Statistical Development of Improved Emulsification Systems for Bakers Shortenings

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ABSTRACT

Statistical design methods were used to observe trends of surfactant functionality for specific cake and icing quality attributes. A two-level factorial design was used in the initial screening activity. Within the favorable region, a rotable central composite design was used to more accurately detect surfactant effectiveness. It was found that sorbitan monostearate improved cake volume and tenderness but reduced icing aeration. Polysorbate esters increased icing aeration but had relatively little effect on cake volume and tenderness. Based on the information obtained, individual blends of emulsifiers were developed to suit specific needs.

INTRODUCTION

The use of emulsifiers in shortening intended for bakers cakes and icings began with the superglycerinated shortenings of the 1930's. This product, which is still commonly used, contains about 5-6% added mono- and diglycerides. The emulsifier gives added cake volume and greater tolerance in the amount of shortening, water and sugar that can be included in the formula. However, some cakes made from superglycerinated shortening exhibit a somewhat heavy, firm texture while icings made from the shortening need improvement in body, air incorporation and texture.

Several specialty shortenings have been developed which contain, in addition to mono- and diglycerides, emulsifiers such as lecithin, propylene glycol monostearate and polysorbate 60. While these products perform well in their intended use, there is a reluctance on the part of the baker to maintain inventories of several specialty shortenings. With this in mind, work was carried out to develop an emulsifier system that would function exceptionally well in most types of bakers cakes as well as in icings.

The most efficient means of developing such a blend would be to determine the role of each surfactant in both applications and then build a blend based on this information. Because of the difficulty of proper planning and interpretation of such a large amount of data, experimental design theory and computer analysis were used.

The use of statistical methods in cake and shortening evaluation has been reported several times (1-3). Response surface methodology (4) was the basic statistical concept used. In our work, there were essentially three phases: screening studies, estimation of optimum blends and confirmation tests.

The basic statistical building block in screening experiments was the two-level factorial (5). They were used to select the blend components and the general concentrations for optimum performance. Final surface estimation requires more sophisticated statistical designs of the multilevel rotable type (6) where optimal concentrations of blend combinations can be better estimated. Finally, as the result of the information gained from the statistical designs, confirmatory experiments were performed in the region of the predicted optimum which resulted in the final formula recommendations.

EXPERIMENTAL PROCEDURES

Four quality attributes were measured for cakes, i.e., rapeseed volume displacement, volume index, symmetry index and tenderness, and one for icings, i.e., icing volume.

All cake measurements were conducted after 2.5 hr of cooling, since prior work had shown that cakes reached equilibrium by this time.

Rapeseed volume is simply the volume occupied by the cake as measured by displacement of low density rapeseeds. Volume index and symmetry index were measured by a modified version of AACC method 10-91. Instead of using the prescribed template, a mechanical device was used

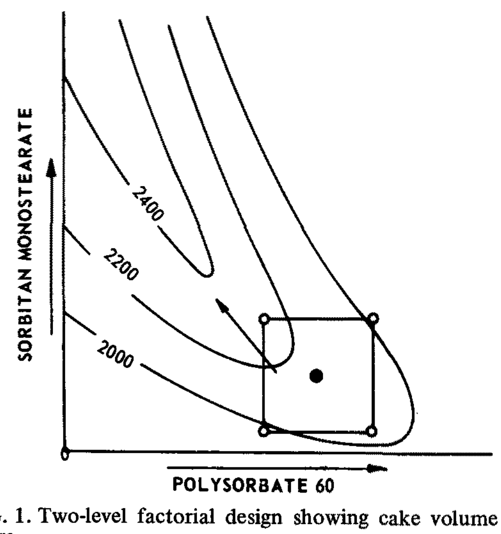

FIG. 1. Two-level factorial design showing cake volume (cc) as contours.

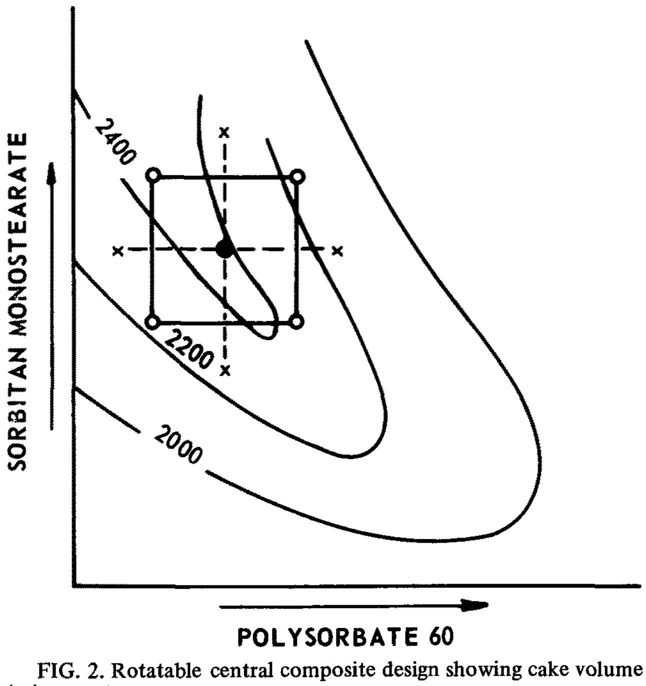

FIG. 2. Rotatable central composite design showing cake volume (cc) as contours.

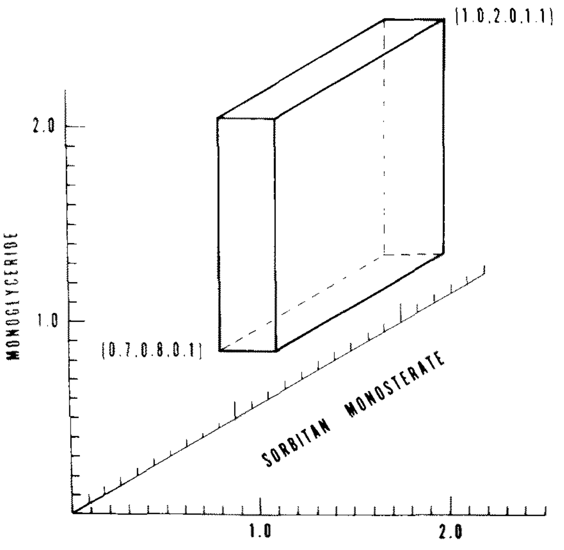

FIG. 3. Basic design points for each monoglyceride-polysorbate combination.

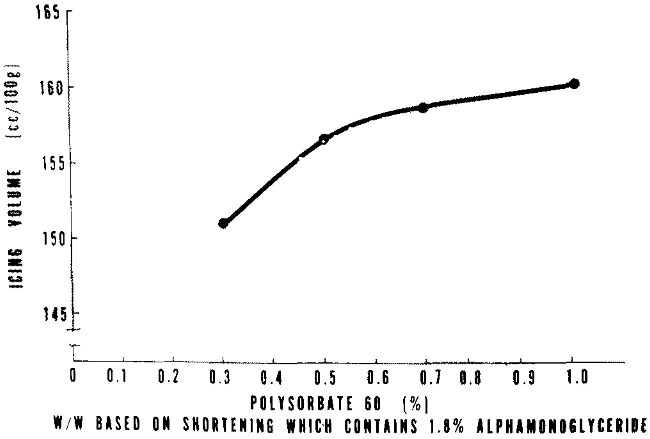

FIG. 4. Icing volume vs. Polysorbate 60 content with specified monoglyceride level. Emulsifier (w/w) based on finished shortening weight.

which measured the distance in 1/16 in. increments from the cake surface to a horizontal fixed line.

Tenderness was measured by Instron compression as follows: the cakes were vertically cut into 0.5 in. slices with an Oliver bread slicer. Tenderness was recorded as the force (g) required for a 1 in. diameter circular foot to penetrate 4 mm into the cut surface of the cake slice.

Icing volume was reported as the volume in cubic centimeters occupied by 100 g of icing. To determine this value, a known volume of icing was weighed after 24 min of mixing. The value obtained was then converted to cc/100 g.

An unemulsified plastic vegetable shortening was used as the base shortening. It had an iodine value (IV) of 78 and contained 0.4% of naturally occurring alpha monoglyceride. The base shortening and emulsifiers were melted together and warmed to 65 C. They were then plasticized in a laboratory heat exchanger and tempered at 30 C for 24 hr. Prior baking tests revealed no difference in functionality between commercially votated emulsified shortening and the same shortening melted and revotated in the laboratory apparatus.

A 130% sugar yellow cake formula was used during the experimental design phases. In Phase 1, an electric rotary oven was employed. The large number of bakings required in Phase 2 necessitated the use of a larger, gas reel oven. Final emulsifier blends were then evaluated in a variety of cake formulas baked in the electric oven. The formula used for evaluation of emulsifier performance in icings consisted of 17% shortening, 70% powdered (6X) sugar, and 13% water.

The experimental plan was divided into several sequential phases. The objective was to quickly evaluate alternate emulsifier candidates and approach the optimum emulsifier concentrations with the aid of statistical screening methods. Then, a more detailed design was used to better evaluate the response surface within a favorable region of selected candidates.

Two-level factorial designs are ideal for the screening phase. An example, shown in Figure 1, represents a hypothetical system containing two independent variables (sorbitan monostearate and polysorbate 60). The response (cake volume) is shown as contour lines. The contour lines indicate how the cake volume changes as the two variables are changed. The central arrow indicates the direction of optimum response. In this example, an increase in volume would be expected with an increase in sorbitan monostearate and a simultaneous decrease in polysorbate 60 from the region in which the factorial experiment is located.

Following Phase 1 selection of a more favorable performance region, the basic two-level factorial was augmented with additional geometric points (Fig. 2). The second design, called a rotatable central composite, is able to detect second order (quadratic) effects because of its additional points. Again, the example of Figure 2 has been simplified to two independent variables, for ease of understanding.

RESULTS AND DISCUSSION

Phase 1

The object of Phase 1 was to determine the general effect of selected surfactants on the five dependent variables. The surfactant blend systems in this screening phase were compared experimentally using four special polysorbate-monoglyceride combinations within three-factor, two-level factorial designs. Figure 3 illustrates a typical combination of surfactants and their levels. Two types of mono- and diglycerides were evaluated: a hard mono with an IV less than 3 and a plastic mono with an IV of 71. Also compared were polyoxyethylene (20) sorbitan monostearate (polysorbate 60) and polyoxyethylene (20) sorbitan tristearate (polysorbate 65). Each of the monogly-

TABLE I

Standard Errors From Replicated Bakings in Phase I

Property	Standard deviation (individuals)	Mean values	Per cent coefficient of variation
Cake volume index	2.00	70	2.9
Cake symmetry index	1.20	1.5	80.0
Rapeseed cake volume	54.8	2300	2.4
Cake tenderness	17.7	200	8.8

TABLE II

Summary of Phase I Significant Effects

Changes in Emulsifier Type/Level	Rapeseed volume, cc	Volume index	Symmetry index	2.5 Hr Instron, g	Icing volume, cc
Phase I Average	2315.2	72.2	1.4	207.3	130.7
Polysorbate					
Type: 60 to 65	144.2	4.1	None	-12.8	-7.1
Level: 0.7% to 1.0%	None[a]	None	None	None	8.2
Mono- and diglycerides					
Type: IV 71 to 3	119.0	4.2	None	-35.4	-16.0
Level: 0.8% to 2.0%	None	None	None	None	None
Sorbitan monostearate					
Level: 0.1% to 1.1%	66.2	1.8	None	-31.7	-25.4
95% Confidence limits for each effect	±17.8	±0.6	±0.4	± 6.5	± 2.3

[a]Not detected as statistically significant.

cerides were combined with each of the polysorbate esters to provide the four appropriate combinations. As shown in Figure 3, sorbitan monostearate was the third component used in each of the four blend combinations.

A special multiple comparison test procedure (7) was used to detect statistically significant effects ($\alpha = 0.05$) of emulsifier types, concentrations and their possible interactions.

The standard errors from replicated bakings in Phase 1 are shown in Table I. Each is based on 32 degrees of freedom.

The values agree well with data from previous studies. The replicated error terms described were compared with the "lack of fit" error in the Phase 1 factorials and showed no significant lack of fit. Since icing volume was not replicated in Phase 1, lack of fit error shows standard deviation 6.2, mean icing volume 130, per cent coefficient of variation 4.8. These estimates were also applied to determine the degree of basic experimental design replication required in Phase 2.

The data of Table II are summarized to show the statistically significant effect of changes in type and concentration of the various emulsifiers. For example, in cakes the use of polysorbate 65 in the blend rather than polysorbate 60 provided a rapeseed volume differential of +144.2 cc. Correspondingly, the use of polysorbate 65 also gave a larger volume index as indicated by the value of +4.1 units. Instron values were lower with polysorbate 65 than with polysorbate 60. It is probable that the increased tenderness of cakes containing polysorbate 65 is due to a density decrease commensurate with the cake volume increase. There was no change in any of the cake quality attributes with an increase in concentration of either polysorbate ester over the range of 0.7-1.0%.

Icing volume showed significance in both type and level of polysorbate ester used. In icings the effect of the type of ester was reversed; that is, the polysorbate 60 was more effective than the polysorbate 65. The former would be expected to provide an icing volume 7.1 cc larger than the latter. An increase in level of either ester from 0.7% to 1.0% would give an expected volume increase of 8.2 cc. As with all of the emulsifiers, there were no significant interaction effects in this screening portion of the study.

The type of mono employed showed significance in four of the five attributes examined. The harder mono (IV 3) produced larger cake volumes than did the plastic mono (IV 71) as measured both by the rapeseed displacement method and the volume index method. Again, the emulsifier giving the larger volume also caused a reduction in cake firmness, i.e., an increase in tenderness. Icings containing the plastic monoglycerides would be expected to have volumes 16 cc larger than their counterparts containing the hard monoglycerides. They would also have a smoother texture and body. No significant effects were observed in any of the attributes where the level of monoglyceride was changed over the range of 0.8% to 2.0%. Also, there were no significant effects of interactions.

No change in type of the third emulsifier (the sorbitan ester) was made for technical reasons. Sorbitan monostearate did show significance of concentration in four of the five attributes measured. As the level of sorbitan monostearate was raised from 0.1% to 1.1%, the cake volume was increased, 66.2 cc by rapeseed measurement and 1.8 units by volume index. Cake tenderness was increased with an increase in sorbitan monostearate as evidenced by the decrease in Instron force expected over the experimental range. Because sorbitan monostearate is known to have poor starch complexing activity, it is believed that most of this change in tenderness can be attributed to the decrease in cake density.

Icing volume values showed a dramatic decrease with an increase in the level of sorbitan monostearate. The change

TABLE III

Summary of Phase 2 Data Analysis

Characteristic	Midpoint average[a]	Significant variables	Change/0.1 % variable
Cake volume, cc	2171.4	Sorbitan monostearate	33.5
Volume index, units	66.7	Sorbitan monostearate	1.0
Symmetry index, units	-1.5	Sorbitan monostearate	0.3
2.5 hr tenderness, g	255.3	Sorbitan monostearate	-10.3
Icing volume, cc/100 g	130.9	Polysorbate 65	4.5
		(Polysorbate 65)^2	-1.1
		Sorbitan monostearate	-2.9

[a]Midpoint emulsifier blend: 0.80% mono- and diglycerides, 0.50% polysorbate 65, 0.35% sorbitan monostearate. Based on finished shortening.

TABLE IV

Cake Emulsifier Blend[a] vs. Mono- and Diglycerides in Several Cake Types

Cake type	Average rapeseed volume, cc: 0.60% sorbitan monostearate, 0.35% Polysorbate 65, 0.65% Mono- and diglycerides (IV 71)	5-6% Mono- and diglycerides
100% Yellow	2027	1881
100% White	2001	1957
130% Devils food	2103	2120
140% White	1700	1530

[a]Emulsifier (w/w) based on finished shortening.

in emulsifier level from 0.1% to 1.1% produced a drop of 25.4 cc. As expected, icings containing the higher levels of this ester were heavy and firm.

Phase 2

Since the objective was to obtain a multipurpose blend for both cakes and icings, the implications of Phase 1 led to a more complete study of the selected emulsifiers at lower concentrations. A multilevel rotatable design containing additional treatment combinations was used to detect possible quadratic effects. References to the geometry of the basic design are shown in Davies (4) where the extreme high and low points and midpoint of variables were: polysorbate 65, high 0.75, low 0.25, center 0.5; mono- and diglycerides, high 1.05, low 0.55, center 0.8; sorbitan monostearate, high 0.6, low 0.1, center 0.35. To increase the precision of the estimated effects, the complete design, requiring 20 cakes per baking, was repeated on five different days. Differences attributable to technicians, confounded within days, showed no significance.

Sorbitan monostearate was chosen as one of the emulsifiers to be studied further because of its benefits in cake volume. Polysorbate 65 was chosen over polysorbate 60 because its improvement of cake volume appeared to be greater than the improvement of icing volume by the polysorbate 60. The plastic mono was chosen rather than the hard mono because the latter produced stiff, low volume icings while the plastic mono produced creamy icings.

Table III summarizes the results of the work of Phase 2. All four cake quality attributes were found to exhibit statistically significant changes with variation in emulsifier level. All four were related only to sorbitan monostearate content. Rapeseed displacement volume and volume by volume index both exhibited positive linear changes with increases in sorbitan monostearate.

The values shown in the last column permit estimation of the change in attribute character as a move is made away from the midpoint emulsifier blend. For example, in line 1 a change in the sorbitan monostearate content of the shortening from 0.35% to 0.45% would be expected to produce a change in volume from 2171.4 to 2204.9 cc. Similar changes would be expected in the other characteristics. An increase of 0.10% in sorbitan monostearate would reduce center dip from -1.5 to -1.2 and improve tenderness by reducing the necessary compression force from 255.3 to 245.0 g.

In icing volume, which is simply overrun, both sorbitan monostearate and polysorbate 65 exhibited significant effects. The influence of polysorbate 65 consisted of a positive linear effect and a negative squared effect. The latter means that above a specified level the incremental improvement in icing volume imparted by the polysorbate 65 diminishes. In the standard icing test used in this work the change began to level off at approximately 0.70%. Further increases in volume were achieved when the polysorbate 65 level was carried beyond 0.70%; however, the effect was diminished.

Within the area of experimentation, sorbitan monostearate exhibited a negative effect on icing volume. That is, as the level of sorbitan monostearate was increased, the icing volume decreased. Therefore, blends to be used for both cakes and icings must have sufficient polysorbate ester to overcome the effects of the sorbitan ester on icing volume.

Icing body and moisture drainage were subjectively noted and generally the body of icings having higher polysorbate 65 content were lighter and smoother. Increased levels of sorbitan monostearate produced a heavier, stiffer body, especially when combined with low levels of polysorbate 65. Drainage of moisture from the icings was generally lessened by addition of higher levels of polysorbate ester.

Phase 3

Knowledge of the effects of each of the surfactants on cake and icing performance allowed an orderly development of specific emulsifier blends. For specialty cake

TABLE V

Evaluation of Several Multi Purpose Emulsifier[a] Blends

Blend composition	Cake volume, cc	Icing volume, cc/100 g
A 1.00% Mono- and diglyceride 0.58% Polysorbate 60 0.42% Sorbitan monostearate	2252	135
B 1.45% Mono- and diglycerides 0.90% Polysorbate 60 0.65% Sorbitan monostearate	2335	136
C 1.50% Mono- and diglycerides 1.08% Polysorbate 60 0.42% Sorbitan monostearate	2271	145
5-6% Mono- and diglycerides	2195	145

[a]Emulsifier (w/w) based on finished weight.

shortening, the systems given in the Phase 2 study provide a nearly optimum emulsifier formula.

The statistical evaluation suggested a level of 0.60% sorbitan monostearate, 0.25% polysorbate 65, and 0.55% mono- and diglycerides in the shortening would give a cake volume of approximately 2255 cc vs. an average of 2195 cc for a typical superglycerinated shortening. Only one of the blends of Phase 2 contained 0.55% mono- and only one contained 0.25% polysorbate 65. Therefore, an increase of 0.1% was made in each of these two emulsifiers to verify results within the favorable region. Average values of replicate bakings showed there was an improvement of about 25 cc with the higher levels. More important was the fact that this emulsifier combination gave cakes 140 cc larger than the superglycerinated control shortening and 80 cc greater than predicted by the computer analysis.

The blend of 0.60% sorbitan monostearate, 0.35% polysorbate 65, and 0.65% mono- and diglycerides was tested in several different cake formulae with results as shown in Table IV. In all cakes except the devils food, the blend noticeably improved volume. It also gave substantial improvement in grain, crust appearance and tenderness. Performance in the 130% devils food was equivalent to that of the superglycerinated control.

The plastic mono had been chosen for the work in Phase 2 primarily because of its texture improvement in icings. Since the cake blend described above is not best suited to icings, a hard mono could be used as a replacement.

The data of Phase 2 clearly indicate the function of sorbitan monostearate and polysorbate 65 in icings. Phase 1 results show that polysorbate 60 is slightly more effective than polysorbate 65 as an icing improver. It was also observed that the effect of the polysorbate esters approached a maximum in the area of 0.70% of the shortening. This data, however, was developed in systems which always contained at least a small amount of sorbitan monostearate. Therefore, work was carried out on blends of monoglycerides and polysorbate 60 to establish the role of the mono and to determine the proper use level of both surfactants.

Duplicate evaluations were made of shortenings containing various levels of polysorbate 60 plus 1.8% alpha mono. The results, as plotted in Figure 4, basically agreed with statistical predictions. The major change in volume improvement occurs over the range 0.3-0.7% polysorbate 60. Beyond that level the effect on icing volume is diminished, although other improvements such as added moisture retention are achieved with additional polysorbate 60. The addition of the glyceride esters improved the general appearance and texture of the icing.

With the information learned from the cake results and the icing results, it was then possible to quickly find the proper blend of emulsifiers which would function well in a wide variety of bakery formulas. An icing volume of 145 units was set as a minimum icing performance and a cake volume of 2200 cc was set as the minimum cake performance. These values are average performances of a typical superglycerinated shortening. According to the data analysis summaries shown in Table II and Table III, this would require a minimum of 0.42% sorbitan monostearate and approximately 0.9% polysorbate 60. In addition, 0.8-2.0% mono- and diglycerides would be needed for cake balance and icing texture.

With this information as a guideline, several combinations in this range were tested in cakes and icings. As Table V shows, changes caused by specific emulsifiers followed fairly well the predicted functionality trends. As the level of polysorbate 60 was increased icing volumes rose. An increase in sorbitan monostearate caused a substantial reduction in icing volume but produced an increase in cake volume.

Based on the previously stated requirements set for cake and icing performance, the composition of blend C best fulfills the objectives. It gave cake volumes of approximately 75 cc over that of the superglycerinated control. Yet the latter had 5-6% emulsifier while blend C was used at a level of 3% of the finished shortening. The icing volumes of shortening containing blend C were about equal to those of the control. However, the general appearance of those icings containing the blend was noticeably smoother than the control icings. They also exhibited less moisture drainage.

The blend has been evaluated in many other cake and icing formulas and has given excellent performance. It is particularly effective in giving lean cakes a richer appearance and mouthfeel. In cream fillings aeration is improved and the fillings have a smoother, more homogeneous appearance.

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