CATALYSIS CONSULTING

Design of Experiments

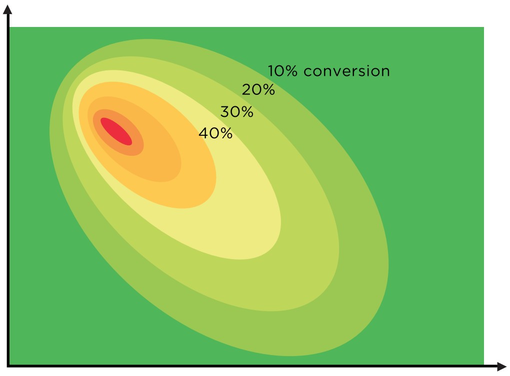
It is prohibitively expensive to experiment in a trial-and-error manner, changing one factor at a time. A far more effective method is to apply a systematic approach to experimentation that considers all factors simultaneously. This approach is called Design of Experiments (DoE) and many scientists use it as an efficient way to solve serious problems afflicting their projects.

A well-performed DoE will provide answers to questions such as:

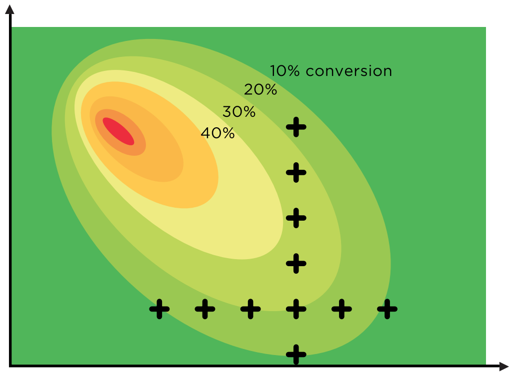
- What are the key factors in a process?
- At what settings would the process deliver acceptable performance?
- What are the key, main and interaction effects in the process?
- What settings would bring about less variation in the output?

DoE, also known as Statistical experimental design or Factorial Experimental Design (FED), deals with planning, conducting, analysing and interpreting controlled experiments. The experiments are designed to enable the evaluation of the factors that may control or affect a change to the experimental outcome (a response).

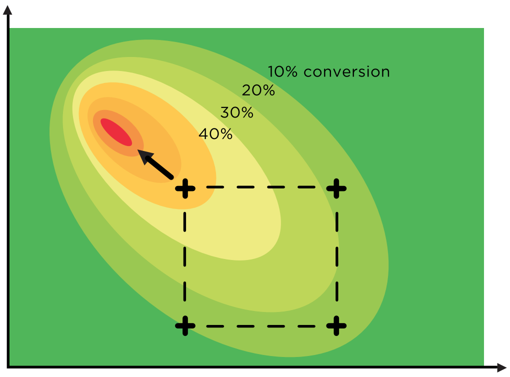
DoE is extremely efficient, enabling the testing of many factors in a limited number of experiments. This drastically shortens the time that would have been spent testing a handful of factors, one factor at a time (OFAT or OVAT) and greatly increases the coverage of the experimental space.



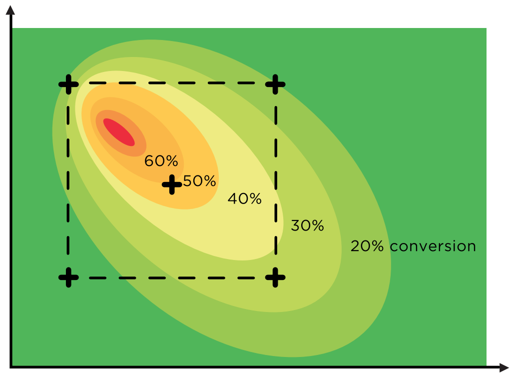
Design Region



OVAT



Screening Design



Optimisation

Any number of factors could affect the performance of a chemical reaction, such as concentration, pressure, pH, solvent, stoichiometry, temperature and time. The factors to investigate are chosen as they are expected to have an effect on the outcome of the reaction (response). DoE enables the experimenter to investigate the direct effect of each factor as well as the combined effect that two or more factors (interaction) may have on the experiment.

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DoE provides information about the interaction of factors and the way the total system works, something not obtainable using the OVAT approach. Another advantage of DoE is that it shows how interconnected factors respond over a wide range of values, without requiring the testing of all possible values directly.

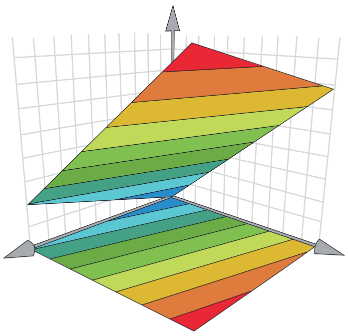
In DoE, the experiments are chosen to investigate the response of the system to changes in the factors. The factors are the sources that are expected to affect the outcome of the experiment. Well-chosen experimental designs maximize the amount of "information" that can be obtained for a given amount of experimental effort.

DoE fits response data to mathematical equations. Collectively, these equations serve as models to predict what will happen for any given combination of values for significant factors. With these models, it is possible to optimize important responses and find the best combination of values for the factors.

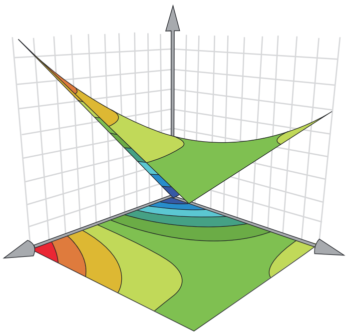
The inclusion of repeated experiments in DoE provides an accurate measure of experimental variation and reproducibility in the reactions, whilst statistical analysis allows for the interpretation of the significance of each of the factors investigated in the reaction.

Designs can be devised to interrogate any factor and its interaction with other factors at several levels of understanding:

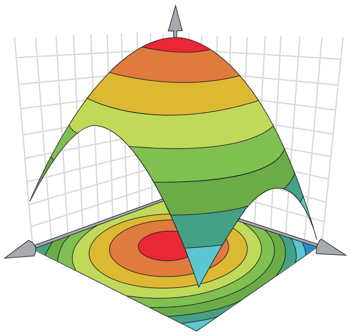
- Linear models provide information about main factors only, but are useful in screening situations with a view to determining which of many factors are important.
- Interaction models provide information about both the main factors and any interactions between those factors.
- Quadratic models can describe non-linear behaviour of factors by taking the curvature of the response surface into account.



Linear Model



Interaction Model



Quadratic Model

The construction of a quadratic model requires many more experiments than the generation of an interaction model and very many more than for a linear model. Therefore, before designing the set of experiments to be undertaken in the laboratory it is essential to consider the objectives of the experimentation and the level of detail required about main factor effects and interactions.

In summary DoE is an invaluable tool to explore important factors in a chemical reaction. It facilitates the systematic variation of multiple factors simultaneously in a highly efficient and scientifically rational manner. The inclusion of repeated experiments provides an accurate measure of experimental variation and reproducibility in the reactions, whilst statistical analysis allows for the interpretation of the significance of each of the factors investigated in the reaction.

Paul Murray Catalysis Consulting provides Consulting and Training in Design of Experiments (DoE), Principal Component Analysis (PCA), homogeneous, heterogeneous and biocatalysis.

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