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The Relation of Different Ingredients of Ice Cream to its Freezing and Supercooling Points

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When ice cream is frozen, two obvious changes take place in the ice cream mixture: first, a change in the physical nature; and second, a change in the temperature. The change in the temperature is considered to be one of the most important factors in controlling the physical nature of the finished product, particularly its texture.

Zoller (1) studied the freezing process of various ice cream mixtures and ices. He found that: (A) The freezing point lowering was three to five times greater than the theoretical molecular lowering and attributed it to a supercooling phenomenon. (B) There was no direct relation between the temperature of the brine and the amount of supercooling. (C) The crystallization point, or point of ice separation, was not sufficiently sharp for different mixtures to be termed a physical constant. (D) There was a typical L shape to the freezing curve.

Zoller (2) also gives evidence that supercooling can be reduced by the addition of 0.5 per cent gelatine, fine sand, or lactose crystals. He attributes this reduction in supercooling to the rapid separation of ice crystals in a finely divided state by preventing convection, thereby giving more smoothness and a better texture to the finished ice cream. He also found that any substance which would tend to increase the viscosity of the mixture tended to retard supercooling.

Reid and Nelson (3) found that an increase in the milk solids-not-fat lowers the crystallization point of the freezing point. However, the amount of supercooling below the freezing point was not affected by acidity, viscosity or milk solids-not-fat content of the mixture.

Leighton (4) furnishes methods for calculating the freezing points of ice cream mixes and shows how the quantity of ice that may be separated from a mix at any temperature in the freezer can be determined.

Procedure

In studying the process of freezing ice cream our interest is directed especially to the freezing and supercooling points of the ice cream mix. Should these temperatures be below that of the solvent (water), the question arises as to which ingredients comprising the ice cream mixture cause a lowering of the temperature and to what extent are each responsible.

The freezing point of pure water is zero degrees Centigrade or 32 degrees Fahrenheit, whereas the freezing point of ice cream ranges from 1.50 to 3.00 degrees Centigrade lower than that of water, depending upon the composition of the mix. It is known that when any substance is dissolved in water, the resulting solution will require a lower temperature to freeze and as the solution is further concentrated the freezing point will continue to be lowered. It seems then that the depression of ice cream may be due to those ingredients which are water soluble. In the basic ice cream mix, the soluble ingredients are sucrose, lactose and the milk solids.

This investigation has been directed to the determination of the effect of the latter ingredients in lowering the freezing and supercooling points of the respective mixes.

The freezing point of milk has been found to be very constant, namely, —0.55 degrees Centigrade, + or —0.2 degrees Centigrade, irrespective of the composition of the milk. Leighton (4) in his work on the depression of the freezing point has calculated that milk sugar accounts for 0.306 degrees Centigrade of the depression and the balance 0.244 degrees Centigrade is attributed to the milk salts present. Leighton also developed two formulae and learned that sucrose and lactose cause about the same amount of lowering in the freezing point. Assuming that 54.50 per cent of the serum solids in an ice cream mix to be lactose, the following formula would apply:

(A) $$\frac{(\%\ \text{of sucrose \& lactose})}{\%\ \text{water in mix}} \times 100 = \text{Parts sucrose \& lactose per 100 parts water.}$$

In order to determine the extent to which the freezing point is lowered in the particular mix studied, Table 1 is used.

TABLE 1

Parts Sucrose Per 100 parts Water	Freezing Point Lowered Degrees Centigrade
10.84	0.65
15.83	0.95
19.80	1.23
22.58	1.37
25.64	1.58
28.51	1.77
32.22	1.99
35.14	2.15

Leighton's (4) formula for determining the freezing point depression caused by milk salts is as follows:

(B) $$\frac{\%\ \text{of Serum Solids} \times 2.37}{\%\ \text{water in mix}} = \text{lowering due to milk salts.}$$

In the conduct of this investigation the ice cream mixtures used had an average composition of:

Butterfat	12 per cent
Serum Solids	12 per cent
Sucrose	14 per cent
Total Solids	38 per cent

Using the average composition of milk as given by Van Slyke, it was calculated that the serum solids of milk contained 10 per cent of milk salts and 54.50 per cent of lactose, the mixtures were compounded on a basis of 100 grams with the following proportions of water, sucrose, lactose and milk salts:

Water	62 grams
Sucrose	14 grams
Lactose	6.7 grams
Milk Salts	1.2 grams

In order to determine the proper amounts of each individual milk salt to be used it was necessary to construct Table 2 by the application of the above data and that of Van Slyke.

TABLE 2.—CALCULATED AMOUNTS OF INDIVIDUAL MILK SALTS IN AN ICE CREAM MIXTURE.

Milk Salt	Per Cent Milk Ash	Grams per 1000 in Milk	Grams in 100 grams of ice cream mix
Calcium Citrate	23.55	2.133	0.28260
Mono-Pot. Phosphate	12.77	1.156	0.15324
Sodium Chloride	10.62	0.962	0.12744
Potassium Chloride	9.16	0.830	0.10992
Di-Pot. Phosphate	9.22	0.835	0.11064
Tri-Calcium Phosphate	8.90	0.806	0.10680
Di-Calcium Phosphate	7.42	0.671	0.08904
Potassium Citrate	5.47	0.495	0.06523
Calcium Oxide	5.13	0.465	0.06156
Magnesium Citrate	4.05	0.367	0.04860

The freezing point of the different solutions was determined by the application of a Hortvet Cryoscope, using a modified Beckman thermometer. This thermometer has a graduated scale which reads in 0.01 degrees and has a range going from —5 degrees to +1.0 degrees Centigrade. The principle of the Cryoscope is that we have cooling by the evaporation of ether brought about by bubbling dry air through the ether. The solution is constantly agitated during the freezing in order to hasten freezing and to make the conditions more constant.

Freezing point and supercooling determinations were made of the following individual salts and sugars and combinations of the same. The data are shown in Tables 3 and 4.

1. Individual milk salts, sucrose, and lactose
2. Sucrose
3. Lactose
4. Sucrose plus lactose
5. Milk salts plus sucrose and lactose
6. Combination of the different milk salts

TABLE 3.—DETERMINATION OF THE FREEZING POINT OF INDIVIDUAL MILK SALTS AND SUGARS AND COMBINATIONS OF SALT AND SUGARS.

Ingredients	Determinations of Freezing Point Expressed in Degrees Centigrade				
	1	2	3	4	Average
Sucrose	—1.31	—1.29	—1.30	—1.30	—1.297
Lactose	—0.51	—0.50	—0.51	—0.49	—0.503
Calcium Citrate*	+0.11	+0.10	+0.10	+0.09	+0.100
Mono-Pot. Phosphate	+0.03	+0.04	+0.04	+0.04	+0.038
Sodium Chloride	—0.015	—0.02	—0.015	—0.02	—0.018
Potassium Chloride	—0.02	—0.025	—0.015	—0.02	—0.020
Di-Potassium Phosphate	—0.01	—0.01	—0.01	—0.01	—0.010
Tri-Calcium Phosphate*	—0.01	—0.01	—0.01	—0.01	—0.010
Di-Calcium Phosphate	—0.02	—0.02	—0.02	—0.02	—0.020
Potassium Citrate	—0.08	—0.08	—0.08	—0.075	—0.079
Calcium Oxide*	—0.07	—0.07	—0.07	—0.07	—0.070
Magnesium Citrate	—0.03	—0.03	—0.03	—0.03	—0.030
Di-Magnesium Phosphate*	—0.03	—0.03	—0.03	—0.03	—0.030
Sucrose + Lactose	—2.00	—1.98	—1.97	—2.00	—1.989
Salts and Sugars*	—2.53	—2.53	—2.51	—2.52	—2.524
Milk salts, combined*	—0.34	—0.36	—0.35	—0.35	—0.350

*Not completely soluble.

The data in Table 3 show that there is considerable difference in the freezing points of the individual sugars and milk salts. The influence of the sugars, particularly, sucrose in depressing the freezing point was very marked and was especially apparent when the milk salts and sugars were combined.

As may be expected there was considerable difference between the freezing point and supercooling point of the individual milk salts and sugars and combinations of these ingredients. The lowest supercooling point was attained in the instance of the combination of the milk salts and the two sugars, with a combination of the two sugars only having the second lowest supercooling point. Of the individual ingredients sucrose gave the lowest supercooling point with sodium chloride, potassium chloride and lactose ranking in the order named.

TABLE 4.—DETERMINATIONS OF THE SUPERCOOLING POINT OF INDIVIDUAL MILK SALTS AND SUGARS AND COMBINATIONS OF MILK SALTS AND SUGARS.

Ingredients	Determinations of Freezing Point Expressed in Degrees Centigrade				
	1	2	3	4	Average
Sucrose	—3.38	—3.34	—3.58	—3.42	—3.430
Lactose	—2.38	—1.98	—1.96	—2.18	—2.225
Calcium Citrate*	—1.90	—0.75	—0.90	—1.12	—1.168
Mono-Pot. Phosphate	—1.66	—1.47	—1.93	—1.78	—1.710
Sodium Chloride	—2.79	—3.46	—2.89	—3.32	—3.115
Potassium Chloride	—2.20	—2.64	—2.36	—2.88	—2.520
Di-Potassium Phosphate	—1.46	—1.56	—1.48	—1.34	—1.460
Tri-Calc Phosphate*	—1.76	—1.89	—1.83	—1.88	—1.840
Di-Calcium Phosphate*	—1.97	—2.05	—1.95	—1.96	—1.983
Potassium Citrate	—1.48	—1.56	—1.68	—1.45	—1.543
Calcium Oxide	—1.75	—1.78	—1.66	—1.59	—1.698
Magnesium Citrate	—1.86	—1.96	—1.84	—1.82	—1.870
Di-Magnesium Phosphate*	—1.96	—1.98	—1.92	—1.86	—1.930
Sucrose + Lactose	—3.98	—4.77	—3.53	—3.84	—4.030
Salts + Sugars*	—4.91	—4.66	—4.34	—4.38	—4.573
Milk Salts*	—2.45	—2.14	—2.26	—2.20	—2.263

*Not completely soluble.

Table 4 shows that a great amount of supercooling is caused by potassium and sodium chloride in comparison with the small amount of the salt that is in the solution.

TABLE 5.—COMPARISON OF THE CALCULATED AND ACTUAL DETERMINATIONS OF THE LOWERING OF THE FREEZING POINTS OF MILK SALTS, SUGARS AND COMBINATIONS OF EACH EXPRESSED IN DEGREES CENTIGRADE.

Solution	Calculated (Leighton)	Determined	Difference
Sugars	—2.053	—1.987	—0.066
Milk Salts	—0.458	—0.350	—0.108
Sugar plus Milk Salts	—2.511	—2.525	+0.014

Table 5 shows that there is a close correlation between calculations according to Leighton's formula and the actual determination of the freezing point of the sugars, milk salts and combinations of each. The greatest difference seems to be in the instance of the milk salts. To account for this, we have to consider that in making freezing point determinations of the individual salts the factor of error enters into a great extent as the lowering of the freezing point is so slight in comparison with the depression caused by the sugars or by the combined sugars and salts.

From the data presented, it may be concluded that the depressed freezing point caused by the milk salts is very slight and perhaps insignificant in comparison with the depression caused by the presence of the sugars, sucrose and lactose, and that Leighton's method of calculation is accurate enough for all practical purposes in research in ice cream.

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