), were used in this experimental work. Water solutions of these salts were prepared so that equal volumes of each solution would contain the same number of active ions. The calcium and citrate ions are the active ions in these salts. In very dilute solutions each molecule of calcium chloride breaks down into one positively charged calcium ion (Ca^{++}) and two negatively charged chloride ions (Cl^-); sodium citrate yields six sodium ions (Na^+) and two citrate ions ($C_6H_5O_7^{\equiv}$). A molar solution (147.02 grams per liter) of calcium chloride and a one-half molar solution (357.16 grams per liter) of sodium citrate were prepared. Ionization of these salts should have been complete when they were added to the ice cream mixes due to the high dilution which resulted; therefore, mixes to which the salts were added should have contained equal numbers of calcium and citrate ions.

The important difference in the salts was in the sign of the charges of their active ions. The calcium ion, carrying a positive charge, is very active while the chloride ion is relatively inert; in sodium citrate, however, the negatively charged citrate ion is the active factor.

The 12-pound mixes of each trial were placed in tall, three-gallon cans. The salts were added to the mixes of each trial, as is shown in table II. Mixes 1 and 4 were used as controls. All materials were added immediately before pasteurization.

Table II

Materials added to mixes of each trial

	Number of mix					
	1	2	3	4	5	6
Ml. water	30			30		
Ml. calcium chloride		30			30	
Ml. sodium citrate			30			30

It should be recalled here that mixes 4, 5, and 6 contained oat flour in addition to the above materials. Mixes 1, 2, 3 contained no oat flour.

After the salt solutions were added, the mixes were heated in a water bath to 160° F. for 20 minutes. The temperature in the bath was regulated by introducing steam and cold water. Mixes were introduced into the bath at five minute intervals to prevent delay at the homogenizer.

At the end of the 20 minute period of pasteurization, each mix was homogenized in a two stage homogenizer, using 2500 pounds of pressure on the first valve and 500 on the second. About 6 pounds of properly homogenized mix was obtained from each 12-pound lot. The homogenizer was flushed with hot water after homogenizing each mix to prevent contamination of the succeeding mix.

Immediately after all mixes were homogenized, they were cooled to 45° F. over a tubular cooler with circulating brine.

After aging for 18 hours at 40-45° F., the mixes were frozen in the freezing unit shown in figure I.

This freezing unit was designed by J. I. Keith and C. W. Rink and constructed by the Department of Industrial Arts Education and Engineering Shop Work of the Oklahoma Agricultural and Mechanical College (8).

The unit is essentially a 4' x 4' brine box with four one-gallon freezers set in it. The pinions from each freezer all engage a main driving gear in the center of the box. An electric motor, through the V-belts, pulleys, and shafts, revolves the main gear. The one-gallon cans are turned at the rate of 90 r. p. m.; the scrapers on the inside revolve in the opposite direction at the same speed. This would be equivalent to revolving the dashers at 180 r. p. m. if the cans were stationary.

Only three of the one-gallon freezers were used in freezing. The cans were exactly half-filled (about 4½ pounds) with mix and placed in position. From a large tank brine at 18° F. was siphoned by means of a rubber tube into the brine box. When the box became about two-thirds filled, the brine solution passed through an overflow pipe into a small galvanized tank. From here it was pumped back into the original brine tank. As soon as the brine solution started passing through the overflow pipe, the electric motor was started and the mixes were frozen to 100 per cent overrun, i. e., until the ice cream in the cans became level with the top.

When the mix reached 100 per cent overrun, it was removed from the freezer and placed in three-ounce, paper cups which were numbered 1 to 24.

Cups 1 to 12 were placed together, and cups 13 to 24 were placed together in storage. This procedure was followed with each of the mixes of all four trials. All samples were stored in a hardening room at -10° F.

At weekly intervals samples 1 to 12 of each mix were removed to an ante-room at 40° F. for one hour. These samples were then returned to the

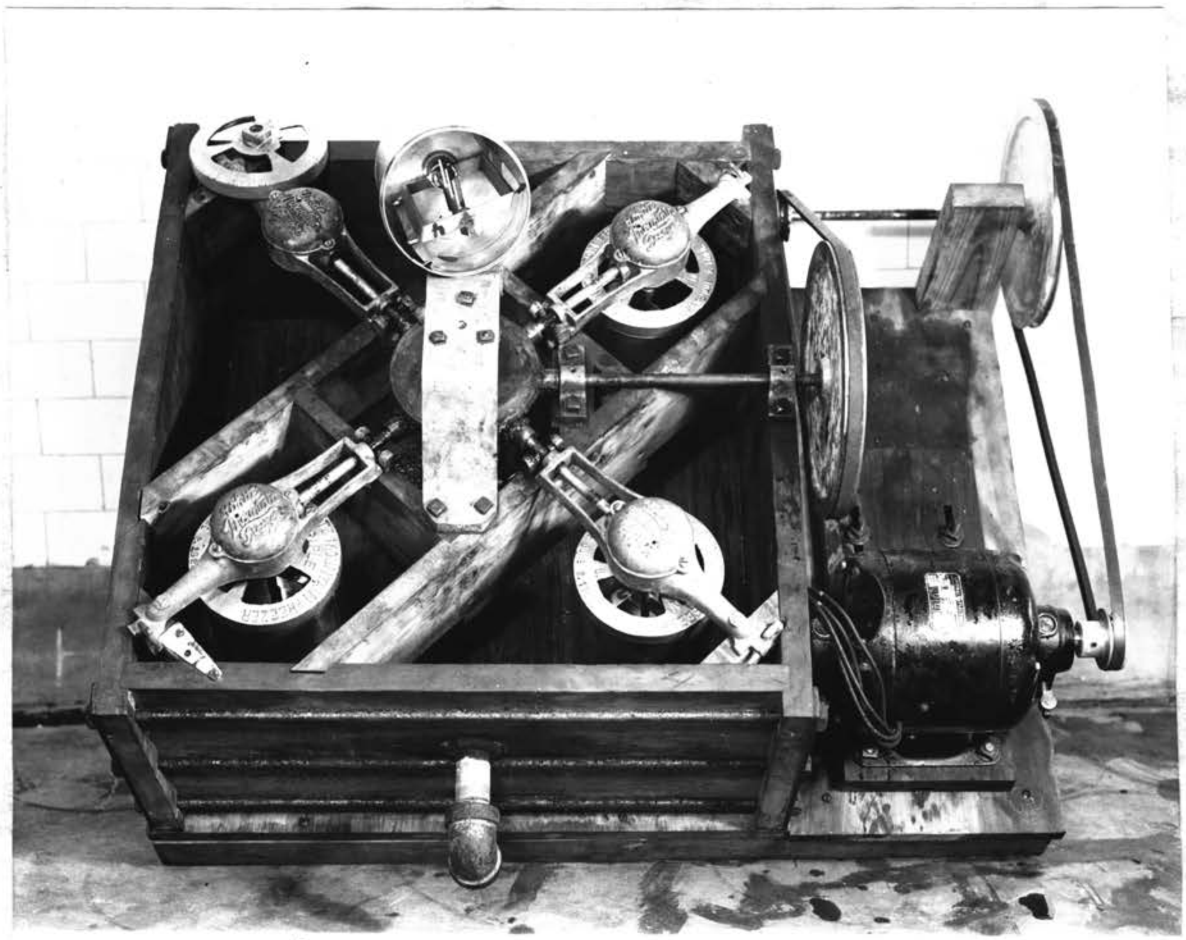

Figure I. Experimental ice cream freezing unit.

hardening room. Samples 13 to 24 were not subjected to such temperature fluctuations.

Samples of mix for laboratory determinations were taken at the end of the aging period. Acidity tests, viscosity determinations, fat clumping determinations, and protein stability tests were made on the mixes.

Acidity Tests. Nine gram samples from each mix were titrated with N/10 sodium hydroxide. The following formula was used to calculate the per cent acid in the mix.

$$\% \text{ acid} = \frac{\text{Ml. N/10 NaOH used x .009}}{\text{Wt. of sample in grams}} \times 100$$

The samples were weighed on a Torsion balance into 150 ml. Erlenmeyer flasks. The sodium hydroxide was dispensed from an automatic burette. Phenolphthalein was used to determine when the end point had been reached. Two results which checked within .05 per cent were secured with each mix.

Viscosity Determination. Although there are several special instruments for determining viscosity, fairly consistent results may be obtained by using a 10 ml. pipette and a stop watch. The pipette was made stationary by clamping it to an iron stand. Distilled water was then drawn up into the pipette to the zero mark, and the time for 9 ml. to flow out was recorded by the stop watch. Rink (8) found that a greater variation in time occurred when the tenth cubic centimeter was allowed to flow through the tip of the pipette. He believed that this was due to the reduced head pressure and the curved sides at the bottom of the pipette. After the time for water had been determined by three or more trials, the mix to be tested was drawn up into the pipette, and the time for 9 ml. to flow out was recorded. Three results which checked very closely were secured. The viscosity of the mix was then calculated by the following formula:

$$\text{Viscosity of mix} = \frac{\text{Time in seconds for mix}}{\text{Time in seconds for water}}$$

The pipette was washed and dried when changing from one mix to another. All viscosity determinations were made at 70° F.

Fat Clumping. To determine the degree of fat clumping 1 ml. of the mix was added to a 300 ml. bottle and it was filled with distilled water. A hanging drop was prepared from the above solution and examined under a microscope, using a 4 mm. objective and a 10x eyepiece. The degree of fat clumping was recorded as none, evident, very evident, prominent, very prominent, pronounced, or very pronounced. In a mix with no clumping the globules appear as distinct individuals. In the case of clumping the globules are distinct but appear as conglomerated masses which have no definite shape or form.

Protein Stability Tests. To determine the protein stability 5 ml. samples were used. A series of test tubes were scratched at the 5 ml. mark with a glass cutter. The mix was introduced into the test tube with a piece of glass tubing, using the glass tube as a pipette. The high viscosity of ice cream mixes prohibits the satisfactory use of a pipette. Ninety-five per cent ethyl alcohol and N/20 hydrochloric acid were used in this test.

To determine the stability of the proteins toward ethyl alcohol, the 5 ml. sample of mix was diluted with less than 10 ml. of water. The added volume was then made to 10 ml. with alcohol. The test tube was inverted quickly three times, and the walls of the tube were examined for a precipitate. The stability of the mix toward alcohol was expressed as the number of milliliters of alcohol which were required to produce a faint precipitate on the walls of the test tube.

The acid stability test was performed in much the same manner as was the alcohol test. All reagents, water, alcohol, and acid, were dispensed from burettes. These tests were performed at room temperature which was about 70° F.

Scoring the Ice Cream. The ice cream samples were scored for flavor and body and texture by three judges, who were members of the faculty of the Dairy Department at Oklahoma Agricultural and Mechanical College. When the samples of the first trial were two weeks old, all number 1 samples were scored; thereafter, the samples in numerical order, were scored at weekly intervals. When sandiness was encountered in the ice cream from any lot, sample number 13 of that lot was removed from the hardening room and scored. The purpose of this practice was to determine whether the temperature fluctuation hastened the development of sandiness. The samples from the mixes of each trial were scored in this manner.

The judges were given no information regarding the identity of the samples. Scores of the three judges for each sample were averaged and recorded. Particular attention was given to sandy texture and oxidized flavor; criticisms on these defects were recorded. The criticism of each judge on each sample was counted as one observation.

EXPERIMENTAL RESULTS AND DISCUSSION

All ice cream mixes in this experiment were pasteurized, homogenized, cooled, and frozen in the manner which has been described. Whether or not any of the added salts were precipitated in pasteurization was not determined. All mixes froze to 100 per cent overrun in 9 to 45 minutes. The time required to obtain the desired overrun varied with the position in the brine box and the condition of the gears, dashers, and scrapers. These factors were of such magnitude that the effect of the salts on the speed of whipping could not be studied.

In all laboratory tests which were made in this study two or more results which checked very closely were secured.

Acidity tests for mixes of all trials checked fairly closely. So far as could be determined in the College Dairy laboratory, distilled water solutions of calcium chloride, sodium citrate, and oat flour were neutral in reaction. Mixes which contained calcium chloride gave a trifle higher acidity than other mixes, although this factor was within the limits of experimental error encountered while titrating with a burette. In no case did the acidity of any mix exceed .31 per cent; the average for all mixes was .28 per cent acid.

A great variation in viscosity appeared between the mixes of each trial and between the same mixes of different trials. The factors which may have been responsible for this occurrence will not be discussed here. In all trials, however, it was observed that the calcium salt increased the viscosity. Some of these mixes were so viscous that they flowed only with great difficulty. A chemical change induced by the calcium chloride may be responsible for this occurrence. The increased viscosity, however, did not retard the development of sandiness as will be noticed in the following pages. The mixes containing sodium citrate and oat flour showed variable viscosities when compared with the control mixes.

Very little fat clumping appeared in any mix in this experiment. Four mixes were marked as "evident". No correlation between the degree of fat clumping and the development of sandiness was observed in this study.

The added salts exerted a pronounced effect upon the protein stability. Calcium chloride decreased the stability of the proteins toward ethyl alcohol and increased the stability toward hydrochloric acid. Sodium citrate exerted opposite effects, increasing the stability toward alcohol and decreasing the stability toward acid (see table III).

Table III

Effect of calcium chloride and sodium citrate on protein stability

Type of mix	Milliliters required to produce a precipitate	
	95% ethyl alcohol	N/20 hydrochloric acid
Control mix	6.3	3.6
Mix containing calcium chloride	4.2	4.1
Mix containing sodium citrate	8.1	3.0

The proteins of milk exist in the form of a sol. The stability of a sol is greatly influenced by the electrical charge on the ions which are adsorbed at the surface of colloidal particles. At their iso-electric points - the point at which the charge on the particle equals the charge of the medium - the particles of a sol are precipitated most easily.

Calcium and citrate ions change the ionic equilibria of the ice cream mix. Calcium ions impart a positive charge; citrate ions, however, contribute a negative charge. When positive ions are added to an ice cream mix, the stability of the proteins toward ethyl alcohol is reduced, but the stability toward hydrochloric acid is increased. Increased stability toward alcohol and decreased stability toward hydrochloric acid may be expected when negative ions are added.

Of the 576 samples which were prepared 452 were scored and the results recorded. Of the 452 samples 150 were scored as sandy and 135 as oxidized. (See table IV).

Table IV

Number of samples scored as having a sandy texture and oxidized flavor

No. of samples scored as		No. of judges scoring sample as		Total number of observations	
Sandy	Oxidized	Sandy	Oxidized	Sandy	Oxidized
83	18	3	3	249	54
37	38	2	2	74	76
30	79	1	1	30	79
Total: 150	135			353	209

The figures of the third column were secured by counting the report of each judge as one observation. Sandy texture and oxidized flavor appeared in the ice cream of each trial as shown in table V.

Table V

Appearance of sandy texture and oxidized flavor in the ice cream of each trial according to the total number of observations

Trial No.	Number of Mix 1		2		3		4		5		6	
	Sandy	Oxidized	Sandy	Oxidized	Sandy	Oxidized	Sandy	Oxidized	Sandy	Oxidized	Sandy	Oxidized
1	53	19	68	18	45	39	31	19	37	13	39	4
2	0	10	4	3	0	4	9	2	1	4	0	2
3	12	9	14	11	3	2	7	9	3	18	0	4
4	0	6	0	8	12	2	3	1	11	4	1	0
Total:	65	44	86	40	60	45	50	31	52	39	40	10

Table V shows that sandiness and oxidized flavor appeared in each type of ice cream in one or more trials. Although no sure methods for preventing these defects were revealed, some factors appeared which are worthy of closer study.

The results concerning sandiness and oxidized flavor in all four trials have been summarized in the following table.

Table VI

Appearance of sandiness and oxidized flavor in the ice cream of all trials

Mix number	1	2	3	4	5	6
Observations of sandiness	65	86	60	50	52	40
Observations of oxidized flavor	44	40	45	31	39	10

By comparing mixes 2 and 5 with 1 and 4 it will be noted that sandiness was much more pronounced in ice cream to which calcium chloride had been added. It may be observed also by comparing mixes 3 and 6 with 1 and 4 that sodium citrate tended to inhibit the development of sandiness. The effect of the calcium ion was off-set to a great extent by the oat flour as will be noted by comparing mixes 4 and 5. This may have been due to a chemical combination of the active calcium ion with some constituent of oat flour.

Oat flour and a combination of oat flour and sodium citrate inhibited the development of oxidized flavor as may be observed by comparing mixes 4 and 6 with mix 1.

The figures in table VII were obtained from table VI. The figures under mixes 1 and 4 were added together; figures under mixes 2 and 5 were added together; figures under mixes 3 and 6 were added together; and, finally, the figures under mixes 1, 2, and 3 were compared with the figures of mixes 4, 5, and 6 to ascertain the effect of the oat flour (See table VII).

Table VII

Effect of calcium chloride, sodium citrate, and oat flour on the development of sandiness and oxidized flavor

Material added	Control	Calcium chloride	Sodium citrate	Oat flour .4% Added	Oat flour None added
Observations of sandiness	115	138	100	142	211
Observations of oxidized flavor	75	79	55	75	134

These figures emphasize the detrimental effects of calcium chloride and the beneficial effects of sodium citrate on the development of sandiness. The oat flour tended to prevent lactose crystallization. This may have been due to the fact that oat flour was used to replace some of the serum solids, which reduced the lactose-in-water concentration. As has been mentioned previously in this report, the higher the lactose-in-water concentration, the greater will be the tendency for lactose to form crystals.

The table shows that oat flour tended to prevent the development of oxidized flavor. The figure in the fourth column indicates that sodium citrate may inhibit the development of oxidized flavor.

The temperature fluctuations which occurred in the hardening room were of such magnitude that the practice of heat shocking yielded negligible results. In fact more cases of sandiness occurred in the group which was not exposed to the severe temperature fluctuations. This was probably influenced by the fact that cups No. 13 to 24 were always filled last which allowed some of the ice cream to melt. The practice of heat shocking apparently inhibited the development of oxidized flavor (See table VIII).

Table VIII

Effect of temperature fluctuations on the development of sandiness and oxidized flavor

	Heat shocked	Not shocked
Observations of sandiness	134	213
Observations of oxidized flavor	99	110

So far as could be ascertained in this study the per cent acidity, degree of fat clumping, viscosity, or protein stability had no effect on the development of oxidized flavor.

CONCLUSIONS

A. Effect of the Calcium Ion.

The effect of the calcium ion on the speed of whipping could not be observed in this study. The calcium ion produced no noticeable effect on the per cent acidity or the degree of fat clumping. A pronounced increase in viscosity was observed which may have been due to a chemical change induced by the calcium chloride. A decrease in protein stability toward alcohol and an increase in stability toward hydrochloric acid was observed. There was an increased tendency for sandiness to develop when calcium ions were added in spite of the increased viscosity. Whether this is due to the electrical charge on the calcium ion or to a chemical change was not determined. This salt apparently had no effect on the development of oxidized flavor.

B. Effect of the Citrate Ion.

The effect of sodium citrate on the speed of whipping could not be studied in this experiment. So far as could be determined in this experiment the sodium salt had no effect on the per cent acidity, degree of fat clumping, or viscosity. This salt produced an increase in protein stability toward alcohol and decreased the stability toward hydrochloric acid. This ion slightly inhibited the development of sandiness and oxidized flavor.

C. Effect of Oat Flour.

The oat flour apparently produced no effect on the per cent acidity, degree of fat clumping, viscosity, or protein stability. It tended to prevent the development of sandiness in ice cream. This may have been due to the fact that it was used to replace part of the serum solids, which reduced the lactose-in-water concentration.

Oat flour tended to inhibit the development of oxidized flavor.

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