Design of Experiments for Coatings

Mark J. Anderson* and Patrick J. Whitcomb

Stat-Ease, Inc., 2021 East Hennepin Ave, #480 Minneapolis, MN 55413

**Telephone: 612/378-9449 (Ext 13), Fax: 612/378-2152, E-mail: Mark@StatEase.com*

1.0 INTRODUCTION

The traditional approach to experimentation changes only one process factor at a time (OFAT) or one component in a formulation. However, the OFAT approach doesn't provide data on interactions of factors (or components), a likely occurrence with coating formulations and processes. Statistically-based design of experiments (DOE) provides validated models, including any significant interactions, that allow you to confidently predict response measures as a function of the inputs. The payoff is identification of 'sweet spots' where you can achieve all product specifications and processing objectives.

The strategy of DOE is simple and straightforward:

1. Use screening designs to separate the vital few factors (or components) from the trivial many.
2. Follow up by doing an in-depth investigation of the surviving factors. Generate a "response surface" map and move the process or product to the optimum location.

However, the designs must be tailored for the nature of the variables studied:

- Components in a product formulation
- Factors affecting a process.

Traditionally the experiments on formulations versus processing are done separately by chemists and engineers; respectively. Obviously collaboration between these two technical professions is essential to the success of any study. Furthermore, mixture components can be combined with process factors into one design for final optimization. In other words, you can mix your cake and bake it too, but this should be done only at the final stages of development – after narrowing the field of variables to the vital few.

We will devote most of this short discussion to process screening because these designs are relatively simple, yet incredibly powerful for making breakthrough improvement. Mastering this level of DOE puts you far ahead of most technical professionals and paves the way for more advanced tools geared to optimization of processes or formulated products.

2.0 STANDARD TWO-LEVEL FACTORIAL DESIGNS

Two-level factorial design involves simultaneous adjustment of experimental factors at high and low levels. By restricting the tests to only two levels, you minimize the number of runs: Don't bother setting three levels of every factor until you get close to the optimum. By then the number of factors (k) should be much reduced via screening studies. The math is simple – as k increases, the number of runs goes up exponentially. Therefore, apply 2^k (two-level) designs when screening many factors versus 3^k to optimize the vital few.

The contrast between two levels provides the necessary driving force for process improvement via the 2^k designs. This parallel-testing scheme is much more efficient than the traditional, serial approach of one-factor-at-a-time (OFAT) as illustrated by Figure 1, which shows designs with equivalent power for calculating effects from the contrasts between high and low levels.

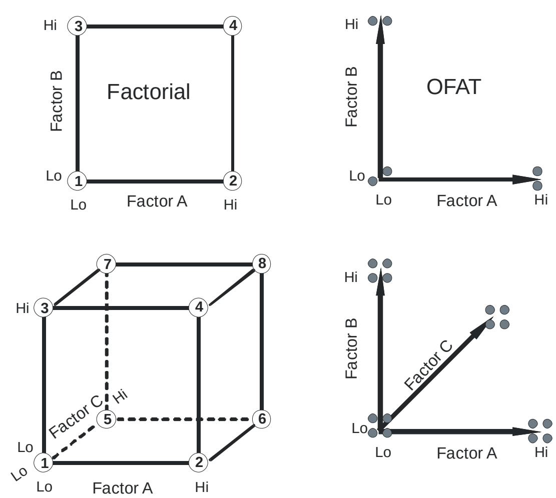

Figure 1: Comparison of two-level factorial designs (left) versus OFAT (right) for two factors (top) versus three factors (bottom)

For example, the factorial design at top-left offers two runs at the high level for A and the same for the low. Similarly, factor B is run twice at the high level as well as the low. Therefore, the OFAT design at the upper right has two runs each at the high levels to provide similar power for effect estimation. However, this necessitates a total of 6 runs for OFAT versus only 4 for the two-level factorial. The efficiency advantage for factorials becomes more pronounced as the number of factors increases as evidenced by the side-by-side comparison of the two bottom designs in Figure 1. Here you see experiments on three factors, in which case OFAT comes out far worse than the two-level factorial with 16 versus only 8 runs; respectively.

Factorial design offers two additional advantages over OFAT:

- It covers a broader area or volume of factor-space from which to draw inferences about your process.
- It reveals "interactions" of factors. Revelation of two-factor interactions (2fi's) often proves to be the key to understanding a process, especially one involving chemicals, such as coatings.

Table 1 shows how many runs are required for doing full two-level designs for up to eight factors. You must run full factorials for four or fewer factors if you want to resolve 2fi's. We advise that if you want to study only two factors that you replicate the design. However, if you need to screen five or more factors, only a fraction of all the combinations need be applied. Ideally resources will be available to achieve high resolution (see column in Table 1 labeled "High-Res") and thus detect 2fi's, but when runs come at a premium, choose a smaller, medium resolution design to screen for main-effects only.

Table 1. A Select Set of Two-Level Factorials

Factors (k)	# Runs Full	# Runs High-Res	# Runs Med-Res
2	4 (2^2)	8 (2x)	NA
3	8 (2^3)	NA	NA
4	16 (2^4)	NA	8 (½)
5	32 (2^5)	16 (½)	NA
6	64 (2^6)	32 (½)	16 (¼)
7	128 (2^7)	32 (¼)	16 ($^1/_8$)
8	256 (2^8)	32 ($^1/_8$)	16 ($^1/_{16}$)

Layouts for these and many other fractional designs, including far more factors, can be found in DOE textbooks (1), or better yet, with the aid of statistical software packages (2). You can choose two-level designs with as little as k+1 runs, where k equals the number of factors you want to test. For example you could test 7 factors in 8 runs, or 15 factors in 16 runs. However, these "saturated" designs provide very poor resolution: main effects will be confused with two-factor interactions. We advise that you avoid running such low resolution designs.

Be sure to randomize the run order of your entire design. Otherwise you leave yourself open to "lurking factors", such as ambient temperature and humidity changes, that could confound your factor estimates. After completing the experiments, standard statistical analyses provide significance tests on the overall outcome and individual effects. Textbooks provide hand-calculation schemes for doing analysis of two-level factorials, but it's much easier to let statistical software (2) do this work for you.

2.1 Case study – Screening factors thought to affect a spin coater

The following study comes from a short-course on DOE (3). It will be described only briefly to give you an idea of what's required by a two-level factorial design. A spin coater used to apply a photo resist to a silicon wafer can be controlled by four machine settings:

A. Speed (spin)
B. Acceleration (spin up)
C. Volume of resist
D. Time (spin)

These four factors are numerical because they can be adjusted to any values. However, for purposes of experimentation, they will be kept within ranges thought reasonable for keeping coating thickness in proximity to manufacturing specifications. Two more factors, both categorical, are thought to affect the process:

E. Vendor of resist (two candidate suppliers)
F. Cover for exhaust (on or off)

At this stage it's uncertain which, if any, of these factors will significantly affect thickness, so it will be advantageous to perform the 16-run (¼th fraction) medium-resolution design shown in Table 2.

Table 2. Spin-coater design of experiments

Std	A: Speed (rpm)	B: Accel.	C: Vol. (cc)	D: Time (sec)	E: Vendor	F: Cover	Thick (Avg)
1	6650	5	3	6	X	Off	4475
2	7350	5	3	6	Y	Off	4630
3	6650	20	3	6	Y	On	4334
4	7350	20	3	6	X	On	4534
5	6650	5	5	6	Y	On	4455
6	7350	5	5	6	X	On	4478
7	6650	20	5	6	X	Off	4222
8	7350	20	5	6	Y	Off	4523
9	6650	5	3	14	X	On	4440
10	7350	5	3	14	Y	On	4664
11	6650	20	3	14	Y	Off	4330
12	7350	20	3	14	X	Off	4515
13	6650	5	5	14	Y	Off	4566
14	7350	5	5	14	X	Off	4456
15	6650	20	5	14	X	On	4115
16	7350	20	5	14	Y	On	4467

The data shown for thickness is collected by performing the design in random run order. Analysis reveals that only four factors are statistically significant: A, B, C and E. With the aid of an eight-run follow-up study, it is further revealed that an interaction exists between factors C and E as shown in Figure 2 below.

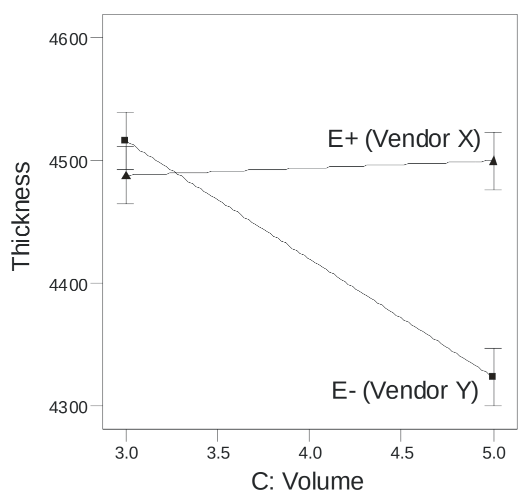

Figure 2: Plot of two-factor interaction for spin-coater

Notice how the effect of changing volume of resist (factor C) depends on which vendor supplies the material (factor E). The bars at either end of each plotted line indicated the least significant difference (LSD) at the 95 percent confidence level. The overlap of these LSD bars at low level of volume (3.0) indicates that it may be possible to run the spin-coater in a way that will be robust to which vendor supplies the resist, thus freeing up the purchaser to pick whichever may be cheapest at any given time. Insights like this can never be revealed by OFAT experimentation. Only by running combinations of factors at two-levels do you learn about interactions.

3.0 OPTIMIZATION VIA RESPONSE SURFACE METHODS (RSM)

If you believe your experiments are near the optimum, consider putting a “center point” in your design. This point represents one set of conditions at the middle of all factor levels (or component ranges in a mixture). For example, in the case study presented above the center point of factors B, C and D is (12.5, 4,10). To get a reasonable estimate of curvature, repeat the center point several times mixed in randomly with the remaining design points. For example, four center points (all at identical conditions!) could be added to the design in Table 2, increasing the number of runs from 16 to 20. If you’re close to a peak of performance, you will see a significantly higher than expected response from the center points, thus revealing curvature (non-linearity) in your response. Then you will need to run additional factor levels and employ response surface methods (RSM).

The central composite design (CCD) is a good choice for response surface experiments. It’s composed of a core two-level factorial surrounded by axial points. Figure 3 shows the layout of points for a CCD on two process factors.

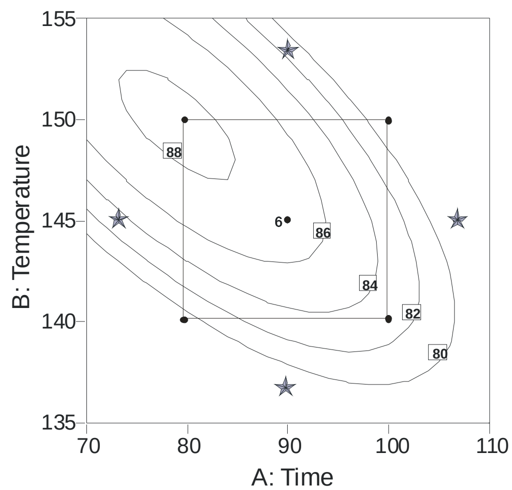

Figure 3: Contour plot showing points for central composite design

The axial points are symbolized by the stars. Any predictions outside of the two-level factorial square, including the star-point locations, requires extrapolation, which may be dangerous.

This design included 6 center points (time 90, temperature 145), half of which were done with the original factorial and the others included in a second block of star points.

Textbooks (4) provide details on CCDs and a variety of other designs for response surface methods. Statistical software supporting RSM is an absolute necessity for fitting the mathematical models (done via regression), validating them via statistical analysis, and generating the response surface graphs.

2.1 Mixture designs for optimal formulation

For coatings experiments that focus only on formulation, we recommend the use of designs geared for mixtures. This involves use of triangular coordinates set up to maintain a total of 100 percent for all ingredients. Figure 4 shows a 3D view of a response surface generated from a mixture design on an automotive coating with three ingredients.

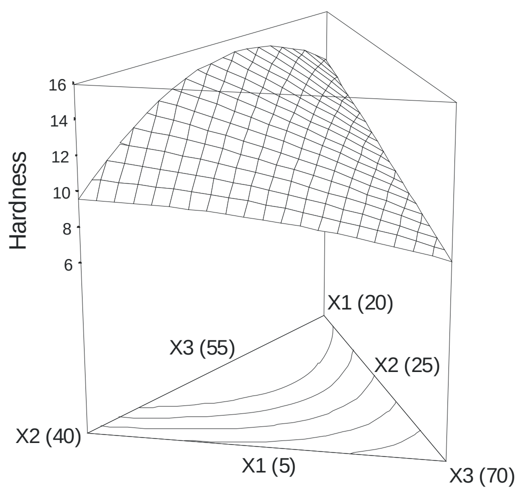

Figure 4: 3D response surface from a mixture design

Details on the methodology can be found in textbooks (5) and case-study articles on coatings experiments (6, 7).

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