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The Relation of the Freezing Procedure
and the Composition of the Mixture
to the Physical and Crystalline
Structure of Ice Cream

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SUMMARY

1. The composition of a mixture markedly affects the crystalline and physical structure of the resultant ice cream.

2. An increase in the per cent fat, solids-not-fat, sugar and gelatine changes the texture of the resultant ice creams from a coarse condition to a fine crystalline and physical structure.

3. The petrographic microscope was used for the identification of crystalline and non-crystalline materials as they actually exist in ice cream. The crystals shown in the microphotographs of ice cream were identified as ice crystals.

4. Immersion oil is superior to air as an embedding medium to be used for the study of the crystalline and physical structure of ice creams.

5. Fine texture in ice cream is associated with the presence of a uniformly dispersed system of small angularly shaped ice crystals and jagged tapering air cell boundaries.

ACKNOWLEDGMENT

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The Relation of the Freezing Procedure and the Composition of the Mixture to the Physical and Crystalline Structure of Ice Cream

W. H. E. REID AND M. W. HALES*

INTRODUCTION

Every phase of ice cream manufacturing reflects directly upon the quality of the finished product. When a desirable ice cream is produced, the successful performance and standardization of the various phases of manufacture determine very largely the uniformity of the finished product. When an ice cream plant is not adequately equipped to perform modern freezing and hardening processes, the composition of the mixture should be modified to assist in securing the smoothness and fineness of texture that are characteristic of a satisfactory ice cream. Even with the application of the most modern methods an improvement in the texture of the resultant ice cream may be acquired by properly balancing the ice cream mixture.

It has been assumed that the texture of ice cream depends very largely upon the presence of varying sizes and arrangements of ice crystals. Likewise it has been assumed that the general effect of fineness or coarseness as judged by the combined senses of taste, touch, and sight is a result of the presence of large ice crystals as contrasted with the more uniformly dispersed small crystals. An accurate study of the texture and crystalline structure of ice cream must not be dependent only upon the reaction of the human senses.

The ordinary microscope as commonly used, usually furnishes a microscopic field that is general and vague. A method that will furnish a more distinct picture and which at the same time can be used for identification studies should be used. Microphotographs can be individually and repeatedly examined and for these reasons, this investigation is confined to an improved method of microphotographic study of the crystalline and physical structure of ice cream as affected by composition.

* The data presented in this bulletin were taken from a paper submitted by the junior author in partial fulfillment of the requirements for the degree of Master of Arts in the Graduate School of the University of Missouri, 1933.

REVIEW OF LITERATURE

Brainerd (1915) made a microscopic examination of ice creams of varying composition and concluded that smoothness depends upon the amount and fineness of division of ice crystals.

Dahlberg (1925) found that the diameter of the air cells was not affected by the composition of the ice cream and the size of air cells could not be definitely related to texture. Sugar improved the texture of ice cream by reducing the amount of ice that was formed while milk fat prevented the formation of large ice crystals by mechanically obstructing their growth.

Reid (1932) made a macrophotographic study of vanilla, chocolate and strawberry ice creams that had a history of temperature changes and varying degrees of hardness. The data show differences in the crystalline structure in coarse ice cream and a finer, more uniformly dispersed crystalline structure in ice creams and sherbets of finer texture and smooth body.

Cole (1928) developed a microscopic technique for examination of crystalline structure of ice cream. In 1932, the same author showed the effect of freezing and hardening on the physical make-up of the finished product. Light filters were used to acquire contrasts and distinction in the microtome prepared sections of ice cream.

Reid (1933) prepared macrophotographs showing the effect that variable increments of butterfat, serum solids, sugar and gelatine, methods of freezing, hardening and heat shocking had upon the texture and crystalline structure of different flavored ice creams and sherbets. The data show the crystalline structure was altered with each variation in the composition of mixtures, modification of the freezing and hardening procedure, and heat shocking. The altering of the crystalline and physical structure reflected itself unfavorably upon the flavor, body and texture of the resultant ice creams and sherbets.

PROCEDURE

The mixtures used in the conduct of this study of the crystalline and physical structure of ice creams were compounded so as to be representative of average commercial mixtures and of extremes, by using variable increments of butterfat, solids-not-fat, sugar and gelatine. The effect of one particular ingredient upon the crystalline and physical structure of the resultant ice cream was studied by varying one ingredient only and maintaining other ingredients

at a constant within the series. The standard mixture was compounded by weight to contain:

Butterfat	12.50 per cent
Solids (serum)	11.50 per cent
Sugar	14.00 per cent
Gelatine	0.30 per cent
Egg Powder	0.30 per cent
Total Solids	38.60 per cent

The standard mixture was processed as follows:

1. Pasteurized by heating to 150 degrees Fahrenheit (65.5 degrees Centigrade) with a holding period of 30 minutes.
2. Homogenized at pasteurization temperature by the application of a pressure of 2,000 pounds on the first stage and 1,000 pounds on the second stage.
3. Cooled immediately to 40 degrees Fahrenheit (4.4 degrees Centigrade).
4. Aged 24 hours at 40 degrees Fahrenheit (4.4 degrees Centigrade).

Four different series of mixtures, each containing variable increments of one particular ingredient, were calculated as follows:

Series 1. Butterfat—8, 10, 12, 15 and 18 per cent.

Series 2. Solids (serum)—9, 11, 13, and 15 per cent.

Series 3. Sugar—12, 14, 16, and 18 per cent.

Series 4. Gelatine—0.25, 0.35, 0.45, and 0.80 per cent.

The freezing of all batch mixtures was performed in a triple dasher, direct expansion freezer with a capacity of 40 quarts of ice cream per batch. The freezing period was divided into two phases, i e., time required to freeze and time required to whip each batch to 100 per cent overrun.

The overrun was obtained by the application of a standard overrun tester reading direct in both percentage and pounds of ice cream per gallon.

When each batch, within each series, had attained 100 per cent overrun, samples of the resultant ice cream were obtained and hardened immediately to a temperature of zero degrees Fahrenheit (—17.8 degrees Centigrade) in about 5 hours.

Preparation of Sections of Ice Cream for Microscopic Examination.—The sections of ice cream were prepared in a hardening room at a temperature varying from —6 to —12 degrees Fahrenheit (—21.10 to —24.40 degrees Centigrade). All instruments including the ordinary and petrographic microscopes and their attachments

were chilled for a period of five hours before using. By using a modified microtome it was possible to prepare sections of ice cream that were only one layer of crystalline material in thickness.

EXPERIMENTAL DATA

The Effect of Variable Increments of Different Ingredients Upon the Freezing Properties of Different Ice Creams.—The data, Table 1, show that as the increments of butterfat were increased from 8 to 18 per cent respectively, the time required to freeze each mixture was increased and the ability of that mixture to incorporate air was impaired. The mixture containing 8 per cent butterfat required 1 minute and 22 seconds to whip whereas the mixture containing 18 per cent butterfat required 4 minutes and 2 seconds to incorporate an equal volume of air. The total time required to freeze each batch increased in almost direct ratio to the increased increments of butterfat.

TABLE 1.—THE EFFECT OF VARIABLE INCREMENTS OF BUTTERFAT UPON THE FREEZING AND WHIPPING PROPERTIES OF ICE CREAM MIXTURES

Batch Number	Butterfat per cent	Temperature of ammonia degrees F	Freezing of Ice Cream						Temperature of ice cream degrees F	Consistency of ice cream	Overrun ice cream per cent
			Time to freeze		Time to whip		Total time				
			Min.	Sec.	Min.	Sec.	Min.	Sec.			
1	8	-20	2	0	1	22	3	22	26	Medium firm	105
2	10	-20	1	50	2	09	3	59	26	Firm	102
3	12	-20	1	40	1	37	3	17	26	Very firm	101
4	15	-20	2	07	2	26	4	33	26	Very firm	102
5	18	-20	2	23	4	02	6	25	26	Extremely firm	102

Increased increments of solids-not-fat, Table 2, had a negligible effect upon the time to freeze each mixture. However, the time required to whip was retarded with each additional increment. When 9 per cent of solids-not-fat were contained in the mixture only 1 minute was required to whip whereas the addition of 15 per cent of solids-not-fat required 3 minutes and 42 seconds or a difference of 2 minutes and 42 seconds. The increased time required to whip may have been due to the increase in the lactose content of the respective mixtures.

TABLE 2.—THE EFFECT OF VARIABLE INCREMENTS OF SOLIDS NOT FAT UPON THE FREEZING AND WHIPPING PROPERTIES OF ICE CREAM MIXTURES

Batch Number	Solids not fat content	Temperature of ammonia degrees F	Freezing of Ice Cream						Temperature of ice cream degrees F	Consistency of ice cream	Overrun ice cream per cent
			Time to freeze		Time to whip		Total time				
			Min.	Sec.	Min.	Sec.	Min.	Sec.			
1	9	-16	2	0	1	0	3	0	26	Medium firm	100
2	11	-16	1	44	1	48	3	32	26	Firm	100
3	13	-17	1	46	2	31	4	17	26	Very firm	100
4	15	-17	1	46	3	42	5	28	25	Very firm	100

The time required to freeze mixtures containing variable increments of sugar was not increased uniformly, but the time required to whip was retarded in direct ratio to the increments of sugar added to the different mixtures, Table 3.

TABLE 3.—THE EFFECT OF VARIABLE INCREMENTS OF SUGAR UPON THE FREEZING AND WHIPPING PROPERTIES OF ICE CREAM MIXTURES

Batch Number	Sugar per cent	Temperature of ammonia degrees F	Freezing of Ice Cream						Temperature of ice cream degrees F	Consistency of ice cream	Overrun ice cream per cent
			Time to freeze		Time to whip		Total time				
			Min.	Sec.	Min.	Sec.	Min.	Sec.			
1	12	-25	2	12	1	39	3	51	26	Firm	100
2	14	-25	2	3	2	30	4	33	26	Very firm	100
3	16	-25	2	17	3	11	5	28	26	Very firm	100
4	18	-25	2	0	3	35	5	35	26	Very firm	100

Table 4 shows that gelatine influences the whipping ability of the mixture similar to that of sugar, as the mixture to which was added .25 of one per cent of gelatine required 1 minute and 33 seconds to whip and the mixture containing .80 of one per cent required 3 minutes and 40 seconds to reach 100 per cent overrun. These differences in the whipping ability of the respective mixtures resulting from the additional increments of the gelatine.

It was thought that the differences in the composition of the various mixtures and the differences in the time required to freeze and to whip the respective mixtures would reflect definitely upon the crystalline and physical structure of the resultant ice cream.

TABLE 4.—THE EFFECT OF VARIABLE INCREMENTS OF GELATIN UPON THE FREEZING AND WHIPPING PROPERTIES OF ICE CREAM MIXTURES

Batch Number	Gelatin per cent	Temperature of ammonia degrees F	Freezing of Ice Cream						Temperature of ice cream degrees F	Consistency of ice cream	Overrun ice cream per cent
			Time to freeze		Time to whip		Total time				
			Min.	Sec.	Min.	Sec.	Min.	Sec.			
1	.25	-20	1	59	1	33	3	32	26	Firm	100
2	.35	-18	1	59	2	13	4	12	26	Very firm	100
3	.45	-17	2	15	3	5	5	20	26	Very firm	100
4	.80	-20	2	15	3	40	5	55	27	Soggy	100

Microscopic Examination of Sections of Different Ice Creams.

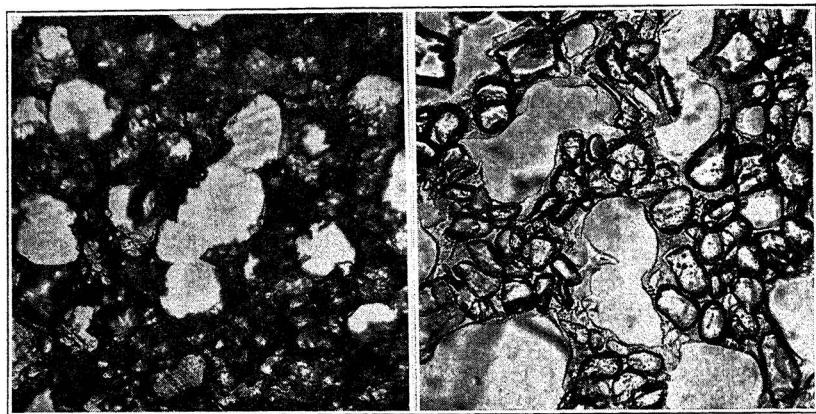
The ordinary microscope with air as an embedding medium and the petrographic microscope using oil as an immersion medium were used for the purpose of comparison. In studying the sections of ice cream with each microscope a magnification of 95X was used.

When using the ordinary microscope it was not possible to distinguish between the crystalline and non-crystalline substances and to identify these substances by optical properties. The application of the technique involving the use of the petrographic microscope made it possible to distinguish between the crystalline and non-crystalline substances and properties of the sections of the different ice creams. When dependent upon air as the embedding medium, the constituents of the ice creams are shown in bold relief and lack distinctiveness due to the difference between the index of refraction of the ice cream and the embedding medium.

It was found during the study that there was less difference in the index of refraction between the ice present in the ice cream and other materials comprising the ice cream than between the ice or other materials and the air. Because of the relatively slight disparity between the differences of the index of refraction of the constituents and ice cream and the greater differences between them and air, it was difficult to differentiate between ice and the bonding material when using air as an immersion medium. To correct this deficiency, the difference in the index of refraction between the material to be studied and the medium in which it is embedded was decreased by mounting the section in an oil medium having a higher index than air. The particular oil used was a mixture of amyl alcohol and kerosene blended to have a standard index of refraction of 1.420 at 21 degrees Centigrade. However the index of refraction of this oil is raised at lower temperatures, and it was

computed to be about 1.435 at the hardening room temperature.

When a thin section is mounted in a medium having an index of refraction approximately the same as that of one of its constituents, that particular constituent of the section shows but little relief whereas the other constituents not having such a close resemblance are more prominent because of their greater difference in index of refraction. The use of an oil as an embedding medium with an index of refraction the same as that of the matrix material in the ice cream causes the material matched in index to become invisible, and the material differing in index of refraction becomes apparent.



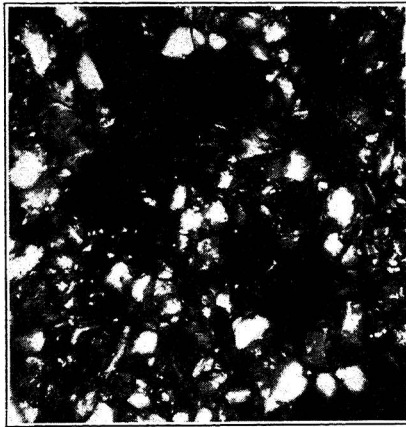
Ordinary Microscope using Air as Immersion Medium.

Petrographic Microscope using Oil as Immersion Medium.

Figure 1.—Microphotographs Showing Application of the Ordinary and Petrographic Microscopes. Air and Oil as Immersion Mediums.

The difference in the microphotographs, Figure 1, is due to the use of air as the embedding medium at the left, while the section of ice cream shown at the right was immersed in oil having an index of refraction approximating the index of one of the constituents in the ice cream. The air cavities in the ice cream as shown at the right in Figure 1 are clear and open. The smaller areas of high relief bounded by a dark border represent ice crystals and are darkly colored at their borders because of the difference in index of refraction contrasted with the surrounding material. It is thought that the less prominent portion represents the residuum

of butter fat, sugars, and solids-not-fat remaining after a considerable quantity of the water has frozen out as ice. This material appears less prominent in the microphotograph because its index of refraction more closely approximates that of the oil than any of the other ice cream constituents. In contrast, the section at the left in Figure 1 lacks definiteness in that the air cells only are shown with any degree of clarity.



Petrographic Microscope with Nicols crossed.

Figure 2.—Microphotograph Showing Crystalline and Non-crystalline Materials in Ice Cream.

Figure 2 shows a section of ice cream in polarized light with the nicols crossed. The cavities which were air filled in ordinary ice cream but are now filled with immersion oil and continue to be isotropic, are marked by patches of no light under crossed nicols. The presence of anisotropic material is shown by areas through which light has been transmitted. These light areas (crystals) are set in a background of material which usually appears isotropic or, in some instances, very slightly birefringent, due probably to very slight ice growth. When studied visually the matrix appears completely isotropic but upon close examination the photograph shows a slight haze in some of the bonding material. Its resemblance to the ice crystals tends to show that the minute volume of light coming through is due to small amounts of ice that have developed in the bonding material. The crystalline anisotropic material studied in greater detail in the coarse-grained sections showed that the

crystals had an extremely low index of refraction. The birefringence was seen to be low and the interference colors were never above light gray of the first order. The interference figure obtained on the crystals was uniaxial and the optical character was positive. It would seem that the crystalline material is no doubt ice. It was particularly characteristic of the crystals that their index of refraction should be below even the low index oil that was used. However, this is in accord with the fact that ice has a very low index of refraction.

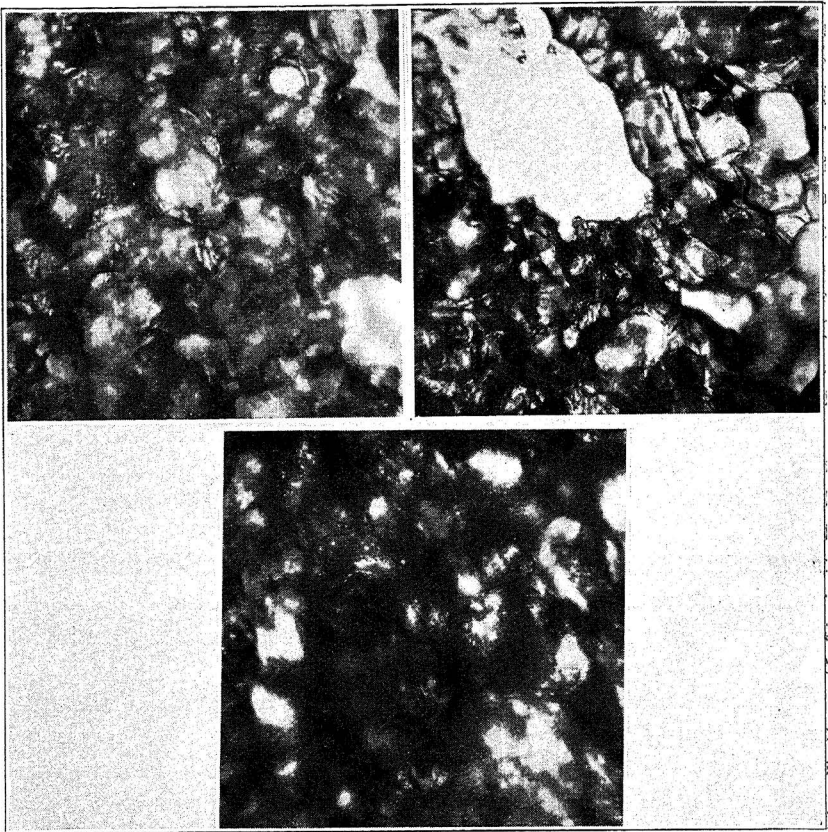
The three different materials shown in the section of ice cream are crystalline ice, isotropic bonding residuum, and air cavities. Minute slender crystal forms thought to be sugar crystals were detected in the amorphous substance or the bonding material; yet their identity was uncertain.

Microphotographs of the sections of ice creams involving the use of the ordinary and petrographic microscope are presented alternately. Figures 3, 5, 7, and 9 include the microphotographs taken with an ordinary microscope using air as an immersion medium, while Figures 4, 6, 8, 10, and 11 were taken with the petrographic microscope using oil as an immersion medium. Figures 3, 5, 7, and 9 were prepared from the same ice creams as were Figures 4, 6, 8, and 10.

The effect of the composition of the different mixtures on the crystalline and physical structure of the resultant ice cream are shown most effectively in Figures 4, 6, 8, and 10. Although the photographs taken with the application of the ordinary microscope, using air as an immersion medium, show general structural differences, the distinctiveness of the component parts of the section of ice cream is not sufficiently definite.

In Figures 3, 5, 7, and 9 there appears to be a gradual transition from a coarse texture in the ice creams containing the lower percentages of the different ingredients to a finer structure or texture with an increase in the percentage of fat, solids-not-fat, sugar or gelatine. From the comparison study, Figure 1, it is logical to assume that the ice crystals are smaller and more evenly distributed in the 18 per cent fat ice cream, Figure 3; the 15 per cent solids-not-fat ice cream, Figure 7; or the .45 per cent gelatine ice cream, Figure 9; than in the lower percentage ice creams illustrated in the same figures.

A gradual decrease in the size of the ice crystals may be seen as the increments of butterfat are increased from 8 to 18 per cent,



Eight Per Cent Butterfat.

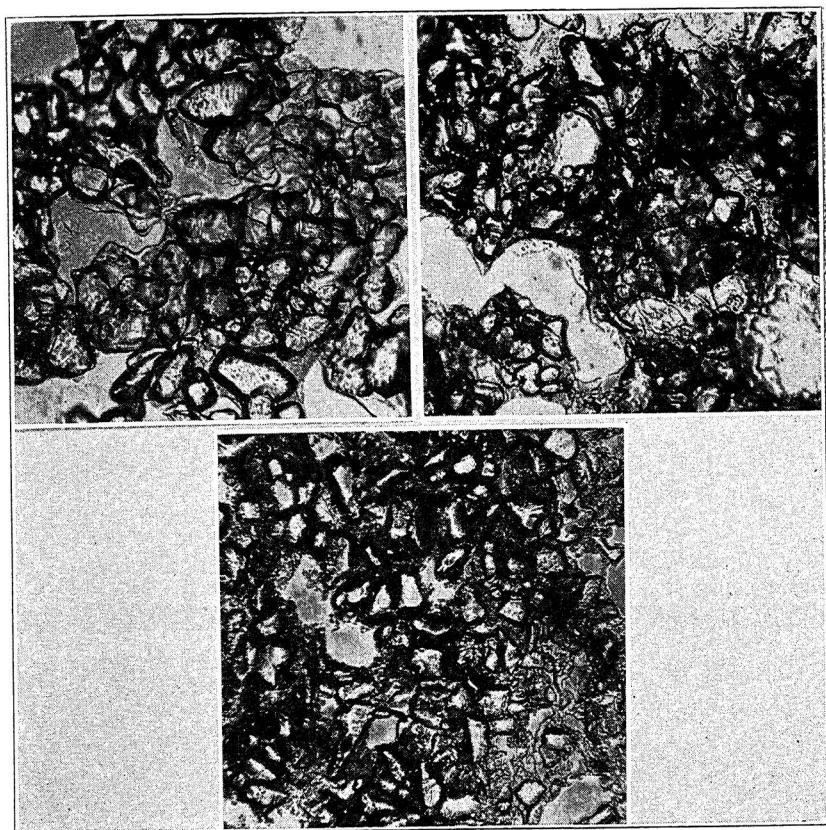
Twelve Per Cent Butterfat.

Eighteen Per Cent Butterfat.

Figure 3.—Microphotographs Showing Influence of Variable Increments of Butterfat Upon the Crystalline and Physical Structure of Ice Cream.

Figure 4. The ice crystals in the 8 per cent ice cream are large, semi-angular and very loosely arranged. The boundaries of the air cells are comparatively smooth and thick and do not taper back into the section. This particular specimen is quite typical of a coarse-textured ice cream.

The section of the ice cream containing 12 per cent fat shows a finer structure than the ice cream containing 8 per cent fat. The ice crystals are smaller in size with sharper edges, although the air cells continue to have the appearance of thickly bordered areas.



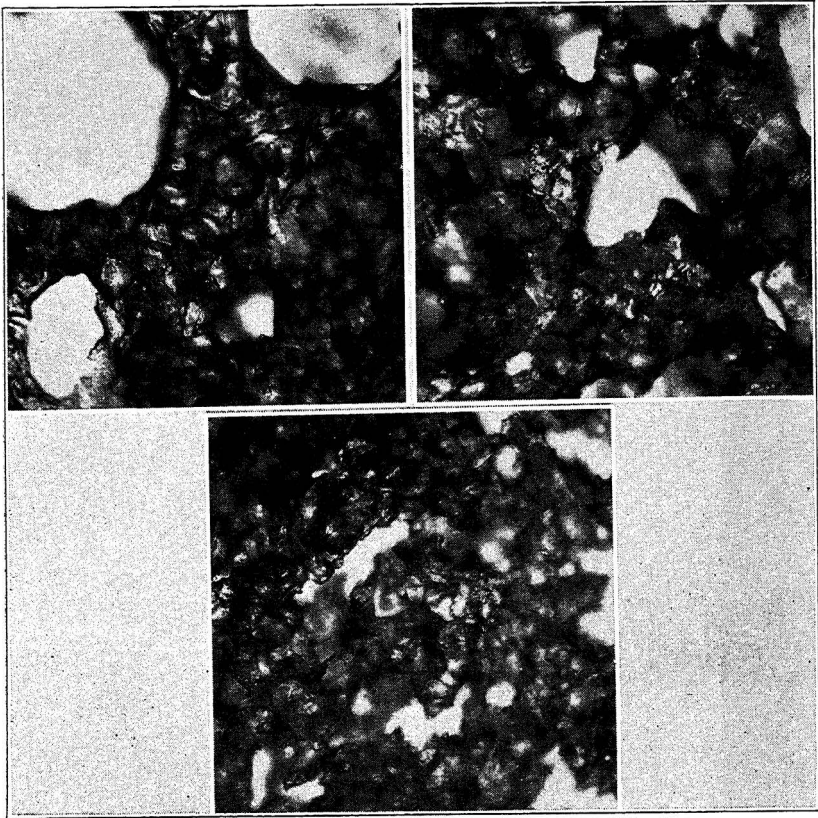
Eight Per Cent Butterfat.

Twelve Per Cent Butterfat.

Eighteen Per Cent Butterfat.

Figure 4.—Microphotographs Showing Influence of Variable Increments of Butterfat Upon the Crystalline and Physical Structure of Ice Cream.

The crystals in the ice cream containing 18 per cent fat are relatively more uniform in size, distinctly angular and appear as though a definite position had been arranged for their systematic setting in the bonding material. The total space occupied by the air cells is probably equal to that of the other sections but vary from an oval to a more elongated shape in most cases. The smooth boundaries of the air cells have changed to rugged contours in which the crystalline and non-crystalline materials are more finely dispersed. The most marked difference in the structure of the ice creams is between 12 and 18 per cent fat. However, the microphotographs seem to show, in the ice creams, an improve-



Nine Per Cent Solids-Not-Fat.

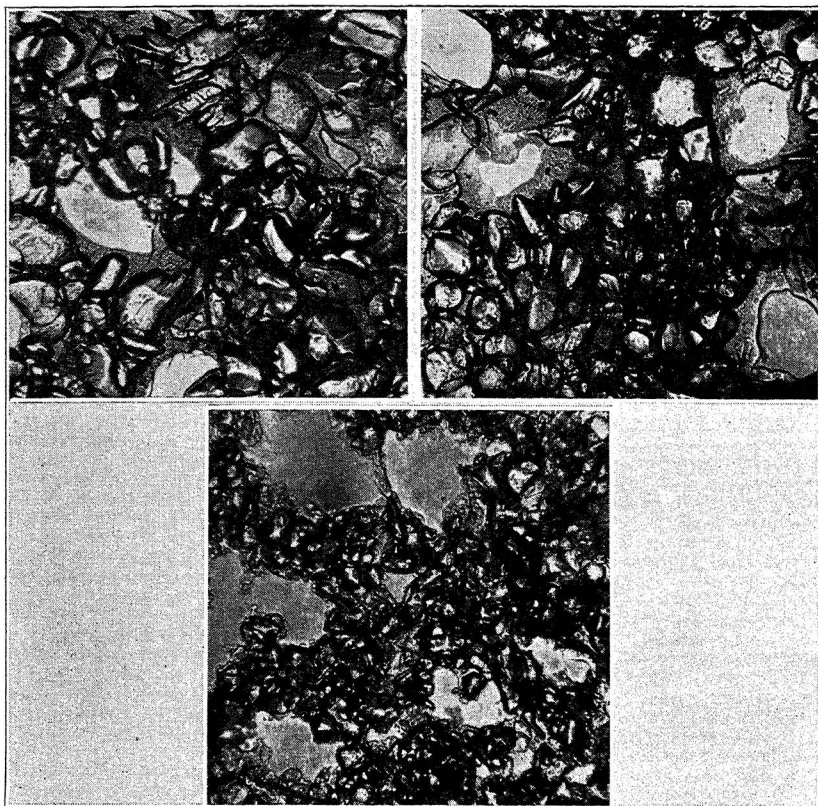
Eleven Per Cent Solids-Not-Fat.

Fifteen Per Cent Solids-Not-Fat.

Figure 5.—Microphotographs Showing Influence of Variable Increments of Solids-Not-Fat Upon the Crystalline and Physical Structure of Ice Cream.

ment in the texture that is directly proportional to the increased increments of fat.

The microphotographs of sections of ice creams varying in solids-not-fat show differences that are very similar to those in Figure 4. The crystalline and physical structure of the 9 per cent solids-not-fat ice cream, Figure 6, illustrates a condition characteristic of coarse ice cream. The ice crystals are not well distributed and areas of clustered crystals are frequent. The clear areas are not marked with small angular shaped crystals but rather by large rounded crystals haphazardly bound by the thick appearing matrix material that ends abruptly at the borders of the air cells. While



Nine Per Cent Solids-Not-Fat.

Eleven Per Cent Solids-Not-Fat.

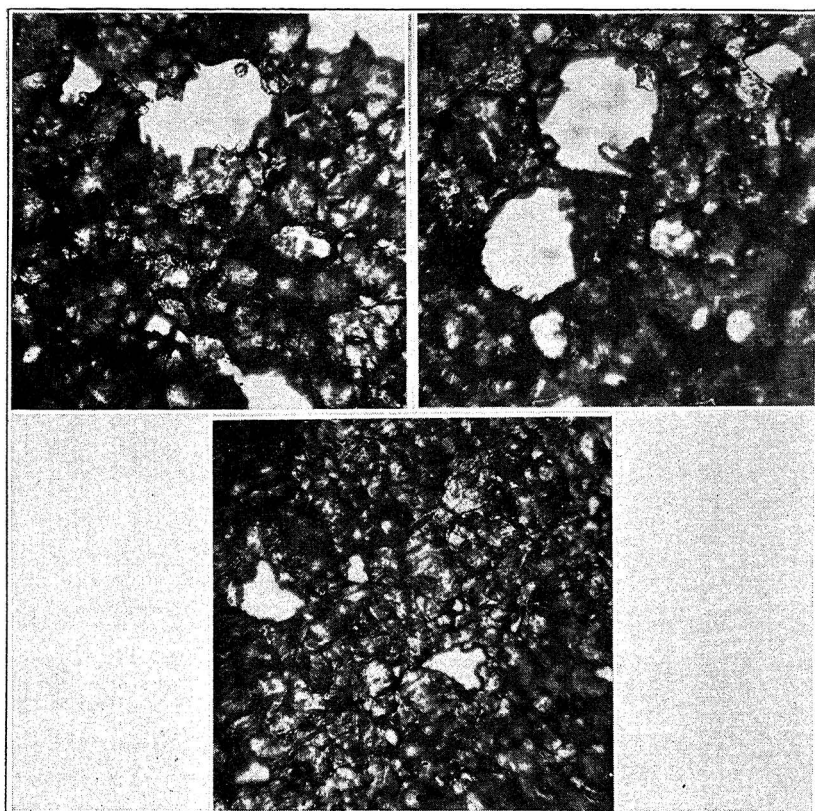
Fifteen Per Cent Solids-Not-Fat.

Figure 6.—Microphotographs Showing the Influence of Variable Increments of Solids-Not-Fat Upon the Physical and Crystalline Structure of Ice Cream.

the air cells do not have boundaries that are entirely smooth, the occurrence of tiny jagged projections is not frequent.

An ice cream containing 11 per cent solids, Figure 6, does not show as massive a structure as the photograph of the 9 per cent solids-not-fat ice cream. However, a general indication of possible coarseness is present. Small fairly angular and more uniformly dispersed ice crystals occupy the spaces that were previously filled with the larger crystals.

A marked contrast in the crystalline and physical structure is evident in the ice cream containing 15 per cent solids-not-fat. The small, angular shaped crystals seem more rigidly fastened in a thinner bonding material. The air cells boundaries are irregular and



Twelve Per Cent Sugar.

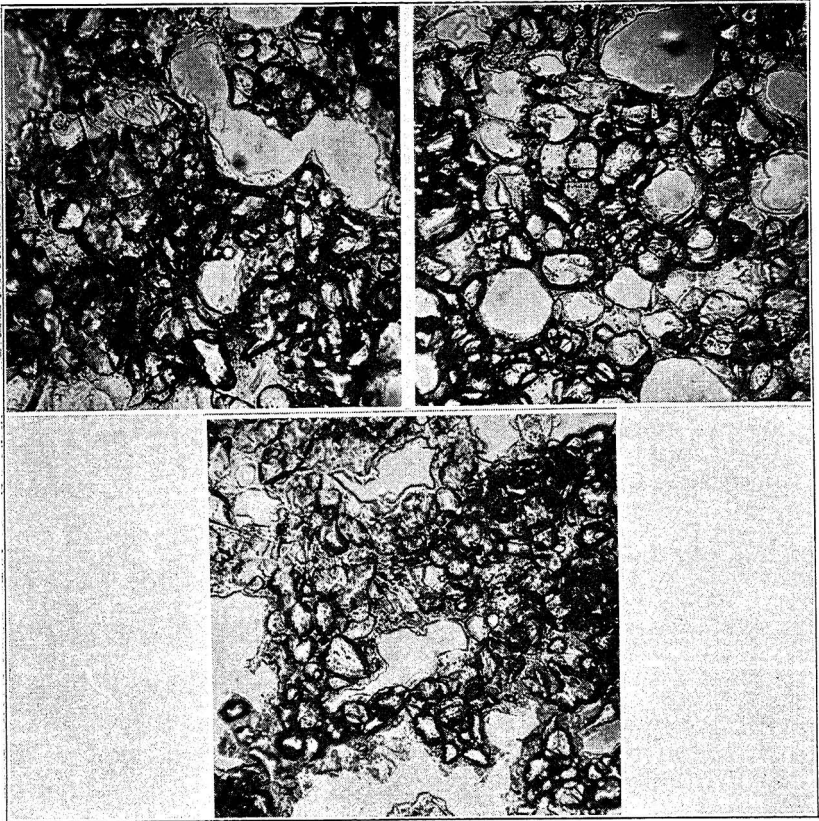
Fourteen Per Cent Sugar.

Eighteen Per Cent Sugar

Figure 7.—Microphotographs Showing the Influence of Variable Increments of Sugar Upon the Crystalline and Physical Structure of Ice Cream.

many sharp edges penetrate what otherwise would be a smooth continuous line. This photograph illustrates finer physical condition.

The sections of ice creams varying in their sugar content show a more gradual transition in their degree of fineness, Figure 8. The texture illustrated by the photograph of ice cream containing 12 per cent sugar is classed as extremely coarse, but rather as a medium close texture. Large crystals do occur but they are generally surrounded by smaller more angular crystals. In other words, the size of the crystals is not particularly large, but their distribution is not representative of fine textured ice cream. Crys-



Twelve Per Cent Sugar.

Fourteen Per Cent Sugar.

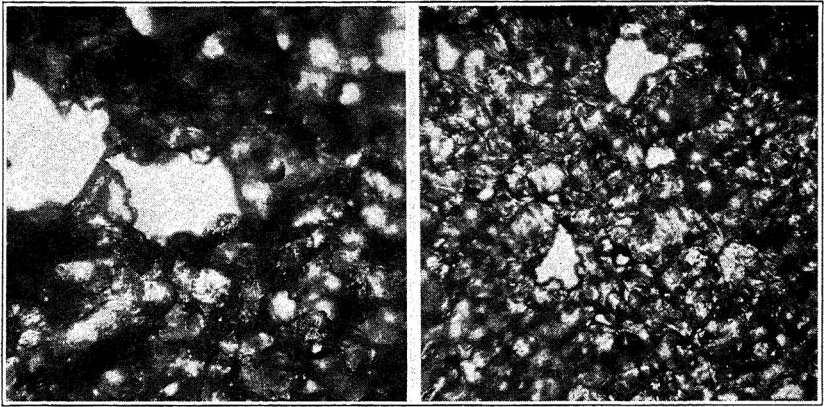
Eighteen Per Cent Sugar.

Figure 8.—Microphotographs Showing Influence of Variable Increments of Sugar Upon the Crystalline and Physical Structure of Ice Cream.

talline clusters occupy a large portion of the photograph. The air cell boundaries are not outstandingly smooth.

The average size of the ice crystals in the ice cream containing 14 per cent sugar appear to be somewhat smaller and to have been taken from the clusters during the freezing procedure, and placed in a more systematic arrangement in the bonding material.

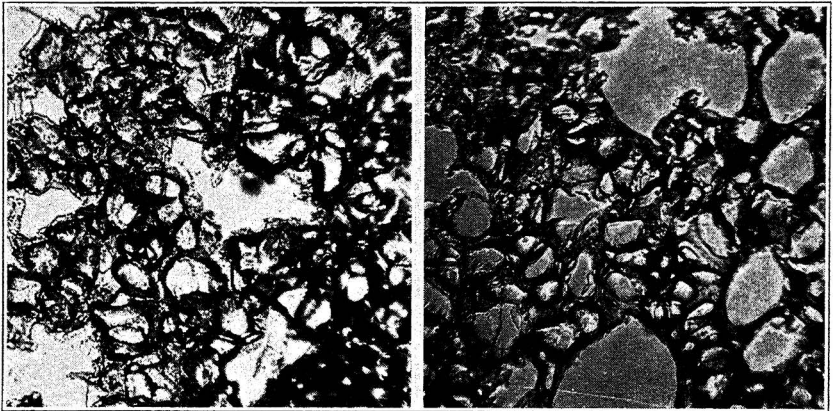
The microphotograph of the section of ice cream containing 18 per cent sugar is representative of a very fine texture. The sharp and pointed crystals are distinctly smaller than those present in the sections of ice cream containing 12 and 14 per cent sugar. The



0.25 Per Cent Gelatine.

0.45 Per Cent Gelatine.

Figure 9.—Microphotographs Showing Influence of Variable Increments of Gelatine Upon the Crystalline and Physical Structure of Ice Cream.



0.25 Per Cent Gelatine.

0.45 Per Cent Gelatine.

Figure 10.—Microphotographs Showing Influence of Variable Increments of Gelatine Upon the Crystalline and Physical Structure of Ice Cream.

air cell boundaries are tapering and are often very rough indicating a finer physical condition of the ice cream.

Figure 9 shows that gelatine does affect the crystalline and physical structure of ice cream. The ice crystals in the ice cream containing .25 per cent gelatine are somewhat rounded and medium in size. They are not very uniformly distributed but are not as loose and massive in their arrangement as the ice creams shown in Figures 4 and 6.

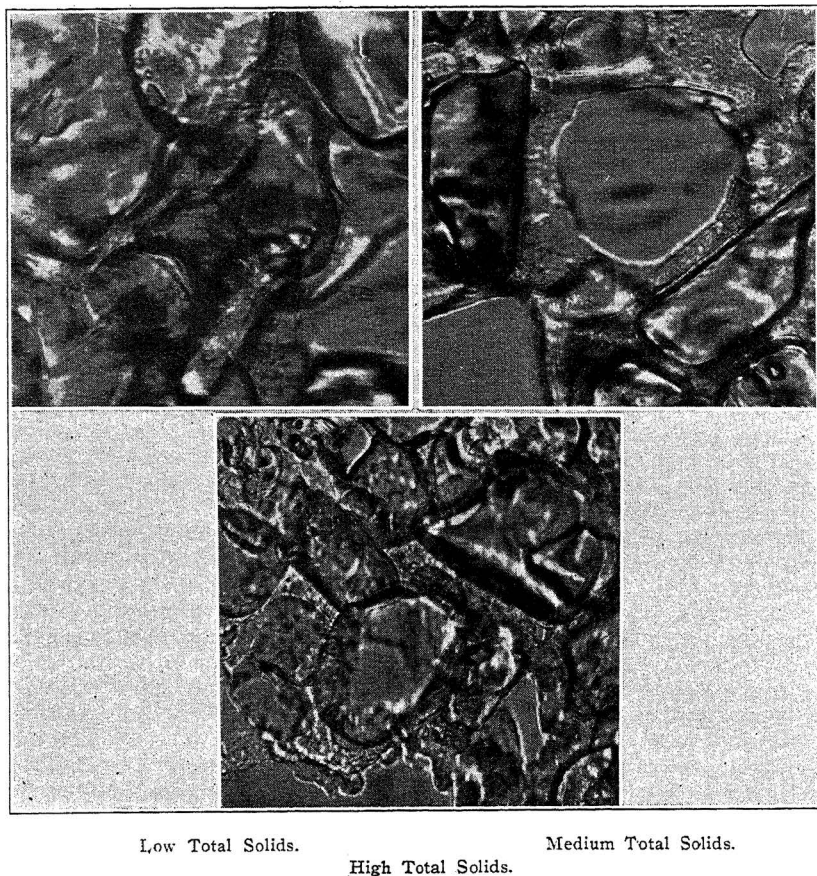


Figure 11.—Microphotographs Showing Influence of Variable Total Solids upon the Crystalline and Physical Structure of Ice Cream.

While the gradation of structure is not great between the two photographs of ice cream with different gelatine contents, they do show that an improvement in the physical structure and the texture of the ice creams has occurred. The principal difference is the reduced size and the more uniform distribution of the ice crystals in the .45 per cent gelatine ice cream as compared with the ice cream containing .25 per cent gelatine.

Microphotographs, Figures 1 to 10 inclusive, illustrate sections of ice creams that vary in coarseness and fineness because of the difference in the composition of their respective mixtures. To show more minutely the fineness of texture in ice creams, micro-

photographs of sections of ice creams varying in their total solids were prepared and magnified 400 times, Figure 11. The ice cream containing low total solids shows large rounded and overlapping crystals that cover a large section of the photograph. A small portion of an air cell that has a smooth and very abrupt border, may be seen projecting into the photograph. Any degree of systematic arrangement of the crystalline or physical structure is lacking.

The photograph of the ice cream having a medium total solids content shows distinctly more angular ice crystals which are more uniformly distributed in the bonding material. The air cell borders are still relatively smooth but appear to taper more than in the low total solids ice cream. The degree of fineness in this sample is much improved although it is not characteristic of a very smooth ice cream.

A very close and fine structure is shown in the photographed section of a high total solids ice cream. The ice crystals are markedly reduced in size and are clearly angular shaped. A section of an air cell is shown that has a rugged, tapering boundary while the several ice crystals that are visible are well spaced and do not extend into one another. This microphotograph shows a distinctly finer textured ice cream than either of the sections illustrating the effect of the low and medium solids.

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