Computer-aided Design of Experiments for Formulations

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Powerful desk-top software tools now make it easy for you to optimize paint formulations. Aided by the computer, you can apply statistically based design of experiments (DOE) for mixtures - a proven method for making breakthrough improvements in cost and performance. Ultimately you may discover a sweet spot where all your customer specifications can be satisfied. To illustrate the method, we will show you a case study that involves formulation of rheology modifiers.

Explore the feasible region

The experimenters explored the impact of three rheology modifiers for architectural coatings[1]: RM74, RM-77 and RM-75 (labeled respectively as components A or X_1, B or X_2 and C or X_3). They measured two responses: ICI viscosity and Leneta flow. Within the additive package, each of the three components varied from 0 to 100 per cent, or 0 to 1 on a proportional scale. The resulting experimental region forms a geometry call a simplex, which is defined as a shape with one more vertex then the number of dimensions. In two dimensions, a simplex is a triangle. In three dimensions, a simplex is a tetrahedron, and so on.

Figure 1 shows the experimental region for the rheology study. The corners of the triangle represent purest blends. Note that these extreme vertices display points representing actual blends. In fact, each of these points were done twice so the pure error could be measured. Additional experiments were laid out according to a specific design called a simplex centroid, which you can get from textbooks[2] or software[3]. The design includes binary blends, and the three-part blend. The formulators augmented the design to include the midpoints between the centroid and the corners of the triangular region. These additional design points are called check blends because they fill gaps that you would like to investigate for unusual behavior.

Not all formulations fit such a neat simplex space. You may impose complex constraints that block out infeasible regions. In these cases computer software can provide optimal designs that will meet your needs for exploring the relationships.

The results from the rheology experiment can be seen in Table 1. The actual run order should always be randomized to insure against lurking factors such as material degradation, machine wear and drifting ambient conditions. Measure as many responses as you like. The results must be quantitative, but even a subjective rating scale as crude as 1 through 5 may be effective.

Powerful predictive models give you a map of performance

With the aid of software, the data from rheology response data were fitted to polynomial models geared for mixtures:

(1) ICI Viscosity = 0.77A + 4.21B + 0.43C - 4.33AB + 0.31AC -5.61BC

(2) Leneta Flow = 7.97A + 4.57B + 9.37C

Statistical analysis indicates with a high degree of confidence, more than 99.99 per cent, that these models are significant. Therefore, although the equations only approximate the true relationships, they will be more than adequate for empirical predictions.

The model for ICI viscosity is a quadratic polynomial. It contains second-order terms which capture interactions. The relatively large coefficients on AB and BC indicate significant antagonism between these components. In other words the combination produces lower viscosity than would be expected based on either of the parent components.

The model for Leneta flow is linear. The second order terms did not improve the fit, so to keep things simple, they were dropped.

Based on these models, graphics programs can produce extremely useful maps. Figures 2 and 3 show 3D surfaces, with projected contour maps, for the rheology responses.

Multiple response optimization finds the "sweet spot"

By shading out areas outside of specification, then overlaying the contour plots for all responses, you often discover a window or "sweet spot" that meets all customer needs. Assume that you need to meet the following rheology specifications:

(1) ICI viscosity: 0.5 to 0.7

(2) Leneta flow: 7.5 to 8.0

Figure 4 shows that these specifications can be met by formulating the three rheology modifiers within specific limits. With the aid of software, you can set a flag at good location and predict what will happen. Many software packages provide numerical search algorithms that will find the most desirable mixture. Typically, you will take cost into account before making your final decision.

What's in it for you

The case study on rheology modifiers illustrates how mixture design can be applied to a coatings application. The design of experiments uncovered a sweet spot where all specifications were satisfied. If you equip yourself with the tools of computer-aided statistical DOE, you will be in a position to make the most of opportunities such as those presented at the outset of this article. Your reputation will be enhanced and the competitive position of your company advanced.

Sources

(1) Acrysol• brand, Rohm and Haas, Philadelphia.

(2) Myers, R.H., Montgomery, D.C., *Response Surface Methodology*, John Wiley & Sons, Inc, New York, 1995.

(3) Helseth, T.J., et al, *Design-Expert*, Windows, Stat-Ease, Inc, Minneapolis, 1996 ($995).

Id	Type	Factor A:RM-74	Factor B:RM-77	Factor C:RM-75	Response ICI Viscosity Poise	Response Leneta Flow ASTM units
1a	Vertex	1.00	0.00	0.00	0.76	8
1b	Vertex	1.00	0.00	0.00	0.75	8
2	CentEdge	0.50	0.50	0.00	1.4	7
3	CentEdge	0.50	0.00	0.50	0.55	8
4a	Vertex	0.00	1.00	0.00	4.1	4
4b	Vertex	0.00	1.00	0.00	4.4	4
5	CentEdge	0.00	0.50	0.50	0.9	7
6a	Vertex	0.00	0.00	1.00	0.42	9
6b	Vertex	0.00	0.00	1.00	0.4	10
7	Check	0.67	0.17	0.17	0.8	7
8	Check	0.17	0.67	0.17	1.7	7
9	Check	0.17	0.17	0.67	0.55	8
10	Centroidr	0.33	0.33	0.33	0.8	8

Table 1. Factors and Levels for Rheology Experiment

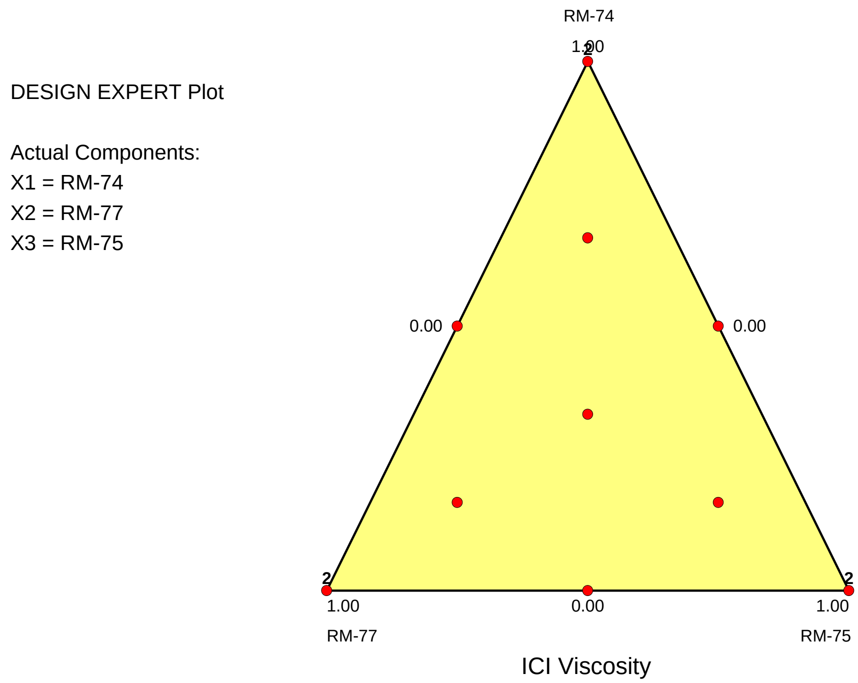

Figure 1. Simplex Centroid Design, Augmented with Check Blends

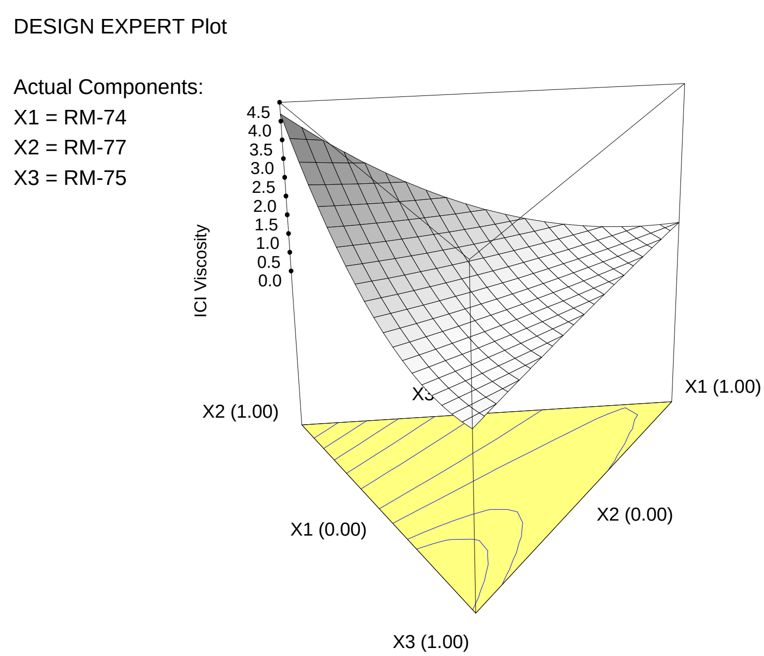

Figure 2. Response Surface Map of ICI Viscosity

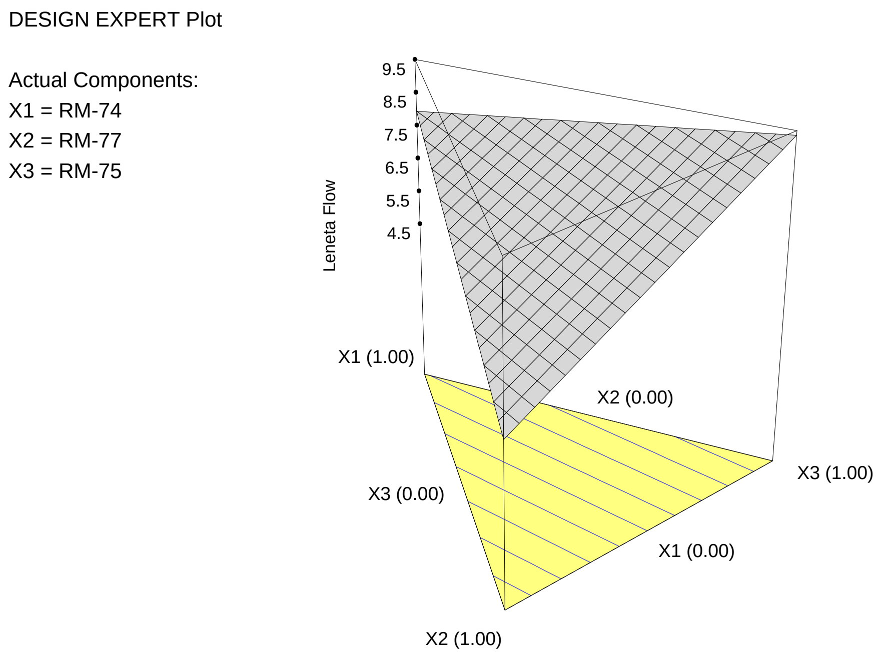

Figure 3. Response Surface Map of Leneta Flow

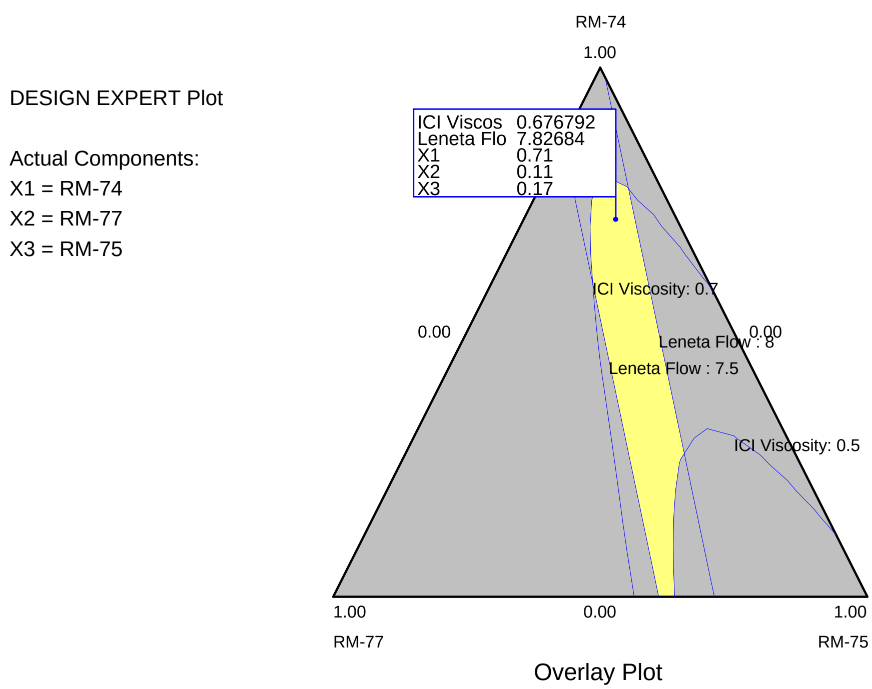

Figure 4. Graphical Optimization