Design of Experiments Helps Perfect Performance of New Gel Coat Product

Interplastic Corporation is a leading producer of gel coat, a material used to provide a high-quality finish on the visible surface of a fiber-reinforced composite material. The company recently developed a new gel coat formulation, but testing showed problems with cratering, the formation of holes in the surface of the coating. Gel coat is made of an intricate blend of different ingredients that have complex interactions with each other. Scott Crump, technical director for Interplastic, addressed this challenge by developing an experimental strategy to optimize the proportion of each ingredient of the formulation with respect to cratering and other key quality parameters. He used the design of experiments (DOE) method to explore the design space within predetermined limits to identify the optimal proportion for each ingredient as well as the robustness of candidate designs with respect to small changes in the proportion of ingredients. The optimized version of the product eliminates the cratering problem and provides consistently excellent quality.

Interplastic is a specialty chemical company with headquarters in St. Paul, Minnesota. Its Thermoset Resins Division focuses on the production and distribution of unsaturated polyester and vinyl ester resins, gel coats, and colorants for the composites and cast polymer industries. Gel coats are widely used in fiberglass composite structures such as recreational vehicles, boats, architectural panels, and other products that require excellent exterior durability. Companies involved in higher volume production typically use robotic spray equipment to evenly apply gel coat into complex contours.

In the open mold process, a wax-like release agent is applied to a glossy, low surface energy mold. The gel coat is spread on the mold in a liquid state at a typical thickness of 20 mils. Then a laminate consisting of fiber reinforcement and resin is applied to the coating and the part is removed from the mold. The gel coat bonds to the laminate structure and is removed from the mold with the part. Many gel coats are based on epoxy or unsaturated polyester resin chemistry. Gel coats typically consist of four classes of ingredients -- polymer, additives, filler/pigments, and reactive diluents. The complexity of formulating gel coats is increased by the fact that there are literally hundreds of different ingredients available to adjust coating performance and these ingredients can be combined in an infinite number of proportions.
Example of gel coat with a cratered surface.

Example of gel coat with various levels of dimpling.
Craters are typically formed because the gel coat lacks the proper surface tension balance with the result that the coating is repelled by certain areas of the mold. The effect is similar to the way that raindrops are repelled from the hood of a waxed car. A challenge of the product development process is that quality variables such as the propensity of the coating to crater are highly dependent on each other. Examples include a dimpling pattern that looks somewhat like the surface of an orange peel and sagging where the coating does not properly adhere to the mold and instead runs down the wall of the mold. Therefore if you increase the proportion of one ingredient to solve a problem, you are very likely to cause another problem.

“Well designed experiments provide a systematic process of experimentation and clarity in the interpretation of results which are often lacking in experiments performed using a random approach,” Crump said. The process of carrying out experiments based on a series of induction/deduction cycles uses the information gained from previous experiment cycles to optimize the responses in the current test cycle. This process of progressively moving toward an experimental objective by the use of relatively small and sequentially executed experiment cycles is the traditional approach used in specialty chemical product development.

“Progressive optimization techniques can be used to develop high quality coatings,” Crump said. “But this approach is too slow, particularly when we are faced with the challenge of developing a new product quickly to meet an urgent market need.” For this reason, Interplastic uses the DOE method to improve gel coat performance. DOE drastically reduces the number of runs required to determine the optimum value of each factor by varying the values of all factors in parallel. This approach determines not just the main effects of each factor, but also the interactions between the factors. DOE makes
it possible to identify the optimal values for all factors in combination. It also requires far fewer experimental iterations than the progressive optimization approach.

Crump used Design-Expert® software from Stat-Ease, Inc., Minneapolis, Minnesota, to design an experiment to optimize the gel coat formulation. “There are several very good general purpose statistical software packages with broad functionality that includes DOE. However, Design-Expert is the only package that I am familiar with that focuses exclusively on DOE,” stated Crump. Designed for use by subject matter experts rather than statisticians, the software walks the user through the process of designing and running the experiment and evaluating the results. “It provides excellent graphics and optimization capabilities,” said Crump. “Stat-Ease also does a great job of supporting their software. If you call in with a problem, they will connect you with someone who not only knows the software but is also a DOE expert.”

Crump chose the mixture design DOE method because it reduced the number of experiments required to develop specialty chemical formulations. The proportion of each ingredient in a mixture must add up to 100%. This reduces the number of degrees of freedom, which in turn substantially reduces the number of runs required to achieve a given level of statistical significance. Crump selected the D-optimal type of mixture design because it provided the minimal number of blends to fit a given predictive model.

Crump selected a design that is appropriate for modeling three factors at four or five different levels each. Design-Expert set-up a 31-run experiment with 28 unique blends and three replicates that fully explored the design space. The use of a mixture design experiment reduced the number of runs required to achieve the desired level of accuracy by 50% compared to a factorial design. The replicated runs were used to quantify the noise in the results. Chemists prepared blends for each of the runs based on the concentrations of each ingredient selected by the software.
Experts rated each panel used in the experiment.

Chemists sprayed each gel coat formulation onto a panel with properties that match a typical mold. Experts examined and rated each panel with respect to the major gel coat quality issues of cratering, dimpling and sagging. All three issues were evaluated simultaneously to avoid the possibility of selecting a blend that would fix the cratering problem, but create another problem. Crump created a desirability function that weighted the response variables to produce a single number representing the overall quality of the formulation.
A response surface map was used to visualize the desirability function as a function of the three factors. The areas of highest quality on the map are indicated by red. Crump selected a mixture in the center of the large red area near the center of the map because it provides high quality results and is also insensitive to small variations in the proportions of ingredients. Chemists ran confirmatory experiments at the optimal value and the results closely matched the DOE prediction. “Manufacturing startup went very well because we had mapped out the formulation space and selected a very robust formulation with no steep cliffs,” Crump said. “Mixture DOE helped us to achieve an exceptionally high level of quality with this product. The product has been very well received by our customers and is a resounding success in the market.”

For more information, contact:

Ph: 651.481.6860, (Toll-free) 800.736.5497, direct dial Scott Crump at 651.481.6876,
Fax: 651.481.9836, Web site: http://www.interplastic.com/

Ph: 612.378.9449, Fax: 612.746.2069, E-mail: info@statease.com,
Web site: http://www.statease.com