# Successful Mixture

## PVC-Optimisation using Statistical Design of Experiments

**The correct tuning of the raw material components lies at the foundation of the production of a high-quality plastic product. Unlimitedly long test series are however unthinkable. In order to find with a surprisingly small number of experiments the optimal material composition, Alcan Airex uses the advantages of statistical design of experiments in the development of rigid PVC mixtures.**

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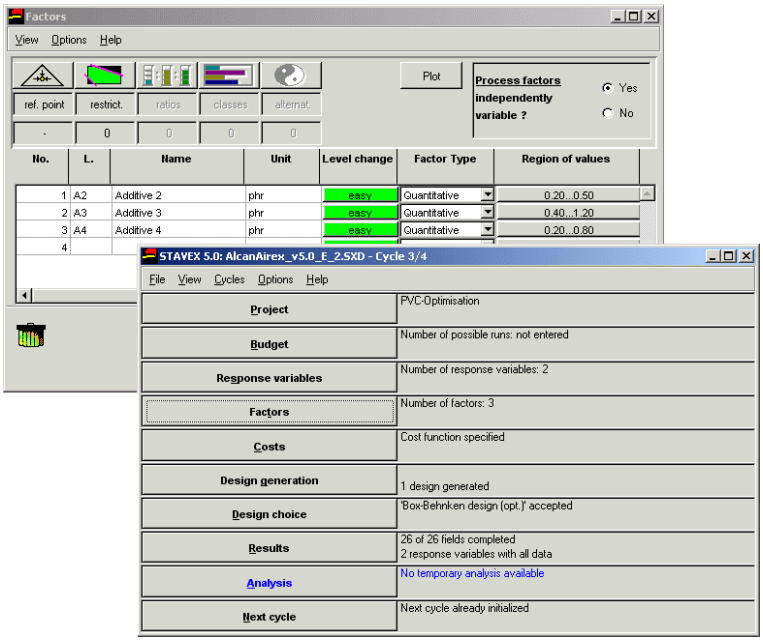


*Figure 1. The main screen of STAVEX guides the user through all stages of experimental design. In the factor screen the individual components are to be specified.*

In the current highly competitive environment, the use of highly effective production processes constitutes a must for the process industry. The ability to reach and to guarantee the quality level required by the market is even more important than the usual maximization of the yield. Therefore, it is generally indispensable to carry out a series of experiments to determine precisely the ideal tuning of the numerous process parameters.

In such a setting, it is too often the case that experiments are performed such that the individual parameters are examined successively, by varying one parameter at a time, while all the others remain unchanged. With this procedure one thing is guaranteed: many experiments are run, however the chances to find the optimum are very low! In other words, laboratory and production resources are efficiently... wasted. But for those who want to get most information with minimal experimental effort, Statistical Design of Experiments constitutes an especially useful tool.
On the one hand, this method suggests optimal experimental designs on the basis of the problem description, i.e., of the response variables to be optimised and of the possible factors of influence. On the other hand, the experimental results are analysed and recommended settings of the process parameters are calculated.

### Tailored designs

For an efficient processing of rigid PVC containing special additives such as stabilisers, lubricants, filler materials etc., an optimal gelling of the PVC mixture is an important precondition. The gelling or plasticizing behaviour of such a mixture can be judged with the help of a measuring kneading machine. This equipment measures the viscosity change over the kneading torque over a given period of time. The goal is to find the best composition of the plastic additives, that

is to say, the composition for which the viscosity is the closest to a given target after a certain kneading time.
Using statistical experimental design, one can identify with a minimum number of tests the relationship between the composition of the rigid PVC mixture and its plasticizing behaviour. The mixture here consists of ten components; the proportion of four of them can be varied. This experimental situation is intricate and the use of an appropriate software almost unavoidable, especially because the problem complexity increases with the number of components. In order to find the composition leading to an optimal gelling behaviour, the above problem was solved with the software STAVEX.
This software tool cleverly guides the user through all the experimental design tasks. First, the response variables including their target values must be entered. Then the user has to specify the individual components with their respective ranges. For instance, the first additive A1 may vary between 0.4 and 1.2 phr, the second between 0.2 and 0.5 phr, etc. (see Figure 1).The mixture can be more precisely characterised by specifying interactions between the components and excluding certain combinations of additives. The specification form is very simple and can be read as: "The amount of additive 3 should be at most as large as that of additive 4". Moreover, pure process settings, such as temperature levels or processing durations, can also be entered.
On the basis of all these factor-related inputs, the software proposes a set consisting of several experimental designs. Here, a factorial experimental design with eight runs was chosen.

## An easily understandable analysis

All specified experiments were performed and their results were entered into STAVEX. A single mouse click suffices to run the data analysis. In the case considered here, a linear regression model was estimated. One can read in the richly documented HTML report, which presents the results in form of fully formulated English sentences, that additive A1 does not have any influence on the optimal viscosity. However, the gelling behaviour can be well controlled over the remaining components. Consequently, the software suggests to set additive A1 to a fixed value and to carry out an additional cycle of experiments with the remaining components. A Box-Behnken-Design with 13 runs was selected from the list of proposed designs. Thanks to the higher number of runs and, at the same time, smaller number of components, it was possible to fit a more detailed regression model with interactions and quadratic effects to the observed data. A more exact result can be then expected.
If it is not possible to comply with the experimental design prescribed by the software, then it is possible to write the actual design in Excel and to import it afterwards into STAVEX. One can proceed likewise if results from unplanned tests exist from the past. The software allows importing and analyzing these as so-called external designs.

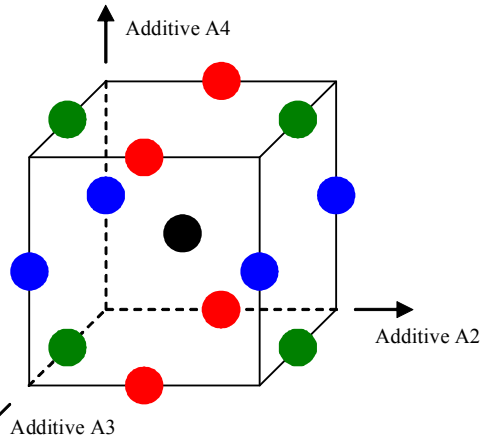


*Figure 2. Box-Behnken-Design for three factors.*

The experimental space, now limited by the ranges of the three components A2, A3 and A4, is represented by the cuboid displayed in Figure 2. The coloured points mark the 13 mixtures used in the tests. With exception of the black point at the center, the quantity of one additive (e.g. A2 for the four red points) is set to the center of its range, while the two other additives are set either to their minimum or maximum level.
After performing those 13 runs in the measuring kneading machine, the observed values of the response variables are entered into the software. In the current example, the analysis yielded that the torque increases, i.e., the viscosity as well, when the proportions of each of the additives A2, A3 and A4 are reduced. A whole panel of graphs enables the user to visualise the numerical results. For example, the 4D contour plot easily allows to determine which combination of the additives leads to an optimal gelling behaviour (see Figure 3).

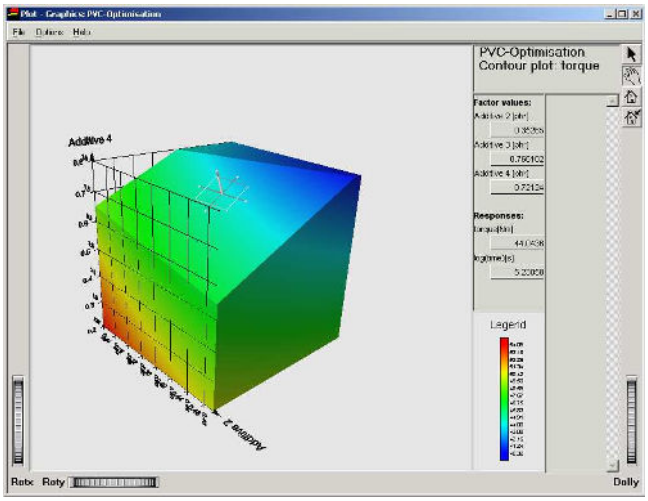


*Figure 3. The numerical results can be displayed using various plots, e.g. the 4D contour plot. To each additive corresponds one dimension; the dimension of the response variable is represented by the colour scale.*

Additionally, the software provides a detailed model diagnosis, including e.g. the corrected coefficient of determination, an analysis of the residuals (normal distribution test and quantile plot), possible transformations of the response variables etc.

The coefficient of determination showed that more than 98% of the variance of the response variable was explained by the model. Furthermore, the resi-dual analysis did not indicate that the assumptions made for the regression were violated. Thus it can be assumed that, for the calculated optimal mixtures, the viscosity indeed lies close to the target value (see Figure 4).

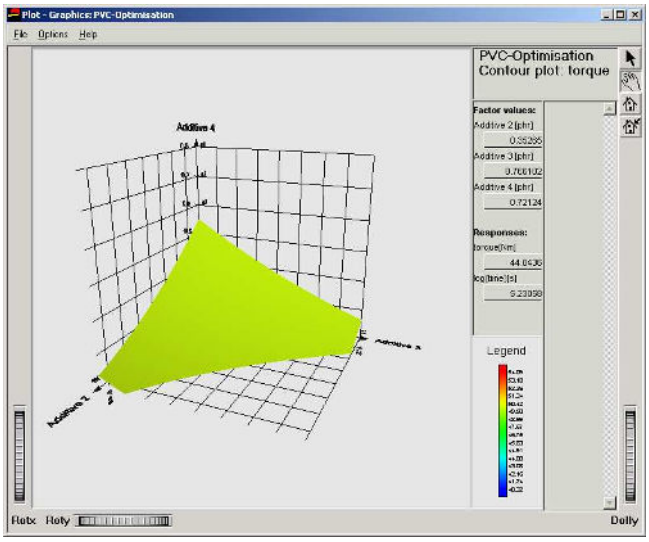


*Figure 4. The surface plot shows those points of the experimental space which lead to an optimal torque.*

In order to validate the model, two confirmatory runs were performed with proposed optimal compositions. In both cases, the viscosity lay within the predicted interval, which is a further confirmation of the good result.

Altogether we have obtained a specific rigid PVC mixture whose composition allows a successful processing in a production plant, with the aid of statistical experimental design and using only 23 runs.

Beside fewer tests and more know-how, the product quality was increased and the costs were reduced. User-friendly software makes these benefits easily accessible, even if the problem is highly complex.

## Statistical Design of Experiments

More than 150 companies of various process industry sectors currently use STAVEX all over the world, for instance: Alcan Airex, Ciba Specialty Chemicals, Clariant, Ems-Chemie, F. Hoffmann-La-Roche, Nestlé, Novartis and Sanofi-Aventis. Version 5.0 offers new diagrams, an increased flexibility, an enlarged design library and many other new features. The software package can be used to optimise laboratory experiments as well as production processes. STAVEX has for example been applied to the following projects:

- Analysis of the dissolution speed of sustained-release formulations
- Maximisation of the output in chemical or biotechnological syntheses
- Optimisation of tablet formulations
- Thermo-sealing of blister packages for tablets
- Identification of important mixture components in the production of adhesives.