Finished Product Applications for Process Cheese Show Improved Appearance Using Design of Experiments

Land O'Lakes continuously seeks out methods for improving the flavor, appearance, and shelf life of its products. Recently, Land O'Lakes researchers set out to improve a new deli process cheese formulation, without impacting its other attributes.

Senior Scientist Mostafa Galal (PhD) and Senior Technologist Michael Scheller used design of experiments (DOE) to create a general factorial design using two factors: emulsifier ratio (four levels) and emulsifier type (two). For this article emulsifying salts will be referred to generally as "emulsifiers." Emulsifier "type" refers to the emulsifier source and delivery system, which are not explicitly stated for reasons of confidentiality. The goal was to find out which ratio and type of emulsifier would result in the desired improvement in appearance without affecting the other responses.

Land O'Lakes is a farmer-owned cooperative that offers farmers, local cooperatives, and customers across the nation an extensive line of agricultural supplies as well as production and business services. It is also a leading marketer of dairy-based products for consumers, foodservice professionals and food manufacturers. Land O'Lakes produces a full line of quality dairy products including: butter and butter blends, margarine and spreads, dairy case cheese, and deli cheese.

Need to improve appearance of cheese sauce using new formulations

"We are continually examining the recipes of our current products in order to find ways to exceed our customers' expectations," Scheller said. "Recently, Mostafa Galal developed a new process cheese formulation that seemed promising but exhibited some Maillard browning when the product was melted in finished product applications. The browning had no effect on the taste or safety of this product, however most consumers prefer to have cheese maintain its white color when melted in Queso sauce." Maillard browning is caused by a chemical reaction between an amino acid and a reducing sugar, usually requiring heat. "This problem was not easy to solve because the formula contains many different ingredients and multiple processing steps," Scheller said. "The effects of these ingredients and the processing steps could interact with each other, which makes it difficult to get a grip on the problem using conventional one-factor-at-a-time (OFAT) experiments. We began using DOE because it lets us look simultaneously at the effects of all the factors. It also provides statistical analysis that helps separate the single factor and multiple factor effects and allows us to optimize the values of each factor."

"You can use spreadsheets or general statistical software to create and analyze designed experiments," Scheller said. "But I have found that a software package tailored specifically to the task does the job in less time and produces more meaningful results. Stat-Ease's Design-Expert[®] software (Minneapolis, MN) makes a wide range of DOE techniques accessible to a user who is not specially trained in statistics. An important advantage of this program is the technical assistance provided by the software vendor. Their technical support team has considerable expertise in applying DOE to realworld industrial problems. Their support was instrumental to the success of this application."

Designing the general factorial experiment

There are two different emulsifiers used in the product. Their total amount is fixed but the relative amount of each emulsifier can vary. Taking a suggestion offered by Stat-Ease's "Stat Help," Scheller decided to use the ratio of the two as a single factor instead of treating each emulsifier separately. This reduced the number of factors required from three to two which in turn substantially reduced the number of runs for the experiment. The factors are:

- A) Emulsifier type (Type 1 or Type 2).
- B) Ratio between different emulsifiers used in product (A, B, C or D)

The goal was to reduce the browning without having any negative effects on either flavor or meltability. Browning can be measured using the Hunter Lab Colorimeter. Using the Hunter, the color space variable "a" is the parameter most useful for monitoring browning in Queso sauce. Positive "a" is red and negative "a" is green. One of the objectives in this experiment is reducing the Hunter "a" level, which corresponds to less browning. At the same time, the meltability of the cheese needs to be maintained at least at the level of the current product. Meltability is measured by cutting a disk of cheese of a specified size, and placing the disk in a glass Petri dish. A technician heats the Petri dish in an oven at 350°F for 7 minutes, then pulls the dish out and measures the diameter of the circle that the melted cheese has formed.



Photo 1: Meltability is measured by the diameter of the circle the melted cheese has formed

In this experiment, the pH of the cheese was used as a benchmark to monitor potential changes to the flavor. The pH needs to be high enough to avoid creating an acidic taste, but not too high because that would affect shelf life. The responses for the experiment were thus:

- 1) Browning (Hunter "a" value)
- 2) Meltability
- 3) pH

Standard Order	Run (Random)	Factor A	Factor B
1	11	А	Type 1
2	2	А	Type 1
3	3	В	Type 1
4	10	В	Type 1
5	4	С	Type 1
6	16	С	Type 1
7	1	D	Type 1
8	5	D	Type 1
9	8	А	Type 2
10	13	А	Type 2
11	15	В	Type 2
12	6	В	Type 2
13	12	С	Type 2
14	14	С	Type 2
15	9	D	Type 2
16	7	D	Type 2

Figure 1: Designed experiment

Scheller input the factors into Design-Expert software and generated a 16run experiment in which each of the 8 design points were replicated as shown in Figure 1. The order is randomized to reduce bias from lurking time-related variables such as change in ambient temperature, or humidity and the like. The results were then entered into the software and analyzed.



Figure 2: Meltability model graph

First the meltability response was considered. A meltability score within the range 3 to 6 was desired for this trial product. Figure 2 shows a graph of the model determined by Design-Expert software for meltability. The model has an F-value of 14.99 which indicates that there is only a 0.05% chance (p = 0.0005) these results could be caused by noise. "The graph shows that for this experiment when the Emulsifier Type 1 is used, meltability is consistently within specification," Scheller said. "On the other hand, when Emulsifier Type 2 is used, meltability is all over the map." The bars indicate the least significant difference (LSD) for 95% confidence. These show a clear separation in meltability by Type, see emulsifier ratio D, for example.



Figure 3: pH model graph for Type 1 emulsifier

Next the model for the pH response was analyzed. It generated an F-value of 16.02 (p = 0.0002). Scheller eliminated the Emulsifier Type 2 runs from this analysis since the meltability model already indicated that this emulsifier produced unacceptable results. To eliminate acidic flavor notes, the ideal pH for this formulation needed to be between approximately 6.00 and 6.15. The model in Figure 3 shows that an emulsifier ratio of D provides good pH results.



A: Emulsifier Ratio Figure 4: Hunter "a" model graph for Emulsifier Type 1

Identifying a workable solution

Finally, the effect of emulsifier ratio on the browning of the cheese was analyzed as indicated by the Hunter "a" value. The model produced an F-value of 9.04 (p = 0.0021). The results indicate that an emulsifier ratio of D provides a Hunter "a" value of about -1.83 which is a significant improvement over the baseline formulation for this product. "DOE helped us show that it is possible to improve the appearance of the new product in cheese sauce application without adversely affecting other characteristics," Scheller said.

"This was the first time that Mostafa and I applied DOE on the project," Scheller concluded. "The application demonstrated the ability of DOE to provide a degree of rigor that imparts more confidence in the results than our previous one-factor-at-a-time experimentation. DOE explores the entire design space which helps eliminate concerns that a better answer might be lurking somewhere in the unknown. For certain applications, DOE can reduce the number of runs required to achieve meaningful results, providing substantial savings in time and materials. Due to the relative ease of using Design-Expert, the technical assistance provided by the software vendor, and the potential for success of these applications, DOE will become more of an integral part of product and process development at Land O'Lakes."

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