Achieving Six Sigma Objectives for Variability Reduction in Formulation and Processing

Apply powerful design of experiments (DOE) tools to make your system more robust to variations in component levels and processing factors.

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"Six sigma" is the new rallying cry for quality improvement in the process industry. For example, Dow aims to generate an extra $1.5 billion per year in profits after training 50,000 of their employees on the methods of six sigma.[3] Statistical tools play a key role in achieving savings of this magnitude. In fact, "sigma" is a Greek letter that statisticians use as a symbol for standard deviation – a measure of variability. If a manufacturer achieves a six sigma buffer from its nearest specification, they will experience only 3.4 off-grades per million lots. This translates to better than 99.99966% of product being in specification. To illustrate what this level of performance entails, imagine playing 100 rounds of golf a year with two putts per hole being the norm (par): At six-sigma you'd make a three-putt (bogey) only every 163 years![4] Even Tiger Woods would be envious of this level of quality.

Of all the statistical tools employed within six-sigma, design of experiments (DOE) offers the most power for making breakthroughs. Via an inspirational case-study, this article demonstrates how DOE can be applied to development of a formulation and its manufacture to achieve optimal performance with minimum variability, thus meeting the objectives of six sigma programs. Armed with knowledge gained from this article and the example as a template, chemists and engineers from any of the process industries (pharmaceutical, food, chemical, etc.) can apply these same methods to their systems and accomplish similar breakthrough improvements.

Minimizing Propagation of Error (POE) from Varying Inputs

After earning his PhD in chemistry and taking a job at a chemical company, a colleague of ours got assigned to an operator for an orientation to the real-world of production. As the operator watched with much amusement and disgust, the chemist carefully weighed out materials with a small scoop. The operator pushed the PhD chemist aside, grabbed a sack of chemicals and tossed it into the reactor. "You're not in the laboratory anymore," he said, "This is how we do things in manufacturing."

Hopefully the operators of your formulation process will be more exacting when adding ingredients. However, at the very least, you can expect some variation due to inherent limitations in equipment. How will these variations affect product quality and process efficiency? Can you do anything to make your system more robust to variations in component levels and process settings? The answers to these very important questions can be supplied via an advanced form of DOE called "response surface methods" (RSM). This statistical tool produces maps of product and product performance, similar to topographical displays of elevation, as a function of the input variables that you (or your clients) control. The objective of six sigma is to "find the flats" – the high plateaus of product quality and process efficiency that do not get affected much by variations in component levels or factor settings. You can find these desirable operating regions visually, by looking over the 3D renderings of response surfaces, or more precisely via a mathematical procedure called "propagation of error" (POE).

To see how POE works, let's look at a very simple response surface (Figure 1) generated by changing only one control factor X1.

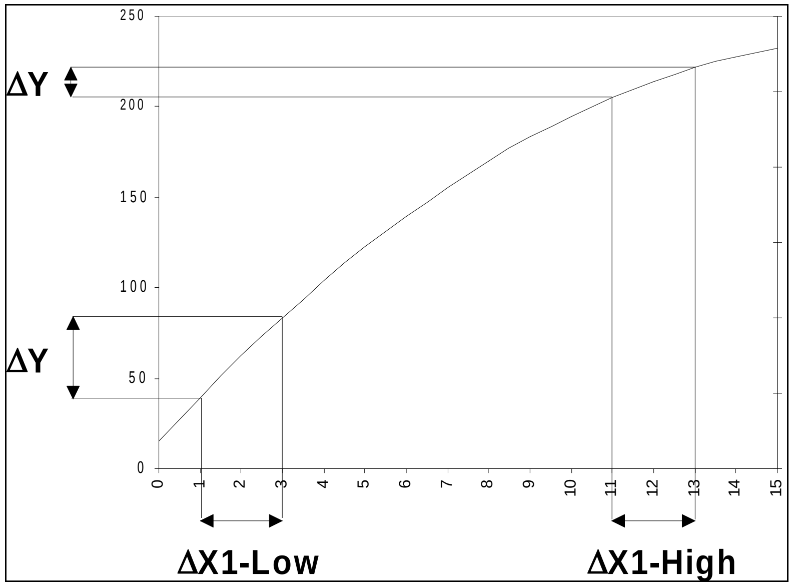

Figure 1/ Propagation of error transmitted from control factor X1 (non-linear response)

Assume that this factor exhibits a constant variation shown on the graph as a difference with magnitude delta (Δ). This variation, or error, will be transmitted to the measured response to differing degrees depending on the shape of the curve at any particular setting. In this example, because the curve flattens out as the control factor increases, a setting at the higher level causes less propagation of error (POE). Therefore, you see a narrower difference (ΔY) on the response axis as a result of setting the factor at the higher, rather than lower level.

With the aid of some calculus, the POE itself can be graphed as a continuous function. In this case the original response surface can be described by the following quadratic equation:

$$\hat{Y} = 15 + 25x_1 - 0.7x_1^2$$

We will spare you the details, but after taking the partial derivative of this function with respect to the input (X1) and taking the square root, the following equation for standard deviation (σ) is produced:

$$\sigma_Y = \sqrt{(25 - 1.4x_1)^2 \sigma_x^2 + \sigma_{Residual}^2}$$

Assume for now that the standard deviation of the control factor X1 equals one ($\sigma_x = 1$) and there are no other sources of variance ($\sigma_{Residual} = 0$). We've now obtained the information needed for graphing the standard deviation of the response (σ_y) transmitted from the variation in the input factor (X), in other words, the POE (see Figure 2). In this case the POE decreases in direct proportion to X1 as it increases.

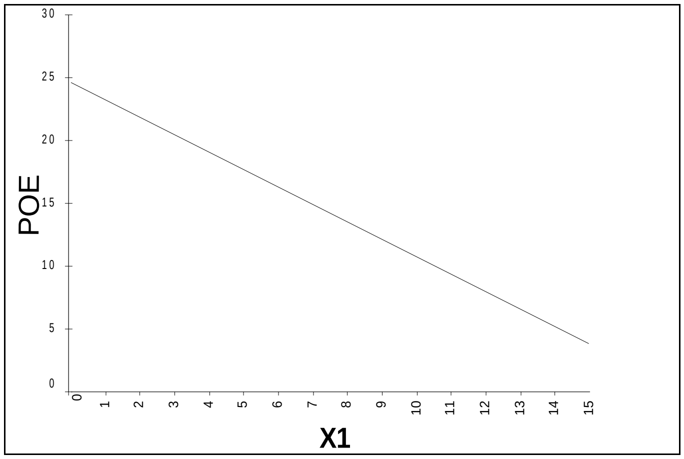

Figure 2/ Response surface graph of POE transmitted from control factor X1

Obviously, reducing response variation is a highly desirable outcome. But, what if the absolute level of response gets shifted out of specification? Ideally you will find another control factor that affects response in a <u>linear</u> fashion, such as that shown in Figure 3. (We added the bell-shaped curves to indicate that variation will likely be distributed according to a normal distribution.)

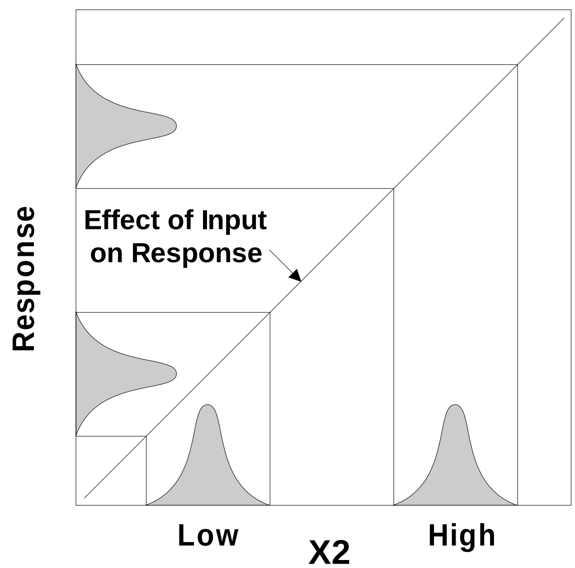

Figure 3/ Propagation of error transmitted from control factor X2 (linear response)

This factor (X2) creates no effect on POE (it's constant), so it can be used to get your response back into specification after changing the other factor (X1) to reduce variability. In this case X2 must be decreased to counteract the increase in response caused by increasing X1 (done to reduce POE). Ideally, as in this simplified example, you can achieve the 'best of both worlds' as shown in Figure 4: On-target response with minimal variability, hopefully to the level demanded by six sigma.

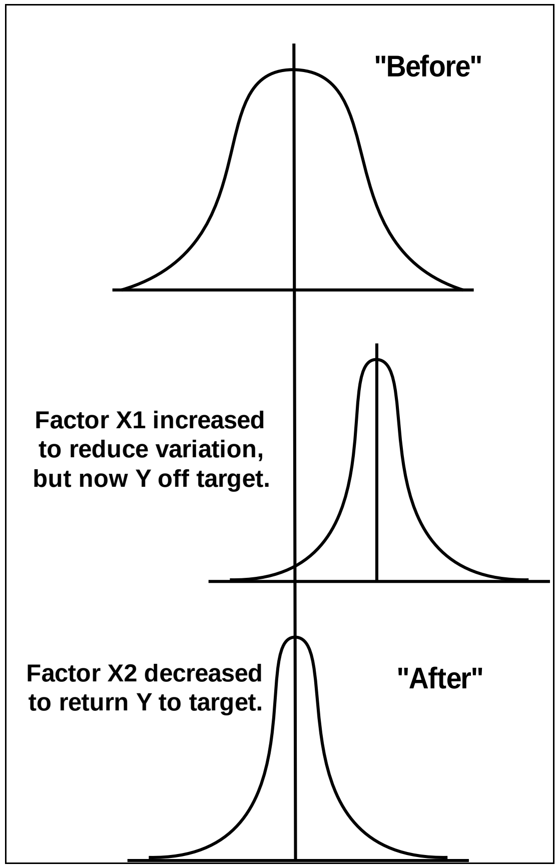

Figure 4/ Achieving variability reduction by intelligent manipulation of factor levels

Let's see how these concepts can be applied to a six sigma project on a coatings system. We chose this example because anyone can identify with problems of paint peeling (who hasn't had this happen at home?). However, we expect chemists and engineers in other process industries (pharmaceutical, food, chemical, etc.) to use this as a template for improving their particular formulation and manufacturing procedures.

A Case Study on POE

The Therabath® paraffin therapy bath[5] (pictured below) is a durable medical device that holds one gallon of molten paraffin wax. Sufferers of osteoarthritis use it for physical therapy. They dip their hands repeatedly in the heated bath, which helps loosen stiff joints. The wax then slowly solidifies as a glove, producing further therapeutic benefits via the heat of fusion. Oils reduce the overall melt point to a comfortable level, facilitate removal of the glove and provide moisturizing for skin.

The tank holding the molten wax is made out of stamped steel. It's then powder-coated electrostatically with an epoxy-based paint. The coating must withstand temperatures approaching 130 degrees F. and exposure to salt-water that collects from the sweat of the Therabath users.

Figure 5/ Therabath paraffin therapy bath

The units are sold with a life-time guarantee. Very few units get returned, but in those that do, two types of off-grade predominate:

- Blisters that appear spontaneously after some time in use
- Scratches that become noticeably corroded.

It's believed that if adhesion can be maintained at a level above 200, with hardness set at 140, the problems noted above will recede to a level of quality consistent with six sigma. (For reasons of propriety, we do not show units of measure of these responses.)

Designing An Experiment That Combines Mixture with Process Variables

Our design of experiments combined variations in mixture components and process factors.[1] Table 1 shows the names and ranges of the input variables, as well as their expected variation in terms of standard deviation, which will come into play when calculating POE.

Table 1/ Input variables for powder-coating study

Label	Description	Units	Type	Low	High	Std. Dev.
A (X1)	Epoxy	Weight %	Mixture	35	45	0.3
B (X2)	Calcium	Weight %	Mixture	20	40	0.1
C (X3)	Titanium	Weight %	Mixture	25	35	0.1
D (Z1)	Bake temp.	Deg. F.	Process	320	400	4
E (Z2)	Bake time	Minutes	Process	15	35	1

The mixture variables (A, B and C) added to a constant of 90 weight percent of the powder. The other 10 percent was made up of bisphenol A, aluminum oxide and silica, all held in constant proportion. The paint chemists expected the three main powder-coating components to interact in complex ways. To properly model such behavior, we wanted to formulate a sufficient number of unique blends to fit a "special cubic" mixture model:

$$Y(x) = \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{23} X_2 X_3 + \beta_{123} X_1 X_2 X_3$$

The process factors of bake time and temperature (D and E) also were expected to interact with each other and possibly create non-linear responses. For this reason, we decided to collect sufficient data for fitting a "quadratic" process model:

$$Y(Z) = \alpha_0 + \alpha_1 Z_1 + \alpha_2 Z_2 + \alpha_{12} Z_1 Z_2 + \alpha_{11} Z_1^2 + \alpha_{22} Z_2^2$$

Models like this are typically used to develop response surface graphs for process optimization.

These two models were crossed to account for possible interactions between mixture and process. For example, the ideal coating formulation may differ depending on the choice of bake time and temperature, which could be a function of the particular type oven available for processing the tanks. The crossed model contains 42 terms (7 from the special cubic mixture model times 6 from the quadratic process model). With the aid of computer software,[6] a d-optimal design[2] was generated to provide the ideal combinations of variables for fitting response data to the combined mixture-process model. We added 5 extra unique points for estimating lack-of-fit for the model, plus 5 replicates of existing points to estimate pure error. We also incorporated one center point (combination #3 on the list in the appendix), representing the standard operating conditions, for a grand total of 53 combinations.

The results (simulated) of this experiment can be seen in the Appendix. The combinations are organized for easier viewing, with replicate points lumped together. However, experiments like this should always be performed as randomly as possible to offset any lurking variables, such as ambient humidity.

Generating 3D Surfaces to See Impact of Inputs on Responses and POE

Without computer software to do the number-crunching, it would be incredibly difficult to fit all the coefficients in the mixture-process crossed models. It's very cumbersome to even sort through all the statistics generated by such software. We advise that you make use of computerized tools for reducing models to their bare essentials. Make sure that the results pass your pre-set thresholds for statistical significance (typically 95 percent confidence) and that you don't violate any of the standard assumptions for normality (diagnostic plots help you check this). In this case, the statistical analysis produced highly-significant models that met all assumptions. To assess the outcome of an experiment like this, one picture is worth a thousand numbers, so let's not get bogged down in the statistics. Instead, take a look at the response surfaces for the mixture variables (Figure 6a and 6b) and the process factors (Figure 7a and 7b) for adhesion and hardness.

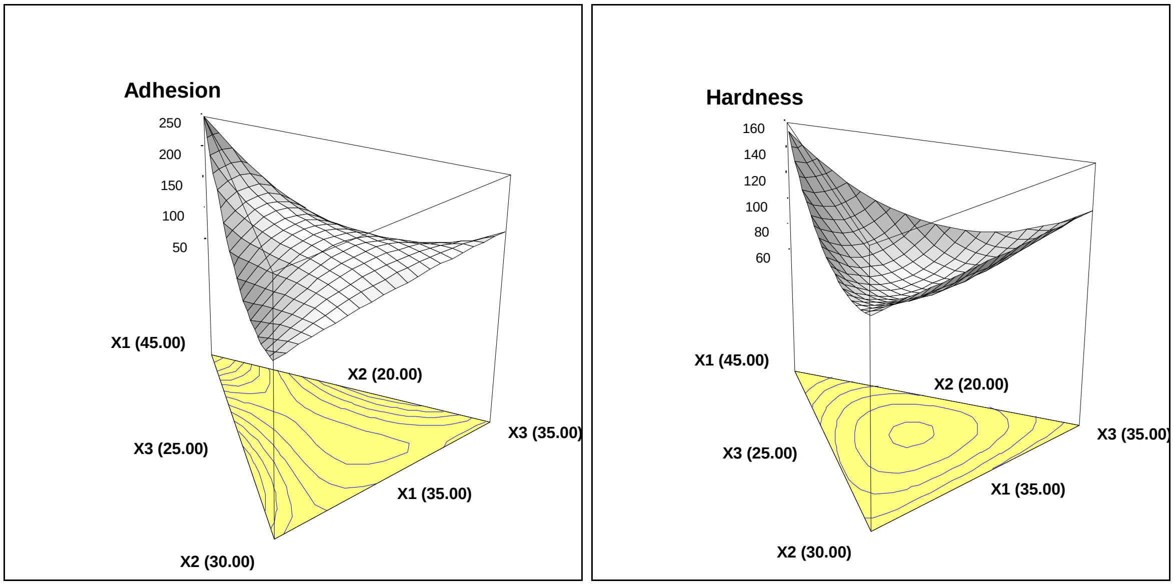

Figure 6a,b/ Effect of powder-coating components on adhesion (left) and hardness (right) with bake time and temperature at mid-levels (centerpoint)

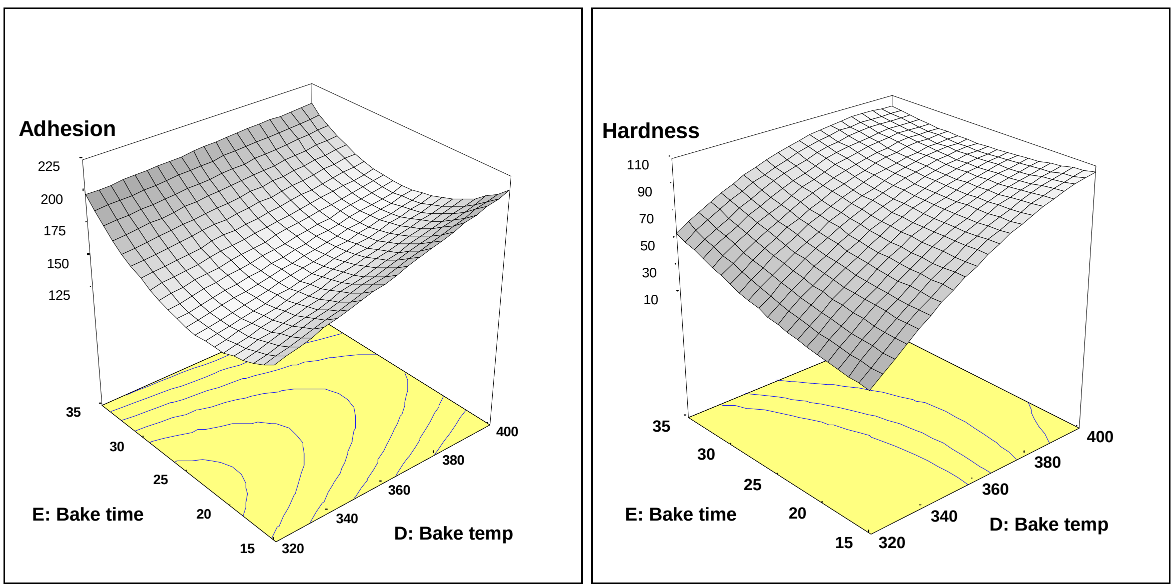

Figure 7a,b/ Effect of process factors on adhesion (left) and hardness (right) with the powder-coating formulated at mid-levels (centroid)

Notice on Figure 7b how the hardness response drops precipitously at the low ends of bake time and temperature. Even assuming you'd accept such a low hardness, this would be a very bad place to set up your process, because results would be very sensitive to variations in the input factors. This can be seen more clearly in the computer-generated graph of POE shown in Figure 8.

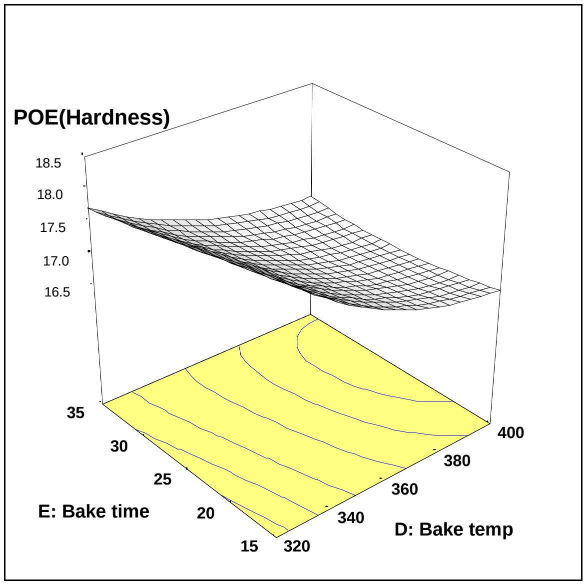

Figure 8/ Effect of process factors on POE of hardness

The highest POE (most variation in response) occurs at the low ends of bake time and temperature. However, before worrying about POE, the system must be set up in a way that meets the basic specifications. Then, assuming that there's optional combinations of input variables that accomplish this mission, pick the one that creates the least POE. This requires use of computer software that can do multiple response optimization.

Finding a Setup that Meets All Specifications with Minimum POE

As noted earlier, the goals for the Therabath powder-coating properties are an adhesion of at least 200 and a hardness of 140, achieved on a consistent basis by minimizing POE. Also, to minimize costs of the powder-coat, the more of the cheapest ingredient, calcium, the better. Furthermore, it would be nice for bake time to be minimized, thus increasing throughput. Obviously, to accomplish all this, some tradeoffs must be made. Therefore, it's necessary to allow for some ranges on all of these goals. Table 2 shows what the Therabath engineers are willing to accept.

Table 2/ Goals for powder-coating study

Response	Goal	Lower	Upper
Adhesion	is in range	200	500
POE(Adhesion)	is minimum	15	30
Hardness	is target → 140	100	200
POE(Hardness)	is minimum	15	25

The input variables aren't listed above, but they do get constrained within their previously specified ranges. Never extrapolate outside of your experimental region because the predictive models probably won't work out there. Stay in bounds or possibly face some dangerous consequences!

Based on the goals and ranges, the software set up desirability scales on all responses and then searched for a solution that maximized overall desirability. (For an enlightening discussion on how desirability gets calculated and applied, see reference 7, reprints of which can be obtained by contacting the authors.) The optimal combination found by the software can be seen in Table 3.

Table 3/ Optimal combinations for mixing and baking powder-coat

Variable	Optimum
A. Epoxy	42.25 wt. %
B. Calcium	20.88 wt. %
C. Titanium	26.87 wt. %
D. Bake temp.	400 deg. F
E. Bake time	26.85 min.
Adhesion	201.1
POE(Adhesion)	18.9
Hardness	140
POE(Hardness)	16.7

Two "sweet spots" can be seen in the process space (bake temperature versus bake time) by overlaying contour plots for hardness and adhesion, with unacceptable regions shaded out (see Figure 9). (The formulation of the powder-coat is set at the optimal component levels shown above.)

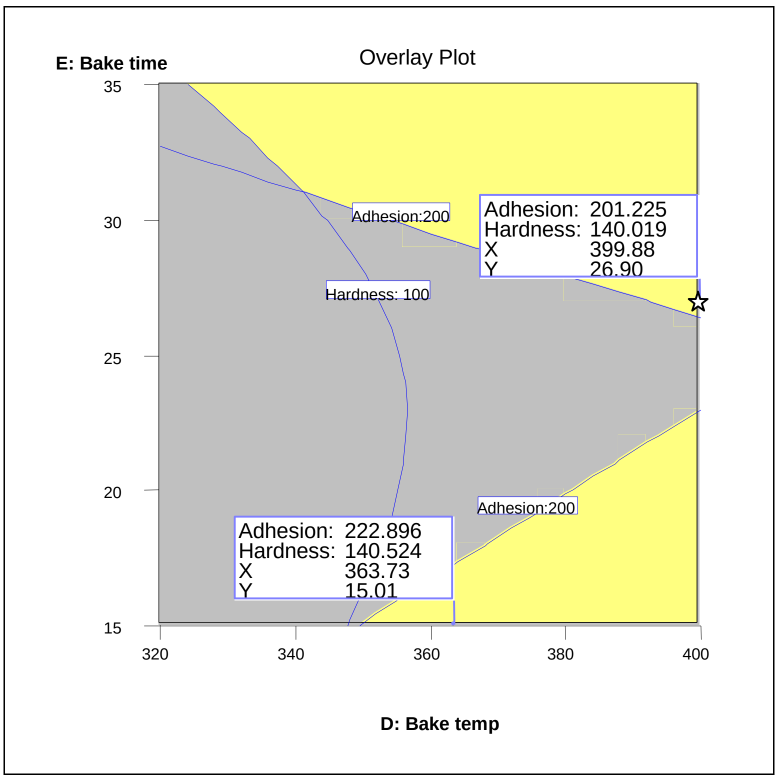

Figure 9/ Sweet spots for processing powder coat made by optimal formulation

In this view of the multivariable experimental space, adhesion appears to be more of a limiting response than hardness (it forms most of the boundary around the operating windows at top and bottom). It's tempting to consider reducing the bake time from the recommended level of approximately 26.9 (flag set at starred point in the upper window) to it's minimum tested value of 15 minutes (see flag in lower window). However, as you can see in Figure 10, the adhesion is the least in the mid-range of bake time (the trough on the response surface).

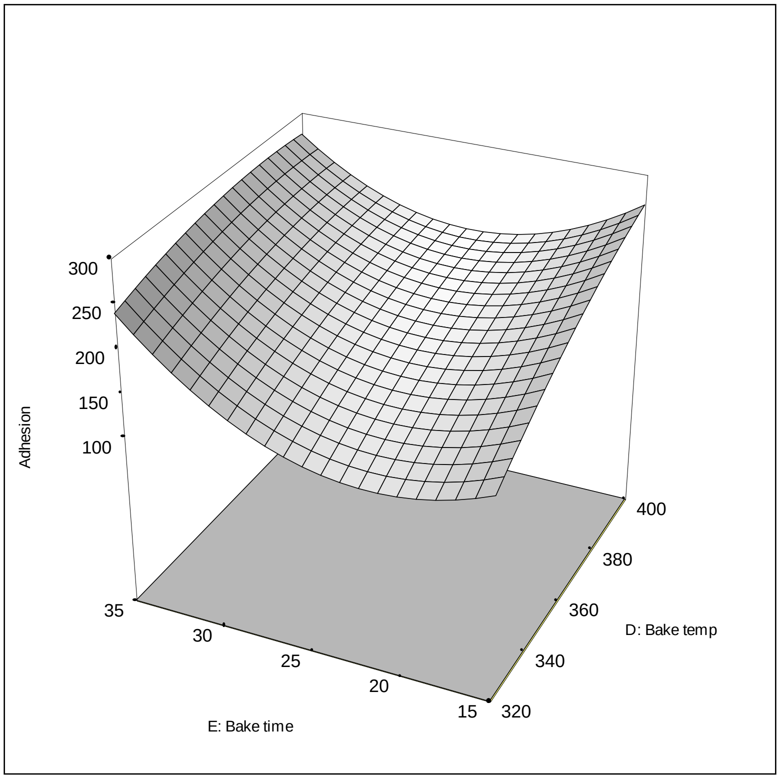

Figure 10/ Response surface for adhesion as a function of bake time and temperature with optimal formulation of powder-coat

At the values of 15 minutes for bake time at 364 degrees (approximately what's shown in the lower flag in Figure 9) the POE of adhesion exceeds 23 units of standard deviation, a substantial increase from the 18.9 result reported in Table 3 for the optimum setup. In companies that place an emphasis on six sigma goals this may be an unacceptable trade-off. However, if time is of the essence, it's easy enough to incorporate this in the optimization criteria – simply add a goal to minimize it. That's what makes the whole DOE procedure outlined in this article so incredibly powerful: It produces statistically-valid predictive models that can be easily manipulated for "what-if" analysis. Then when goals shift, or get weighted differently, it's just a matter of plug-and-chug and the answer magically appears. To ensure that it's correct, confirmation runs must be performed. Hopefully, the results will agree well with what's predicted to happen.

Conclusion

As a result of doing systematic experimentation, using sound statistical principles, the quality of the powder-coating of Therabath tanks can be improved and became more robust to variations in the levels of components and processing factors. With the proper knowledge and software tools, chemists and engineers from any of the process industries (pharmaceutical, food, chemical, etc.) can apply these same methods to their systems and accomplish similar breakthrough improvements.

For more information on design of experiments, contact Stat-Ease, Inc., 2021 East Hennepin Ave., Suite 480, Minneapolis, MN 55413; phone 612.378.9449; fax: 612.378.2152; e-mail: mark@statease.com.

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Appendix: Data for powder-coating study

Comb.	A: Epoxy wt %	B: Ca wt %	C: Ti wt %	D: Bake temp deg F	E: Bake time min	Adhesion	Hardness
1	40	20	30	360	25	112.1	115
2	35	30	25	360	25	116.4	103
3	38.3	23.3	28.3	360	25	156.0	72
4	45	20	25	360	25	245.0	167
5	35	20	35	320	15	169.0	140
6	45	20	25	320	15	174.8	19
7	35	30	25	320	15	126.8	130
8	35	20	35	320	35	87.2	57
9	45	20	25	400	15	339.3	294
10	35	30	25	400	35	146.6	119
11	35	20	35	400	15	213.0	143
12	45	20	25	320	35	327.0	142
13	35	30	25	400	15	102.2	52
14	35	20	35	400	35	146.1	153
15	45	20	25	400	35	356.9	241
16	35	30	25	320	35	144.2	123
17	35	20	35	320	25	152.9	92
18	35	20	35	360	30	195.7	127
19	45	20	25	360	35	392.5	226
20	35	25	30	320	15	164.6	102
21	40	25	25	320	15	101.0, 75.2	11, 17
22	40	20	30	320	15	106.9	19
23	35	25	30	320	35	183.9	109
24	40	25	25	320	35	149.7, 139.7	89, 78
25	40	20	30	400	15	208.7	163
26	35	25	30	400	15	150.1	130
27	40	25	25	400	15	156.1	91
28	40	20	30	320	35	189.8	93
29	35	25	30	400	35	137.9	109
30	40	25	25	400	35	147.0, 171.9	126, 152
31	40	20	30	400	35	219.1	152
32	35	25	30	320	25	146.7	104
33	40	25	25	320	25	73.3	47
34	35	25	30	360	30	117.6, 150.9	107, 136
35	40	25	25	360	30	151.6, 135.6	139, 93
36	40	20	30	360	35	183.7	124
37	38.3	23.3	28.3	320	15	146.7	16
38	38.3	23.3	28.3	400	15	230.8	120
39	38.3	23.3	28.3	320	35	178.9	33
40	38.3	23.3	28.3	400	35	223.4	120
41	38.3	23.3	28.3	320	25	159.4	43
42	38.3	23.3	28.3	360	15	193.2	68
43	35	30	25	360	15	131.9	73
44	36.7	21.7	31.7	400	25	169.0	98
45	45	20	25	400	25	250.3	173
46	35	30	25	400	25	102.9	108
47	35	20	35	360	20	147.8	154
48	35	30	25	360	35	143.0	110