The Use of Solid Triglyceride Stearines as Fluid Shortening Ingredients

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FLUID SHORTENINGS could offer the housewife advantages compared with plastic shortenings in ease of measuring and blending with other ingredients. Simple liquid oils however do not have the aerating action in the mixing of the conventional type of cake batters which is a primary function of the plastic shortenings. The successful use of salad oils in cakes has been confined to the chiffon type wherein beaten egg whites act as the aerating agent.

The addition of emulsifiers to the liquid oils to produce fluid shortenings with improved culinary properties is covered in patents to Cross *et al.* (1) and Trempel (2). Permanently pumpable dispersions of *beta*-phase stearines in liquid oils, which however have no aerating action in cake batter mixing, have been patented by Holman and Quimby (3), and Mitchell (4, 5).

This paper describes the study of various stearine suspensions in salad oil with respect to cake-batter aerating effect, fluidity and permanence of fluidity, and settling tendency. The term stearine herein applies to predominantly saturated acid triglycerides, which are substantially insoluble in liquid oil.

The mechanism of cake-batter aeration is discussed by Carlin (6) and Bailey (7). Briefly, air is trapped by the batter while it is being mixed and becomes dispersed in the form of fine bubbles in the fat droplets throughout the batter. The baking process produces steam from the water and carbon dioxide gas from the baking powder action. These gases penetrate and greatly expand the previously produced, fine air-bubbles in the oil droplets. Very few new bubbles are formed by the baking powder or steam, and if the fine bubbles are not present in the fat of the batter when it goes to the oven, the cake will be coarse-textured and of poor quality. Liquid oils, such as salad oils, do not accomplish the required aeration during the mixing of batters and are therefore unsatisfactory for cake making.

When a plastic shortening is overheated or melted and allowed to resolidify slowly at room temperature, it may lose much of its batter-aerating ability. This is not caused by the loss of occluded gas in a texturated shortening but rather to a change in crystal structure. The normal texturation of vegetable-oil shortening produces solids of 1 to 5 micron particle size in the *beta*-prime phase and with considerable interlocking of crystal lattices. Slow solidification results in coarse crystals, usually in the *beta*-phase polymorphic form and less interlocked. It was reasoned therefore that a satisfactory fluid shortening would require solid ingredients in the form of fine particles having aerating action in cake-batter mixing but at the same time having little tendency to form interlocked crystals.

The role of emulsifiers in cake baking is well known. They improve texture by causing the air bubbles in the fat component of the batter to be smaller and more numerous. It is to be expected that some form of emulsifier is desirable in a fluid shortening.

Experimental

Most of the experimental shortening formulations prepared in this study contained cottonseed salad oil as the liquid phase so as to obtain pourability at lower temperatures. The stearines tested as solid-phase components were prepared on a laboratory scale by conventional hydrogenation procedures from common oils and fats or solvent-separated fractions of these fats. The dispersions were prepared in most cases by melting the stearines in the salad oil at 175°F. and allowing the blend to cool over-night in an 85°F. room. Deviations from this procedure will be noted when used. The stearine dispersions were held at least 24 hrs. at room temperature prior to baking. Yellow layer cakes of conventional formula, prepared by a creaming method, were used as the test for batter-aeration efficiency. The creaming method, which mixes the sugar and shortening prior to the addition of other ingredients, was found to be most sensitive to the batter-aeration property of the dispersions. With this method the air bubbles in the batter are relatively large, and the batter volume, which is the reciprocal of the specific gravity, is high and correlates fairly well with the volume of the resulting cake.

A detailed procedure for the creaming cake follows:

YELLOW LAYER CAKE
Creaming Method

Formula	*Grams*
Flour—Softasilk	200
Sugar—Fruit Fine	200
Baking powder—Calumet	10
Salt	3
Shortening	100
Whole milk	180
Eggs (fresh)	100
Vanilla	4

Mixing Method (Sunbeam Mixmaster). Cream, sugar, salt, and shortening 2 min. on No. 4 setting. Scrape bowl and add eggs. Mix 2 min. on No. 4 setting. Scrape bowl. Add ½ milk, then sifted flour and baking powder, and mix smooth. Add balance of milk and flour, mix 1½ min.

Take batter volume. Scale 380 g. into each of two 8-in. greased, layer-cake pans. Bake approximately 23 min. at 375°F. Take cake volume and score the day after cakes are baked.

Fluidity and settling tendencies of the dispersions were noted periodically at storage temperatures over the range from 50 to 100°F. Observations were qualitative in nature.

Table I shows the cake-test results obtained with 5% dispersions of several common stearines. As seen from the batter volumes, palm oil stearine was the only one which produced an appreciable aerating effect. The addition of the stearines, except for soybean, resulted in cake volumes slightly better than obtained with liquid oil but considerably inferior to that obtained with plastic shortening. The effect of adding distilled monoglycerides of lard stearine fatty acids, Myverol 18:00, to some of the same stearine dispersions is also shown in Table I. This addition

TABLE I

Cake-Making Action of Various Stearines as 5% Dispersions in Cottonseed Salad-Oil

Stearine	Iodine value	Yellow layer cake (creaming method)					
		Without added emulsifier			With 1.2% Myversol 18:00[a] emulsifier added		
		Batter vol. (cc./g.)	Cake vol. (cc.)	Texture	Batter vol. (cc./g.)	Cake vol. (cc.)	Texture
None	—	0.98	1000	Hard	0.90	1000	Hard
Soybean oil	1	0.96	1000	Med. hard	0.96	1060	Hard
Cottonseed oil	1	0.94	1080	Med. hard	1.03	1155	Med. hard
Palm oil	2	1.05	1080	Med. hard	0.93	1155	Med. hard
Herring oil	2	0.89	1055	Med. hard	—	—	—
Lard	7	0.94	1100	Med. hard	0.85	1000	Med. hard
Plastic shortening	—	1.23	1210	Soft	1.23	1210	Soft

[a] Distilled monoglyceride of 93% assay.

slightly improved the batter volume with cottonseed oil stearine but actually reduced the amount of aeration with palm and lard stearines. The cakes made with the cottonseed and palm stearine formulas were slightly larger than before, but coarse and hard.

Fluidity observations on these "inactive" stearines (Table II) reveal that soybean oil and lard stearines which are strong *beta*-phase formers remained fluid at the storage temperatures. Addition of Myverol 18:00 to these stearine dispersions (Table II) impaired their fluidity somewhat, except in the case of the lard, where it will be remembered from Table I that it destroyed some of the batter-aerating property.

TABLE II

Fluidity of Various Stearines as 5% Dispersions in Cottonseed Salad-Oil

Stearine	Fluidity observations after one week					
	Without added emulsifier			With 1.2% Myverol 18:00[a] added		
	at 55°F.	at 70°F.	at 95°F.	at 55°F.	at 70°F.	at 95°F.
None	Good	Good	Good	—	—	—
Soybean oil	Good	Good	Good	Fair	Good	Good
Cottonseed oil	Good	Good	Fair	Poor	Poor	Poor
Palm oil	Good	Good	Solid	Solid	Poor	Solid
Herring oil	Good	Fair	Fair	Poor	Poor	Poor
Lard	Good	Good	Good	Good	Good	Good

[a] Distilled monoglyceride of 93% assay.

The precipitated solids in a number of stearine dispersions were filtered out, X-ray diffraction measurements were made, and the results were interpreted according to the terminology of Lutton (8, 14). From the crystal-phase-form results and particle-size observations shown in Table III, it can be concluded that the aerating activity of the stearines is not directly related to the phase of the solids or the crystal size. Some of the dispersions which had a coarse structure were put through a homogenizer to obtain a fine dispersion. Cake-batter aerating action was not improved irrespective of whether they were in the *beta* or *beta*-prime form.

TABLE III

Crystal-Phase Form of Various Stearines When Allowed to Precipitate from Cottonseed Salad-Oil at 85°F. Ambient Temperature

Stearine	Iodine value	Crystal phase of solids[a]	Crystal size (as precipitated)
Soybean	1	β + Trace β'	Coarse
Cottonseed	1	β'	Coarse
Palm	1	β'	Coarse
Sunflower	3	β	Med. fine
Peanut	7	$\beta + \beta'$	Coarse
Whale	9	β	Fine
Herring	2	β' + Trace β	Fine
Lard	7	β	Med. fine

[a] 5% stearine in salad-oil allowed to cool from 175°F. over-night in 85°F. Precipitated solids filtered out and examined by X-ray diffraction measurements.

In the search for "active" stearines from natural sources, rapeseed oil and mustardseed oil were tested. These oils contain 40–50% erucic acid, which on hydrogenation yields behenic acid containing triglycerides. Data on Table IV show the cake-making properties of these stearines as 5% dispersions in salad oil with and without emulsifier. It can be readily seen that rapeseed- and mustardseed-oil stearine dispersions produce fair cakes even without emulsifier; better cakes are obtained with the lower iodine value variations. The addition of 1.2% Myverol 18:00 improves the volumes and gives a more desirable cake. RSO stearine showed a faster decrease in aerating action with increased iodine value than MSO stearine.

TABLE IV

Cake-Making Action of Rapeseed- and Mustardseed-Oil Stearines as Preecipitated 5% Dispersions in Cottonseed Salad-Oil

Stearine		Yellow layer cake (creaming method)			
Type	I.V.	Myverol[a] 18:00(%)	Batter vol. (cc./g.)	Cake vol. (cc.)	Texture
Rapeseed	1	—	1.01	1045	Med. soft
Mustardseed	1	—	1.15	1120	Soft
Mustardseed	30	—	1.17	1150	Soft
Rapeseed	1	1.2	1.31	1165	Soft
Rapeseed	27	1.2	1.13	1140	Med. hard
Mustard seed	1	1.2	1.38	1210	Soft
Mustard seed	30	1.2	1.19	1210	Soft
Plastic shortening	—	—	1.28	1210	Soft

[a] Distilled monoglyceride of 93% assay.

Various levels of 1-IV RSO stearine were tested for cake performance, aerating action, and retention of fluidity with the results shown in Table V. The data demonstrate that a 5% level offers the best combination of properties.

TABLE V

Effect of Rapeseed-Oil Stearine Concentration in Fluid Shortenings[a] on Cake-Batter Aeration and Fluidity

Yellow layer cake (creaming method)				
RSO stearine (1-IV)%	Batter vol. (cc./g.)	Cake (cc.)	Texture	Fluidity after 1 wk. at 50–90°F.
1	0.90	1000	Hard	Good
2	0.98	1005	Hard	Good
3	1.07	1095	Hard	Good
4	1.12	1115	Soft	Good
5	1.31	1165	Soft	Good
6	1.35	1195	Soft	Fair at 50°F.
7	1.32	1130	Soft	Fair at 50°F.
8	1.33	1170	Soft	Fair at 50°F.

[a] Dispersions in cottonseed salad-oil with 1.2% Myverol 18:00 added.

Fluidity and settling tendency observations on rapeseed- and mustardseed-oil stearine dispersions, Table VI, demonstrate that 1-IV RSO and 30-IV MSO yield the most stable dispersions at the 60–

TABLE VI

Relative Fluidity and Settling Tendencies of Rapeseed- and Mustardseed-Oil Stearines as 5% Dispersions in Cottonseed Salad-Oil

Stearine type	I.V.	Myverol 18:00 %	Fluidity 60–100°F.	Settling tendency
RSO	1	0	Good	Very slight
MSO	1	0	Fair	Solidifies
MSO	30	0	Good	Slight
RSO	1	1.2	Good	Slight
RSO	27	1.2	Good	Slight
MSO	1	1.2	Fair	Solidifies
MSO	30	1.2	Good	Slight

100°F. storage temperatures. The 1-IV MSO stearine dispersions soon gelled on standing, even at room temperature, but the higher iodine value MSO stearine retained its fluidity much longer.

Prolonged storage and cycling temperatures caused both the rapeseed and mustardseed stearine dispersions to lose fluidity. This loss of fluidity was considerably minimized by using blends of 1-IV rapeseed- and 30-IV mustardseed-oil stearine as the dispersion components.

Examination of precipitated solids from 5% blends of rapeseed- and mustardseed-oil stearines in salad oil gave the results in Table VII. As can be seen, a difference in crystal phase was produced by the difference in cooling rates. Baking tests made on dispersions prepared by cooling 1-kg. samples of material at ambient temperatures of 70° and 85°F. showed equal aerating action for a given stearine irrespective of this difference in cooling rate. Therefore it can be concluded that there is no direct correlation between crystal-phase form and the cake-batter aerating action.

TABLE VII

Crystal-Phase Form of Rapeseed- and Mustardseed-Oil Stearines as Precipitated from 5% Solution in Cottonseed Salad-Oil by Slow Cooling

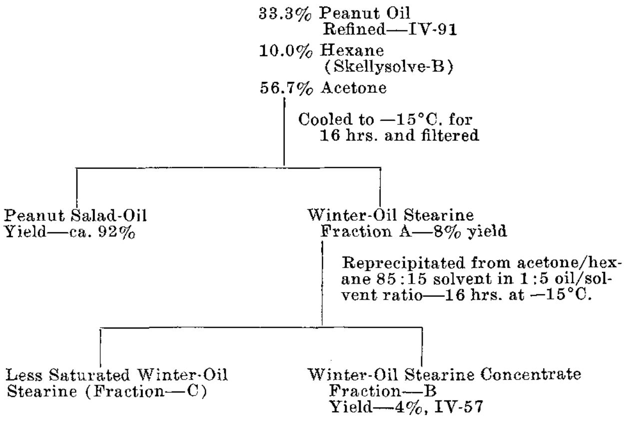
Stearine		Crystal phase of solids[a]		
Type	Iodine value	Cooled at 70°F.	Cooled at 85°F.	Crystal size
RSO	1	β'	β	Fine
RSO	27	β'	β	Fine
MSO	1	β'	β	Fine
MSO	15	β'	—	Fine
MSO	30	β'	—	Fine

[a] Solids filtered from oil and examined by X-ray diffraction measurements.

It has been reported in the literature that peanut oil contains somewhat more than 1% of disaturated triglycerides, in which one fatty acid per mol is C_{20} or longer chain length. From the experience with the rapeseed- and mustardseed-oil stearines, it was speculated that an active fraction could be separated from peanut oil by winterization. The diagram on Table VIII shows the modification of the solvent-winterization process of Boucher and Skau [9] used to obtain a 4% yield of a winter-oil stearine Fraction B having an iodine value of 57. This fraction, rich in long-chain acids, was hydrogenated to stearines of various iodine values for testing. Data in Table IX show the excellent batter-aerating action of the 2 iodine value Fraction B even without added emulsifier. The 20-IV stearine did not have good activity with or without emulsifier.

TABLE VIII

Solvent Winterization of Peanut Oil



Fluidity and settling tendencies of dispersions containing Fraction B stearine are shown on Table X. It can be noted from the data that a 5% dispersion of peanut-oil, winter-oil stearine, hydrogenated to 2-IV, in a salad oil substrate gives a dispersion which is stable in fluidity over the 50–100°F. temperature range and resists settling.

The solids from the dispersions were separated, and X-ray diffraction measurements were made. From data in Table XI it is evident that these stearines can be made to precipitate in either the *beta*-prime or the *beta*-phase by a slight alteration of the cooling rate. The long spacing data indicate a double-chain-length structure orientation of the triglycerides in the crystals.

TABLE IX

Cake-Making Action of Hydrogenated Fraction B from Peanut Oil as a Precipitated Dispersion in Cottonseed Oil

PNO-WO-St. Fraction B stearine I.V.	Emulsifier Myverol 18:00 %	Yellow layer cake (creaming method)		
		Batter vol.-cc./g.	Cake c.c.	Texture
2	0	1.21	1200	Soft
20	0	0.90	1040	Hard
2	1.2	1.24	1265	Soft
20	1.2	0.99	1000	Hard
Plastic shortening		1.28	1210	Soft

Active stearines precipitate from solution in the *beta*-prime phase when chilled more quickly and in the *beta*-phase when cooled more slowly. Both these cooling rates produced "active" dispersed solids as

TABLE X

Fluidity and Settling Tendencies of Hydrogenated Fraction B from Peanut Oil as 5% Dispersions in Oil

PNO-WO-St. Fraction B I.V.	Oil media	Emulsifier Myverol 18:00	Fluidity[a]		Settling	
			50–70°F.	70–100°F.	50–70°F.	70–100°F.
2	CSO	0	Fair	Good	None	Trace
20	CSO	0	Fair	Good	None	Some
2	CSO	1.2	Fair	Good	None	None
2	CS–Salad-oil	1.2	Good	Good	None	None

[a] Fluidity Ratings: Good—under 4000 cps
Fair —4000–6000 cps
Poor —over 6000 cps
Brookfield No. 3 spindle—12 r.p.m.

shown by baking tests. Both cooling rates produced a fine particle size; the greater portion of the particles was under 1 micron.

In observing the stearine suspensions which remained fluid at storage temperatures in the 50–100°F. range over a period of time, it was found that the stearine settled somewhat, especially at the higher temperatures. The addition of powdered *beta*-phase stearine as an anti-settling agent was found beneficial. Soybean stearines of 1 to 30 IV were prepared (3) in the *beta*-phase by thermal treatment and powdered in a hammer mill with dry ice. When the powdered, *beta*-phase soya stearines were added at a 5 to 8% concentration to dispersions of 5% "active" stearine in salad oil, settling was materially retarded without adverse effect on fluidity.

To investigate which triglycerides in the "active" stearines were most desirable with respect to batter aeration and fluidity, rapeseed-oil and winter-oil stearine from peanut oil were solvent-fractionated. The procedure of Hilditch and Meara (10, 11, 12), which is diagrammatically shown on Table XII, was applied to rapeseed oil. According to these workers, their original rapeseed oil contained about 28% of mono C22-acid triglycerides and 55% di C22-acid triglycerides; the former were concentrated in Fraction A and the latter in Fraction C. After hydrogenaton of the fractions to 1 IV, their contents of mono and di beheno glycerides were roughly estimated by using the saponification values of the fractions and of the 1 IV rapeseed oil. An assumption was made that our original oil was of approximately the same composition as the one examined by Hilditch.

TABLE XI

Crystallization Habits of Hydrogenated Peanut-Oil, Winter-Oil Stearine (Fraction B) Solids[a]

X-Ray Diffraction Measurements

Fraction B stearine I.V.	Cooled at 70°F.		Cooled at 85°F.		Crystal size
	Phase	Long spacings Å	Phase	Long spacings Å	
34	β'	48.6	$\beta' + \text{tr}\ \beta$	48.6	Fine
20	β'	49.1	β	48.2	Fine
2	β'	50.5	β	50.5	Fine

[a] Solids separated by filtration from a 5% dispersion in cottonseed salad oil prepared by melting at 170°F. and cooling as noted above.

Table XIII gives cake performance and fluidity-evaluation results obtained with 5% stearines in salad oil. The stearines were prepared from the original oil and from Fractions A and C. The product of Fraction C containing 60% monobeheno distearin produced the better cake and retained its fluidity to a greater extent. It was concluded that the monobeheno triglyceride is more desirable than the dibeheno triglyceride.

TABLE XII

Solvent Fractionation of Rapeseed Oil

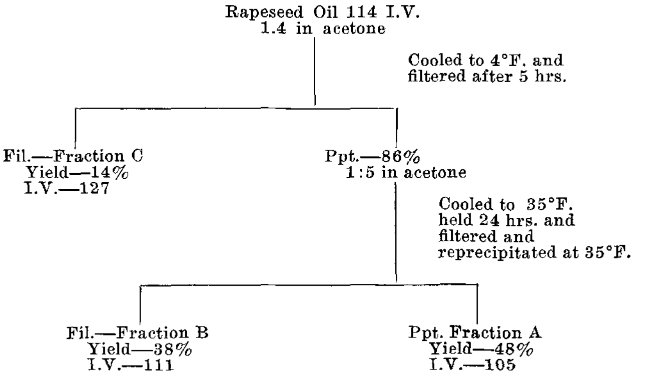


TABLE XIII

Approximate Composition and Performance of Stearines Derived from Rapeseed Oil and Its Solvent-Separated Fractions

	Original I.V.	Saponification value after complete hydrogenation	Monobehenate (%)	Dibehenate (%)
Rapeseed oil	113	176.5	28	55
Fraction A	105	174.3	20	80
Fraction C	127	179.0	60	40

	Yellow Layer Cake[a]			Dispersion fluidity
	Batter volume (cc./gm.)	Volume (cc.)	Texture	
Rapeseed oil	1.27	1145	Soft	Good
Fraction A	1.17	1110	Soft	Solid after 5 days at 105°F.
Fraction C	1.26	1160	Soft	Good

[a] Stearine—5% in salad oil, 1.2% Myverol, 18:00 added.

The fatty-acid composition of the solvent-separated peanut oil fraction was calculated from methyl-ester distillation data. The composition of this fraction is compared with peanut oil in Table XIV. A threefold concentration of the long-chain acids was effected by the solvent-winterization process.

TABLE XIV

Fatty-Acid Composition of Fraction B from Peanut Oil Enrichment of Long-Chain Acid Content by the Solvent Winterization

Fatty acid	Peanut oil[a]	PNO-WO stearine[b] Fraction B I.V. 57
	(%)	(%)
C_{14}	0.5	1.0
C_{16}	7.8	18.5
C_{18}—Stearic	3.1	4.4
Oleic	56.0	40.8
Linoleic	26.0	12.6
C_{20}	2.4	9.0
C_{22}	3.1	13.7
C_{24}	1.1	

[a] Date from Hilditch.
[b] By distillation of methyl esters and calculation from I.V.–T.V.

Using the method of Handschumacher, Thompson, and McIntyre (13) for the concentration of disaturated triglycerides, the triglycerides of the peanut-

TABLE XV

Concentration of Disaturates and Removal of Triunsaturates by Acetone Crystallization of Peanut-Oil, Winter-Oil Stearine (Fraction B)

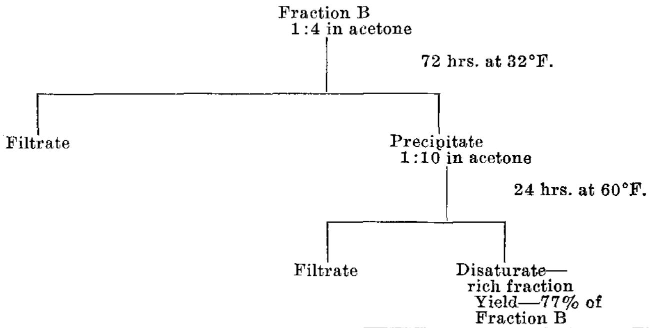


TABLE XVI

Estimate of Triacid Triglyceride Content of Fraction B from Peanut Oil[a]

	PNO-WO-St. (Fraction B)	Disaturated enriched Fraction B yield 77%
Analytical constants		
Iodine value	50.1	38.9
Thiocyanogen value	42.9	31.9
Calculated fatty acids as triglycerides		
Olein—%	41	28
Linolein—%	9	8
Saturated—%	50	64
Calculated triglycerides		
Monosaturated	—	8
Disaturated	71	92
Triacid	67	—

[a] A second solvent-winterization of peanut oil to obtain a Fraction B in 4.5% yield.

oil, winter-oil stearine were examined. A diagram of the process is given on Table XV. After the removal of the triunsaturated triglycerides, it was possible by using simultaneous equations to calculate the content of disaturated triglycerides present in the enriched fraction (Table XVI). The value of 71% disaturated triglycerides was calculated to be present in Fraction B from the data obtained on the enriched fraction and then the figure was corrected for the saturated C_{18} component to give an estimate of 67% triacid triglycerides. These triacid triglycerides result from the disaturated triglycerides already present in the Fraction B from the hydrogenation of the C_{18} unsaturated acids present in the disaturated triglycerides. It can therefore be concluded that the active principle of peanut-oil, winter-oil stearine is probably triacid triglyceride or solid solutions in which they are the major components.

Discussion

The propensity of some stearine triglycerides at *ca.* 5% concentration in cottonseed salad oil to precipitate as fine particles which can promote aeration in the mixing of cake batters is related in a general way with the presence of long-chain fatty acids and a suitable diversity of fatty acids in the constituent triglyceride. Composition *versus* performance data strongly suggest, for example, that tristearin is inactive regardless of particle size, phase, or thermal treatment whereas the diacid triglyceride, behenodistearin, is an effective, batter-aerating agent. The general type of triacid triglycerides represented by SPBe also appears to form active dispersions. The batter-aeration property does not correlate directly with the crystal phase or molecular orientation as measured by X-ray diffraction techniques. For example, both *beta*-prime phase and *beta*-phase, rapeseed stearine solids are active when precipitated by proper thermal treatment, but soybean stearine is inactive in either phase regardless of thermal treatment.

The above conclusions were not drawn from tests made with pure triglycerides but rather from test materials rich in the triglyceride in question. As indicated by the long spacings of X-ray diffraction measurements, solid solutions were involved in most cases.

In contrast to the tendency of plastic shortenings to lose their texture and become fluid on high-temperature exposure (95 to 110°F.) most experimental fluid-shortenings had a tendency to solidify at such temperatures. This solidification tendency was, in some cases, related to the presence of unstable phases, but this was not true for mustardseed stearine 1 IV, for which the 5% dispersion eventually lost fluidity even though it was completely *beta*-phase. The solidification tendency seems more likely to be related to the leaching out of ingredients initially present in solid solution and their redeposition as long, interlocked, crystal lattices (11).

All formulas containing dispersed solids tended to settle out and form a clear, oil layer. A specific gravity difference of about 0.1 exists between the solids and the liquid oil phase at room temperature. At higher temperatures the specific-gravity difference is increased while the viscosity of the liquid oil phase is materially reduced. Both the foregoing changes promote a much faster rate of settling at higher storage temperatures. The rate of settling however was slower than calculated by Stoke's law, assuming spherical-shaped particles. The decreasing of the particle size of the solids and the adding of finely divided stearines, which were inactive in cake-making and without tendency to solidify, were the best ways found to inhibit settling. Thus the addition of powdered, *beta*-phase, soya stearine of 1- to 10-microns particle size was quite effective in inhibiting settling.

Summary

One deficiency of liquid oils as all-purpose shortenings is their failure to cause the aeration in the mixing of conventional cake batters which is a normal function of the shortening ingredient. The addition of substantially insoluble, solid triglycerides in very finely divided form to liquid oils, with the object of improving their cake performance but not destroying fluidity, has been studied. Stearines made from rapeseed and mustardseed oils and certain peanut-oil fractions possessed the desired properties. The effective triglycerides in these stearines were of the monobehenodistearin and stearopalmitobehenin type.

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