stat teaser

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Bread DOE Part 2: Semifold Confirms Cause for Failure

Mark's Experiment
by Mark J. Anderson

Mark's DOE on machine-made bread shows how a clever application of statistical methods quickly screens alternative ingredients to see which, if any, impair the desired reaction.

In the last issue of the "Stat-Teaser" I told you about how I tried to cut corners when baking machine-made bread by using cheaper raw materials. My first DOE involved the following factors:

A. Liquid: Water (-) or Milk (+)
B. Oil: Butter (-) or Margarine (+)
C. Flour: Regular (-) or Bread (+)
D. Yeast: Regular (-) or Bread (+)

I decided that doing the 16 loaves required for the full factorial would take too long, so I designed an experiment that required only 8 loaves - the half-fraction. I did this with the expectation that nothing would be significant; therefore, I could use the inexpensive ingredients. However, I was wrong - materials did matter. As pictured in my prior report, I accidentally made some doughy bricks. Surprisingly, the bread consistently failed to rise (coded as 0 for the response labeled "Rise") when I used water (A-) and regular flour (C-) - at least that's the way it looked at first glance (see standard ("Std") orders 1 and 3 in Figure 1 below).

Upon closer inspection, notice that column AC follows the same pattern as BD. Thus, these two interactions cannot be separated - they're statistically aliased. I didn't really think that the interaction of oil (factor B) and yeast (D) made sense. However, just to be sure I wasn't making a wrong assumption, I perfomed a "semifold" on my bread DOE to free up

Std.	A	B	C	D	AB	AC	AD	BC	BD	CD	ABC	Rise
1a,b	-	-	-	-	+	+	+	+	+	+	-	0,0
2a,b	+	-	-	+	-	-	+	+	-	-	+	1,1
3a,b	-	+	-	+	-	+	-	-	+	-	+	0,0
4	+	+	-	-	+	-	-	-	-	+	-	1
5	-	-	+	+	+	-	-	-	-	+	+	1
6	+	-	+	-	-	+	-	-	+	-	-	1
7	-	+	+	-	-	-	+	+	-	-	-	1
8	+	+	+	+	+	+	+	+	+	+	+	1

Figure 1: First Bread DOE: Design Layout in Coded Levels with Interactions Shown

--Continued on page 3.

3M Enhances SPE Disk Performance with DOE

Solid phase extraction (SPE) disks created using designed experiments take a special form with a process patented in 1989 by the 3M™ Company of St. Paul, MN. The extraction disks, known by the trademarked name Empore™, are prepared by blending polytetrafluoroethylene (PTFE, generic for Teflon™) with particle adsorbents to make thin membranes from which the disks are produced. Ranging in size from 6mm to 90mm in diameter, these disks are used to capture everything from pesticides in drinking water to drugs in biological samples.

Several months ago, various PTFE membranes were reformulated to make faster-flowing disk products that would be less prone to plugging. SPE disks that plug during extractions can result in incomplete or inconsistent analyte recovery, costing laboratories valuable time -- especially if an entire analysis procedure must be repeated. Although the 3M Company had previously developed a successful Empore extraction disk, the company saw the need for improvement to increase customer satisfaction. Both new and established customers wanted a durable disk to rapidly extract analytes without the problems often associated with competitive SPE products. The goal in improving Empore technology was to redesign the disk to make a strong, fast-flowing product capable of consistently yielding high recovery rates.

Empore extraction disks are an important component of 3M's 96-well plates. In this application, disks are punched from a large membrane and placed in an array of 96 wells, forming what is known as a 96-well plate. These plates are used in hospitals, pharmaceutical companies, and bioanalytical labs to analyze blood and urine specimens, conduct drug screens, or perform drug metabolism studies. An individual well in a plate of 96 is one analysis, meaning 96 specific samples can be prepared per plate. (See Figure 1.)

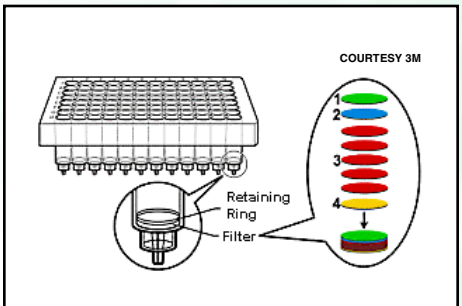

Figure 1: An improved 96-well extraction plate patented by 3M™ provides high analyte recovery while maintaining excellent flow characteristics.

A plate of 96 wells measures approximately five inches by three inches, with each well holding up to 2.5 ml of solution. The bottom of each well contains a pre-filter and an Empore extraction disk. Small retaining rings secure each disk in place at the bottom of every well. Customers fill the plates manually with micropipettes or insert them into robotic machines that prepare and extract the plates according to preset software routines. Typically, robotic instruments place a small amount of sample into each of the 96 wells, then apply a vacuum that draws liquid through the disk. As the drug of interest is drawn through each well, it is captured by the Empore membrane. In a subsequent step, solvent is added to the well to remove or elute the drug analyte from the membrane. The eluted material is placed into a collection plate (also 96 wells) before being injected into HPLC or LC/MS instrumentation for analysis.

3M's solution for an optimized extraction disk formula required analyzing how process variables or factors were interacting - an ideal scenario for DOE. Using Design-Expert®, 3M conducted a series of reformulation experiments. In this effort, experimenters studied different Empore disk formulations for membranes of various thickness. The project determined the effects of formulation changes on the membrane by analyzing various responses such as flow rate and other responses. Four formulation factors were studied:

1. Organic solvent amount
2. Particle binding materials
3. Dough solids or water added
4. Number of mill passes of Empore pre-disk dough through a proprietary mill process.

After reviewing several experimental options, a four-factor, fractional-factorial, two-level experiment was chosen as the best approach. A partial factorial design protocol yielded eight formulas. This was found to be a desirable number of experiments, considering the number of analytical tests that would subsequently have to be conducted on each membrane. Such tests were required in order to evaluate the effect of various formula changes generated by the software.

Two-level (high and low) testing used in DOE is highly effective. The software uncovered the Empore formula's most dominant factor interactions and identified the variables that had the greatest effect on disk performance (See Figure 2.)

The results of this study yielded new

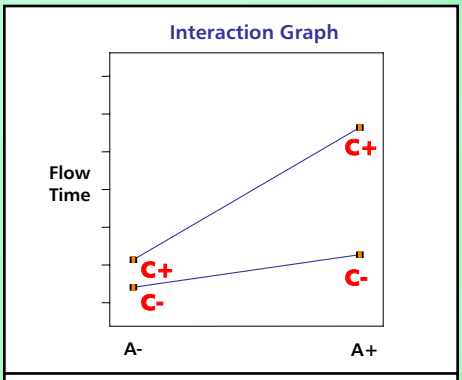

Figure 2: The Interaction of Factor A and C Affects Empore™ Disk Product Flow Time.

--Continued from page 1.

and confirm the AC interaction. This technique was developed specifically to improve resolution of two-level fractional factorial designs, such as that used for bread-making, with aliased two-factor interactions (2fi's). It requires that you:

1. Lay out a single-factor foldover from the original design. (Suggestion: choose a factor that's involved in the largest significant two-factor interaction that's aliased with other 2fi's.)
2. Perform only half of the foldover runs by selecting those where the chosen factor is either at its low level or high level, whichever you believe will generate the most desirable response(s).

Std.	A: Liquid	B: Oil	C: Flour	D: Yeast	Taste	Rise
9	Water -	Butter -	Bread +	Regular -		
10	Milk +	Butter -	Bread +	Bread +		
11	Water -	Marg +	Bread +	Bread +		
12	Milk +	Marg +	Bread +	Regular -		
13	Water -	Butter -	Regular -	Bread +	5.0	0
14	Milk +	Butter -	Regular -	Regular -	6.5	1
15	Water -	Marg +	Regular -	Regular -	5.5	0
16	Milk +	Marg +	Regular -	Bread +	5.5	1

Figure 2: Semifold on Bread-Making Experiment (Second Block Only)

Std.	A	B	C	D	AB	AC	AD	BC	BD	CD	Rise
13	-	-	-	+	+	+	-	+	-	-	0
14	+	-	-	-	-	-	-	+	+	+	1
15	-	+	-	-	-	+	+	-	-	+	0
16	+	+	-	+	+	-	+	-	+	-	1

Figure 3: Second Bread-Making DOE: Coded Levels with Interactions Shown

Figure 2 shows how I applied a semifold to my bread-making DOE. To de-alias the AC interaction, I chose factor C (flour) as the single column I folded over (notice how the levels go opposite from the original block of runs shown in Figure 1). Then I performed only half the laid-out runs - the ones with regular flour (because I am cheap!).

Now it could be seen that the combination of water and regular flour caused the bread-making to fail (zero rise). As shown in Figure 3, this finding is unequivocal because the semifold of four runs de-aliased the interaction of factors A and C from that of B and D. As you can see in the green columns, the patterns no longer match.

Therefore, I concluded that the interaction of factors A and C accurately described what affected the bread-making process. I must avoid the combination of regular flour and water.

What you do as a result of running screening designs like the one I first ran on my bread depends on which, if any, effects come out significant. Here's a general strategy for follow-up:

Scenario 1 - Nothing significant: Look for other factors that affect your response(s).

Scenario 2 - Only main effects significant: Change these factors to their best levels.

Scenario 3 - Two-factor interaction(s) significant: De-alias by performing a semifold.

By following this strategy you will increase your odds of uncovering breakthrough main effects and interactions at a relatively minimal cost in experimental runs. This is an ideal situation - akin to baking your bread and eating it too. In today's extremely competitive world it boils down to the "knead for speed" in making more and more "dough."

- Mark Anderson

(Mark wrote a much more detailed article on this case study that's been tentatively accepted for publication in a technical magazine. You can view this at www.statease.com/pubs/breaddoe.pdf .)

3M Disk Performance DOE (Continued)

Empore formulas that provide faster flow rates with no membrane plugging problems -- while maintaining high analyte recovery. These benefits were obtained while maximizing physical disk strength and the integrity needed in a finished consumer product. High-performance membranes mean that customers can use Empore products with the assurance that they will provide the best possible performance in all applications. With the statistical assistance of Design-Expert software, the improved extraction disk now meets the needs of 3M customers worldwide with its faster-flowing, high analyte recovery capabilities.

By Craig Perman
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3M Company
Filtration Products Laboratory
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New Class! - Statistics for Technical Professionals

09/01

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