A panel is shown after undergoing exposure to 504 h of neutral-salt-spray (NSS) testing. Henkel was approached by an agricultural equipment manufacturer to help optimize a tough coating application.

Determining the optimum conditions for applying coatings suitable for tough applications such as agricultural vehicles is a challenge. The conventional method is to perform a series of experiments while varying one factor at a time. This approach is usually capable of finding a single set of conditions that will provide the desired result. However, there is rarely enough time to test all combinations. Thus, there is no way to ensure that quality will be maintained despite inevitable processing variations.

Henkel Corp. has overcome this challenge by using designed experiments that utilize a small number of runs to evaluate all factors simultaneously.

“Statistical analysis of these experiments determines all of the combinations of operating conditions that will produce the desired results,” said Bill Fristad, Technical Director, Automotive & Industrial R&D for Henkel. “Then we can design the application to ensure high levels of quality despite variations in process conditions.”

Recently, an agricultural equipment manufacturer asked Henkel to help optimize a tough coating application. The applied coating had to withstand a number of tests including cross-hatch adhesion, reverse impact, pencil hardness, methyl ethyl ketone (MEK) double-rubs to substrate, and 504-h neutral-salt-spray (NSS) testing.

The last test is the most challenging in this application. The paint is applied to the panel and then a line is scribed all the way through to the bare metal. The panel is exposed to a saltwater fog for 21 days, and the total amount of coating that has been lost on both sides of the scribe, a value known as creep, is measured. In this case, the customer required that creep be no more than 3 mm (0.12 in).

Henkel engineers wanted to evaluate two different coatings for this application. Normally, these coatings are cured at 325°F (163°C) for 25 min. In this case, Henkel engineers wanted to evaluate
temperatures within +/-25°F (+/-4°C) and times within +/-15 min of the standard values. The conventional approach would have been to run a series of experiments, changing one variable at a time to determine its effect on creep. For example, engineers would pick a time and then vary the temperature for a series of batches. Or they would pick a temperature and vary the time. It takes approximately one month to coat a batch of samples and run the 504-h NSS test.

The problem with conventional one-factor-at-a-time (OFAT) experiments is that they do not capture interactions between different factors. For example, raising curing time may have a much greater effect on creep performance when the temperature is higher. Using this method, it is necessary to test every possible combination of factors to understand the entire range of process conditions.

For their application, Henkel engineers estimate this iterative series of experiments would have taken six to 12 months. “The nature of the OFAT method plus time limitations on virtually every project make it necessary to take guesses about the best process conditions,” said Chris Weller, Research Chemist at Henkel. “It’s usually not that hard to find acceptable operating conditions, but it’s very possible that a slight change in these conditions might cause quality problems.”

To overcome these limitations, Henkel several years ago switched to the design of experiments (DOE) method. By varying the values of all factors in parallel, DOE reduces the number of runs required to determine the optimal value of each factor. This approach determines not just the main effects of each factor but also the interactions between the factors.

“DOE gives us an understanding of how any combination of factors within the selected range will affect our responses,” Fristad said. “This makes it possible to provide our customers with values of each factor that will not only work but will maximize the robustness of the application.”

Henkel uses Design-Expert software from Stat-Ease Inc. because it is designed for use by subject-matter experts, such as scientists and engineers, who are not necessarily experts in statistical methods. The software walks users through the process of designing experiments and evaluating the results for significant outcomes.

In this application, Henkel engineers selected a response surface method that provides a considerable amount of information on experimental variable effects for the optimization of processes. Weller entered the range of factors, and the software laid on a “central composite” design of nine unique combinations.

Henkel technicians performed each experimental run specified by the software for each coating and then ran the NSS test and measured the creep of each sample. They entered the results of the NSS test into Design-Expert, and the software generated response surface maps that provided visual depictions of the entire application space. These maps provide an intuitive understanding of how coating quality responds to the various factors.

Jerry Fireman, President, Structured Information, wrote this article for SAE Off-Highway Engineering.