Design of experiments reduces product cost while improving performance

By Jason Pandolfo

Industrial product development can often be a frustrating process, especially in the case of formulations. Many commercial formulated products can have 20 or more components. With so many possibilities for multi-component blending effects to impact performance, it is difficult to optimize a formulation without design of experiments (DOE).

It is not uncommon for existing products to be slightly modified for the purpose of improving performance or reducing costs. Oftentimes, these changes will have the desired effect with no unexpected negative impacts on critical product behaviors. However, there will be some cases where a seemingly simple modification may result in difficult-to-explain problems with performance. The root cause of the problem may not be easy to determine, much less fix.

Quaker Houghton of Conshohocken, PA modified one of their high-performance metalworking fluids to improve performance traits while reducing raw material costs. The modification met all of the goals except for machinability, a key attribute of metalworking fluids. Final testing determined that the product's lubricity unexpectedly worsened due to the changes. Lubricity is an indicator of machining performance, so this discovery was troubling.

Shortly after this performance decline was verified, the laboratory shifted focus to investigating the cause of the lubricity issue. Experimental design would be the most effective and efficient means to investigate this case. Quaker Houghton chemists have been trained in DOE and equipped with software tools by Stat-Ease, Inc. (Minneapolis, MN). They turned to the DOE toolbox to design statistically advantageous studies that provided understanding of the underlying issue as well as a path to optimizing lubricity.

The Problem

The original formulation (Product A) needed an overall reduction of raw material costs while increasing product stability. Additionally, the new formulation (Product B) needed a higher level of reserve alkalinity, so it incorporated a higher level of amines than Product A. The differences between the two formulations are summarized in Table 1.

Table 1: The Reformulation of Product A to Product B

Raw Material	Original Product (A)	Modified Product (B)
Carrier Oil	Moderately Expensive	Similar, Less Expensive
Lubricity Additives	No Changes	
Amine Package	Needed higher alkalinity	25% increase in amine content
Emulsifiers	Expensive Ethoxylated Emulsifier	Similar, Less Expensive Ethoxylated Emulsifier
	--	Additional Stabilizing Emulsifier (1.00% on total)

The laboratory did not expect any major performance differences from this straightforward modification to Product B. However, the new product's tapping-torque, a measure of machinability, increased by a surprising amount…15% over Product A. This indicates that Product B has worse lubricity than Product A. Considering that the lubricity-additive packages remained the same, this unexpected result required further study.

To investigate the root cause of the increased tapping-torque, the laboratory team deployed Design-Expert® software from Stat-Ease to lay out a full, 16-run, two-level factorial design on the four primary ingredients. Table 2 summarizes the experiment-design specifications.

Table 2: Factors and Levels for First DOE

Factor	Category	Factor Level	
		Low (Product A)	High (Product B)
A	Carrier Oil	More expensive	Less Expensive
B	Amine Package	Lower alkalinity	Higher alkalinity
C	Ethoxylated Emulsifier	More expensive	Less expensive
D	Additional Emulsifier	0%	1%

Due to resource constraints in the laboratory, it was not possible to replicate any of the samples that were run in this experiment. However, the software showed that this experiment provided adequate resolving power (more than 80%) to resolve any multi-factor interactions on tapping torque. (For details on resolving power, see reference 1.)

After analyzing the tapping torque for each of the 16 formulations, Design-Expert’s specialized factorial-analysis tools revealed that Factor B, the change to higher alkalinity in the amine package, caused a significant increase in the tapping torque. The Pareto plot—an ordered bar-chart red-lined at 95% confidence level, shown in Figure 1, visually illustrates the results of the experiment.

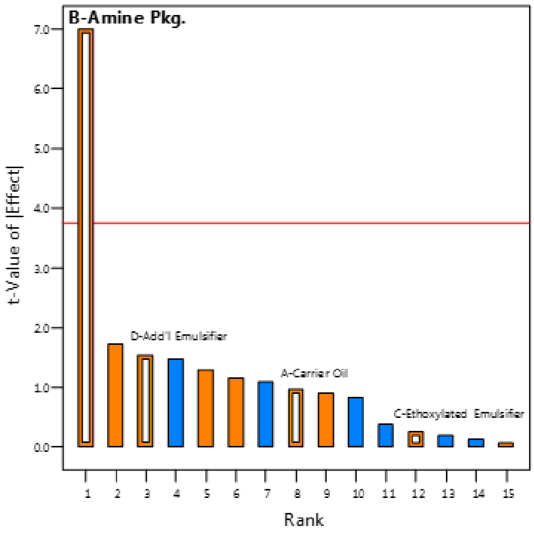

Figure 1: Pareto plot of effects

The amine package is ranked first, which signifies that it is the strongest effect relative to the other main and multi-factor effects. The t-value of the amine package is considerably higher than the significance threshold, which stems from a t-test which is tuned to assessing multiple comparisons. The three other main effects (D, A and C) are labeled only for reference sake—they fall far short of the significance threshold and are therefore not impacting the response.

Exploring the Amine Package:

The impact of changing the amines was not expected, as amines are not generally regarded as key lubricity drivers. The next logical step was to experiment on individual amines to see how they might be manipulated to reduce tapping torque while keeping all other specifications in line. Design-Expert provides specialized tools for mixture DOE, making it easy to set up a second experiment to study six amines, labeled A through F (undisclosed for proprietary reasons), with the ranges shown in Table 3.

Table 3: Components and Levels for Second DOE

Component	Component Range (milliequivalents)	
	Low Level	High Level
Amine A	0	82.00
Amine B	0	42.70
Amine C	0	27.60
Amine D	15.20	30.50
Amine E	0	22.50
Amine F	0	39.70
All Else (G)	0	67.95
Total	***135.90***	

Amines A, B, and C stemmed from the initial reformulation (Product B). The lab team added three new amines (D, E and F) to see if they might identify new drivers for tapping torque.

The "All Else" component added inert material (water) which served to allow a varying range of total amine equivalents while not violating the "unity" rule of mixture designs in which the sum of all components must equal 100%. In effect, the amines as a whole were tested over a range from 67.95 to 135.90 total milliequivalents. This approach would allow the project team to assess the performance impact of total amine level in addition to that of individual amines.

Anticipating nonlinear blending effects, that is, possible synergisms or antagonisms between components, the laboratory team chose an optimal custom design for a quadratic mixture-model which emphasizes two-component blending effects. (For details on mixture design and analysis tools used for this study, see reference 2.) They bolstered the experiment with 10 check blends and 5 replicates. Aided by the software, the 44 resulting runs in the experiment were divided into two blocks of 22 for the sake of convenience.

The results for tapping torque exhibited a strong fit to the designed-for quadratic model (>0.95 R^2). This confirmed that although amines are critical for controlling alkalinity of metalworking fluids, they also affect machinability. The trace plot in Figure 2 reveals the impacts of the main components.

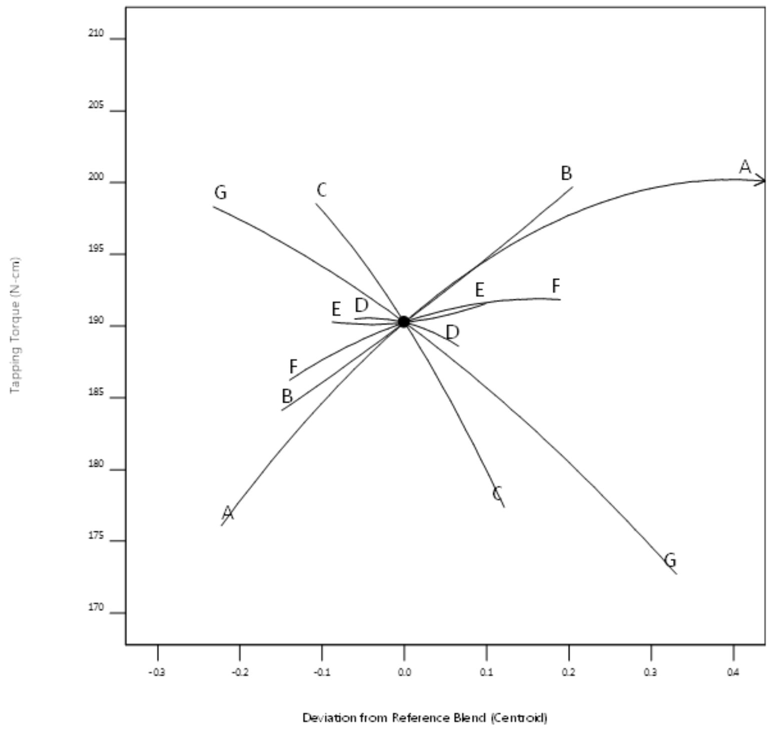

Figure 2: Trace Plot of Amines

The trace plot is read by starting at the point in the middle, the geometric center of the design space, and tracing along an individual line. Moving to the right shows the effect of increasing the amount of an individual amine while proportionally lowering the other components. It reveals that Amine A creates the greatest increase in tapping torque (and thus reduction in machinability), followed by Amine B. The trace plot shows that Amine C can be increased to counteract the negative impact of Amine A and B. The relatively flat traces of new Amines D, E and F indicate that they create small effects on the tapping torque, but they remain in play as substitutes. Component G, the non-amine portion of the formulation, produces the most revealing trace—it creates a substantial decrease in tapping torque at a higher proportion. In other words, higher overall-levels of amine degrade machining performance.

Searching out a new formulation based on the results from the mixture DOE

Traditional one-factor-at-a-time (OFAT) methods of experimentation, although extremely inefficient, can identify a solution by hit or miss. However, multifactor DOE produces an extremely valuable predictive model which OFAT methods cannot provide. In this case, the model enabled Quaker Houghton's formulators to "dial in" the tapping torque by manipulating amines while also taking their costs into account—plus all other critical-to-quality attributes measured and modeled. This “sweet spot” can be adjusted as needed in ‘what if’ fashion to produce variations in the metalworking fluids as requested by customers.

Keeping the optimization focused only to the goal of minimizing tapping torque led to a new formulation that substitutes Amines A and B with two of the new ones—E and F (keeping Amine C in the formulation), creating a significant improvement as shown on the side-by-side response surface graphs in Figure 3.

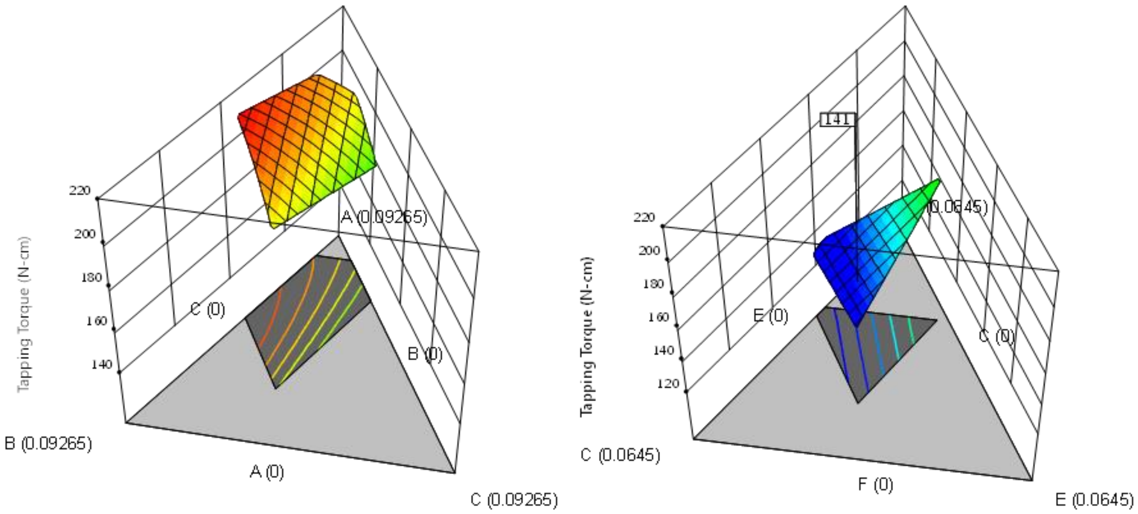

Figure 3: Response surfaces for Product B (left) versus a far better formulation (right)

The response surface on the left (Formulation A) has a considerably higher tapping torque than the proposed amine package response surface on the right. It is possible to generate any number of amine combinations and use the software to predict performance, thus removing a tremendous amount of guesswork from the product development process.

Conclusion

The impact of this study was immediate and long-lasting. First, the learnings from the factorial investigation ensure that amine packages will not be taken for granted in the future. The follow-on mixture design is a durable model that will enable Quaker Houghton’s development chemists to design amine packages for optimal lubricity in similar product matrices which translates to the best possible machining behavior. Furthermore, the cost-function added to the model allows further optimization for both performance and favorable raw material costs. For an expenditure of a few days of work, Quaker Houghton gained a model that can potentially save many man-hours of development time in the future.

This case study highlights the value of experimental design in the commercial R & D environment. The DOE toolbox provided a means for unmistakably identifying the source of the problem. As DOE is an iterative process, the follow-on experiment further explored the combined impact of several drivers on the response, revealing interesting effects for individual components. The time that was invested yielded high-impact results that were backed by statistics. All told, compared to their peers who remain mired in old-fashioned OFAT experiments, practitioners of DOE will invariably learn far more about their product with statistically-valid, reliable results, and often in a shorter amount of time.

For more information about Design-Expert and DOE, contact:
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References:

1. *DOE Simplified: Practical Tools for Effective Experimentation, 3rd Edition*, Anderson and Whitcomb, Productivity Press, NY, NY, 2015.
2. *Formulation Simplified: Finding the Sweet Spot through Design and Analysis of Experiments with Mixtures*, Anderson, Whitcomb and Bezener, Productivity Press, NY, NY, 2018.

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