

FACTORIAL EXPERIMENTAL DESIGN TO CONTROL AND OPTIMIZE PRODUCTION PROCESSES

THE ISSUE

Manufacturers are engaged on a day-to-day basis with challenges, including:

- Making products with superior end properties (Strength etc.)
- Making products with more consistent end properties
- Dealing with unexpected drops in yield

Often, in-house teams are able to take corrective action, but sometimes the issue is more complex and demands out-sourced support. In these instances Lucideon offers:

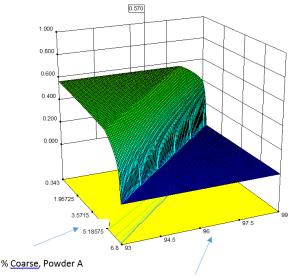
- A broad awareness of the connected process steps that convert raw materials into shaped final products and the associated variables to work with
- Experience in optimizing these process steps in a number of different industries
- A broad range of analytical techniques that characterize starting materials, intermediate and final shaped products
- Expertise in Factorial Experimental Design (FED)

FED AND ITS IMPORTANCE

Variables impacting on final product performance rarely operate in isolation. FED helps to save time and expense by identifying a reduced number of investigative experiments to perform. These experiments are based on process variables you are willing to work with and are usually identified in consultation with Lucideon. Once the series of experiments have been performed and the key measurements have been made on the product from each experiment, FED software calculates the allimportant subtle interactions between variables. Without going through this process, robust solutions leading to better products and yields will not be found.

The graph in Fig. 1 shows a typical output from FED. The z-axis represents "desirability" with

1.0 equating to 100% delivery of one or more properties needed. In this example, the ability of a tablet to disintegrate in aqueous media made up a significant part of "desirability". The x and y axes show how two chosen variables impact on desirability. The 3-D contour plot clearly shows regions where desirability is good. In this example it can be seen that the desirability clearly benefits from lower levels of coarse material in both powders. What is also encouraging is the fact there is a "plateau of opportunity" for desirability offering more flexibility in terms of acceptable powder size distributions. Where there are more variables the software offers a sliding scale to show how the contours shown in Fig 1 alter as each additional variable is changed from a low to a high value. In this particular example we were able to see that increasing the wt% of a third powder caused the upper plateau region to shift to 0.9 (90% desirability).



% <u>Coarse</u>, Powder B

Fig. 1 FED output of two variables showing that less coarse powders are desirable in this application.

POWDER PROCESSING

Regardless of industrial sector, there are many similarities in the way powders are processed.

Although it is accepted that emerging step change technologies like Additive Manufacturing (AM) are changing the landscape, Lucideon has still been called upon to apply the same skills to AM products.

Effective trouble-shooting requires a portfolio of appropriate analytical tests to apply at each stage of processing, and the ability to interpret the data that is generated – this requires understanding of materials and processing, as well as expertise in testing and analysis. The results from these tests help quantify the agreed variables and responses gained from FED work. Some examples of important tests are:

- Dry powders & granulates: particle size, particle shape, powder flow, powder packing, surface area, porosity
- Powder suspensions (fluid or paste-like): rheology, zeta potential
- Shaped products: strength (tensile & compressive), porosity, microstructure, homogeneity

HOW WE WORK

We begin by meeting with the client to understand the issue. This normally includes a detailed walk of the production line to fully appreciate the variables at each step and to discuss which variables clients are willing / not willing to change. Client insight is also crucial here as their experience from previous trials can often help to rule out variables that are known to have little impact.

Where insufficient knowledge of the input and output materials exist, Lucideon would then recommend additional testing. For example, knowledge of particle size distribution might be good but knowledge of powder shape or powder flow might be lacking. Discussions with the client also lead to an agreement on what "responses" to measure. These are usually critical properties in the end product. Applying a weighting to indicate an order or priority for responses is also important.

Lucideon would then use the FED software to define experiments based on the number of variables agreed. This could range from 8-12 experiments for 3 variables and up to 16-20 experiments for 4 or 5 variables. Where the client is able to stop production or provide a pilot processing line, the client may conduct the experiments. Lucideon or the client would then carry out critical tests on the intermediate and / or final product – such "responses" are critical to the FED work and represent all the properties required by the client. If the client is unable to perform the experiments in-house, Lucideon can, in many cases, offer controlled small-scale processing equipment to mimic production.

Finally Lucideon would employ staff skilled in the use of the FED software to process the data and present a summary of the findings. Often we find it useful to actually run through the procedures / assumptions made in the FED analysis with the client. This gives the client the opportunity to re-visit and shape the final "desirability" graphs.

The FED exercise often needs to go through 2 or more iterations to provide optimum solutions. This has the advantage of allowing the client to review progress by applying the recommendations after each FED analysis. A decision can subsequently be taken on whether more FED work has the potential to significantly improve the process further. In all cases the emphasis is on Lucideon working closely with the client as an extension to their existing team, using both party's knowledge to complement each other's.