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1.5 Design of experiments – what’s that?

1.5.1 Design, factors and effects

The planning and conduction of statistical analysis (also termed “design of experiments” or, abbreviated, DoE) commence at the previously mentioned stages, and therefore gives access to a huge economic potential. But at this stage one question is obvious: what is design of experiments in detail? We consider once more the example of developing a filler concentrate. In contrast to Chapter 1.4.1 experiments are performed only at two concentration levels (meaning low and high amount of surfactant like 2 and 4 % or 20 and 40 % filler).

In case of the traditional one-factor-at-a-time method the design will look like in Figure 1.16a. In case of DoE the set of experiments will analyze all possible combinations of both factors surfactant and filler according to a certain pattern. Usually the experiments are laid out in a symmetrical fashion. But how many experiments will be necessary? Four experiments, as shown in Table 1.3. Either both factors are at high or low concentration levels.

Table 1.3: Design matrix with possible combinations in case of the experiments with two factors and two levels

<table>
<thead>
<tr>
<th>Number of experiments</th>
<th>General identification</th>
<th>Factor A</th>
<th>Factor B</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>(1)</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>2</td>
<td>A</td>
<td>+</td>
<td>-</td>
</tr>
<tr>
<td>3</td>
<td>B</td>
<td>-</td>
<td>+</td>
</tr>
<tr>
<td>4</td>
<td>ab</td>
<td>+</td>
<td>+</td>
</tr>
</tbody>
</table>

Figure 1.15: Volume of new information vs. number of experiments

Figure 1.16: Geometric presentation of the experimental design for the filler concentrate: a) one-factor-at-a-time method, b) design of experiments with a two-factor factorial design
Design of experiments – systematic mania?

low level (low-low, high-high), or one of the factors is high and the other low (low-high, high-low). So, there is an additional fourth experiment in contrast to the one-factor-at-a-time method in which both factors are varied simultaneously. This design is called a factorial design. However, if we look more in detail, this additional experiment offers a lot of advantages, which will be described in the following analysis.

If both factors are fixed at two levels and the response viscosity is measured, a design as shown in Figure 1.16b and Table 1.4 can be concluded and geometrically interpreted as a square. Hence the experimental region is said to be of regular geometry. It is in general strongly recommended to make graphical presentations of the design, because they are much more instructive as listing all experiments in tables. The important point is that each data point has its own identification and corresponds to one experiment, which will be used in the analysis step.

Figure 1.17: Development of a filler concentrate: Presentation of the analysis with effects and main effects

Figure 1.18: Main effect diagram in case of the example filler concentrate

Table 1.4: Factorial design for two factors at two levels

<table>
<thead>
<tr>
<th>Number of experiments</th>
<th>General identification</th>
<th>Factor Surfactant (A)</th>
<th>Filler (B)</th>
<th>Response</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>(1)</td>
<td>-</td>
<td>-</td>
<td>2360 mPas</td>
</tr>
<tr>
<td>2</td>
<td>a</td>
<td>+</td>
<td>-</td>
<td>900 mPas</td>
</tr>
<tr>
<td>3</td>
<td>b</td>
<td>-</td>
<td>+</td>
<td>9500 mPas</td>
</tr>
<tr>
<td>4</td>
<td>ab</td>
<td>+</td>
<td>+</td>
<td>5470 mPas</td>
</tr>
<tr>
<td>Average value (= a0)</td>
<td></td>
<td></td>
<td></td>
<td>4558 mPas</td>
</tr>
</tbody>
</table>
The measured data of the response viscosity from Table 1.4 will be analyzed as shown in Figure 1.17. The first step is the calculation of the effects \( E \) for each factor at each level. They match directly with the difference of the response values.

Effect \( E \) from the surfactant (N) at a low level of the filler (F–):

\[
E(N,F-) = a - (1) = 900 \text{ mPas} - 2360 \text{ mPas} = -1460 \text{ mPas}
\]

Effect of the surfactant at a high level of the filler:

\[
E(N,F+) = ab - b = 5470 \text{ mPas} - 9500 \text{ mPas} = -4030 \text{ mPas}
\]

Effect of the filler at a low level of the surfactant:

\[
E(F,N-) = b - (1) = 9500 \text{ mPas} - 2360 \text{ mPas} = 7140 \text{ mPas}
\]

Effect of the filler at a high level of the surfactant:

\[
E(F,N+) = ab - a = 5470 \text{ mPas} - 900 \text{ mPas} = 4570 \text{ mPas}
\]

The main effect \( HE \) is the average increase of a response due to varying one factor from its low level to its high level. It can be calculated from the average difference in response values when moving from low to high level and keeping the other factors at a constant level:

\[
HE(Filler) = \frac{E(F,N-) + E(F,N+)}{2} = \frac{(b - (1)) + (ab - a)}{2} = \frac{7140 + 4570}{2} = 5855 \text{ mPas}
\]

\[
HE(Surfactant) = \frac{E(N,F-) + E(N,F+)}{2} = \frac{(a - 1) + (ab - b)}{2} = \frac{-1460 + (-4030)}{2} = -2745 \text{ mPas}
\]

On average, an increase of the surfactant concentration leads to a reduction of the viscosity and an increase of the filler content leads to greater thickening. From a perspective of coating formulation, this is comprehensible.

The effects can be illustrated in a so called main effect diagram (see Figure 1.18). Software packages use very often another way, which is shown in Figure 2.16. Real effects are given by the coefficients of the regression model. Details are described in Chapter 1.7 and 3.3.

In alternative to the analysis used until now, main effects can be calculated also from the average of the response at all data points at the higher and lower level and a subtraction of these two average values afterwards (see Equation 1.8). This way of calculation is especially helpful when dealing with more than two factors (see Chapter 2.2.2).

\[
HE(Surfactant) = \frac{E(N,F-) + E(N,F+)}{2} = \frac{(a - 1) + (ab - b)}{2} = \frac{ab - b + a - (1)}{2}
\]

\[
= \frac{ab + a}{2} - \frac{b + (1)}{2} = \frac{5470 + 900}{2} - \frac{9500 + 2360}{2} = -2745 \text{ mPas}
\]

**Basic principle:** The main effect \( HE \) is the average increase of a response due to varying one factor from its low level to its high level. It can be calculated from the average difference in response values when moving from low to high level and keeping the other factors at a constant level. The effects can be illustrated in a so called main effect diagram.
In contrast to the one-factor-at-a-time design a much more general statement about the experimental area (and the system) is possible by DoE, because the factors are analyzed at all levels. The already described alternative factorial design of experimental approach is balanced and objective, no combination has more importance than another. The one-factor-at-a-time method prefers all experiments with a low concentration of the surfactant, because two data points are analyzed at this level (Figure 1.16a). In case of linear behaviour and a good reproducibility of the measurements, with this approach much more deep going statements can be achieved. However, the main effects calculated by the DoE approach offer a general explanatory power and therefore much more possibilities.

**Basic principle:** In contrary to the classical approach, design of experiments offers in the whole experimental area a general, objective and balanced explanatory power. DoE deals with main effects of each influencing parameter, which describe the average increase of a response due to variation of the influencing factor inside the experimental area.

In case of this introductive example just two factors has been used. In the following chapters also examples with much more factors are analyzed (starting from Chapter 2.2.2 and 2.3).

### 1.5.2 Interactions

Already Aristotle said, that “the whole is greater than its parts”. Looking on the effects, it is obvious, that the extension of each effect depends highly on the level of the factor. When this occurs, there is an interaction between the factors.

- Effect of the surfactant at a low level of the filler: - 1460 mPas
- Effect of the surfactant at a high level of the filler: - 4030 mPas

Especially in chemistry interactions have a significant importance in case of non-linear effects (see Chapter 1.4.2.2), but are very often ignored. Think about the synergy between UV-absorber and radical scavenger (e.g. HALS-additives). The whole is also in this case more than only the single effects.

From a perspective of coating formulation, also this effect is comprehensible. The surfactant will wet much more surface of the filler at a low level of the filler concentration. Thus, the effect on viscosity will be different from that one at a high level. However, interactions are not correlations, because the filler content is not changing due to the variation of the surfactant concentration. Only the influence of the surfactant on the response (e.g. viscosity) depends on the filler content, the adjustment of the second factor.

**Basic principle:** Essential statements regarding interactions can only be generated with the DoE approach due to additional data point at high filler and surfactant concentration and the variation of both factors simultaneously. In addition, the one-factor-at-a-time method or the intuitive method offers not such a deep inside into the system.

It is state of the art to calculate a classification number for the interaction WW. In contrast to the main effect it’s the difference from the single effects like shown in Equation 1.9. The interaction WW(FN) is equal to WW(NF) (see Equation 1.9).

\[ WW(FN) = \frac{E(N,F^+)-E(N,F^-)}{2} = \frac{-4030 - (-1460)}{2} \text{ mPas} = \frac{(ab - b) - (a - (1))}{2} \]

\[ = \frac{(ab - a) - (b - (1))}{2} = \frac{E(N+,F) - E(N-,F)}{2} = WW(NF) = -1285 \text{ mPas} \]
Interactions are also plotted in so called **interaction diagrams** (see Figure 1.19, 2.19 or 2.24). This diagram plots also the single effects, which assist during the data interpretation.

Interaction can occur in a different manner (see Figure 1.20). Normally both single effects have the same prefix as the main effect (**ordinary interaction**). In case of strong interactions it is possible, that the single effects cross each other (**disordinary interaction**). In this case, the value of interaction is larger than that of the main effect. Thus, a clear interpretation of the main effects is not possible and statements of the main effects are irrelevant.

![Interaction diagram for the example filler concentrate (viscosity values in mPas)](image)

*Figure 1.19: Interaction diagram for the example filler concentrate (viscosity values in mPas)*

![Types of interactions](image)

*Figure 1.20: Types of interactions*
In such situations it is advisable to look on the effects in diagrams. During dealing with DoE, the interactions should always be smaller than the main effects. Otherwise the main effects cannot be interpreted and analyzed apart from the other effects. Normally interactions – especially higher ones like ABC interactions (see Chapter 2.2.2) are small, because it is not very probable that the effect of a factor depends on the adjustment of three or more other factors. In case of strong interactions statements and interpretations about main effects must be modified, because they do not represent the reality. In these cases it is suggested to increase the distance between the levels, to clarify if the factor has a significant effect or not.

**Basis principle:** Main effects must be larger than interactions. Otherwise they cannot be clearly allocated for interpretation. If this is not the case it is suggested to increase the investigation range and allow the effect of each factor to be captured.

## 1.6 Where is the statistics?

From the beginning of this book we talk always about a statistical approach in design of experiments. But, where is statistics? Until now it was not present.

To achieve the desired accuracy in data interpretation it is necessary, that multiple replications of the measurements are performed. The presence of a certain variance in the data cannot be avoided. These errors are the slight variations that occur when successive measurements are made by the same person under as nearly identical conditions as possible. However, omnipresent not controllable undesirables (noise – fluctuations in materials, surrounding conditions, the measurement, the execution of the experiments, etc.) will always influence the result. Considering the example with the filler concentrate it is obvious, that a certain fluctuation in the data of multiple formulated (replications) concentrates will always occur.

**Basis principle:** Replicate measurements are normally associated with very small random errors, the noise. It’s very unlikely, that replicates show identical average values, despite being obtained under identical conditions.

For the set of data in Table 1.5 following characteristic parameters can be calculated (see Appendix 2 and 4):

- **Arithmetic mean** $\bar{x} = 3425$ mPas
- **Standard deviation** $s = 386$ mPas
- **Confidence interval** $VB (95\%, f = 3) = 614$ mPas

The average viscosity of the filler concentrate has to be declared by:
Viscosity $= 3425 \pm 614$ mPas (at a shear rate of 100 s$^{-1}$, 20 °C)

Table 1.5: Viscosity of the filler concentrate in case of a multiple formulation ($N = 4$) at the data point (1) with 2% surfactant and 20% filler

<table>
<thead>
<tr>
<th>Viscosity in [mPas]</th>
<th>2900</th>
<th>3600</th>
<th>3800</th>
<th>3400</th>
</tr>
</thead>
</table>

This confidence interval is in the simplest case equal in the whole experimental area. Otherwise an average from all variances (the square of the standard deviation $s^2$, see Appendix 2 and 4) of each combination of factors has to be calculated by the geometric mean (see Appendix 2).

What is the sense of a confidence interval $VB$ in case of the average viscosity? This
interval defines a region in which the true value (see Appendix 3) of the viscosity can be found with a certain probability (in this case 95 %) (see Figure 1.21). There is no absolute security for that, because the region has to be calculated (or better estimated) on the base of only 4 values. To gain 100 % security in the ideal case an endless, at least a huge number of experiments has to be collected. This is due to time and cost aspects not possible. Thus, a control sample of 4 runs has been analyzed. The calculation of the confidence interval gives the possibility to implicate what will happen if endless runs will be done. At least we can come up, that the calculated average value will be in case of 95 data points from totally 100 inside this interval. Only in case of 5 experiments the value will be outside the predicted area (see also Appendix 4 and 5, t-test).

**Basis principle:** The confidence interval defines a region in which the true value of the viscosity can be found with a certain probability (e.g. 95 %).

What are the consequences for data analysis in case of DoE? We have to consider these fluctuations! Every statement about effects on the viscosity has taken into account these fluctuations. In other words, not every difference between two values is an effect. The difference between two values is only significant (and therefore an effect can be stated), when the averages are separated by at least two times of the value of the confidence interval (see Figure 1.22). Otherwise the viscosity intervals of the two average values overlap and cannot be differentiated with 95 % probability. In other words, the probability that the difference between the two average values is only random is with 5 % very small.

In case of the filler concentrate a viscosity of 3000 mPas cannot be distinguished from a concentrate with 3100 mPas, because each value of viscosity has a fluctuation of +/- 614 mPas. The formulation and afterwards the viscosity measurement allow no more precise statement. Two average values need a difference of at least 2 · 614 mPas to be sure with 95 %, that there is a difference. In this context very often the terminus **confidence interval or the standard deviation of the main effects** can be found in literature (see Appendix 4). This uncertainty has to be taken into account already during the target formulation. Thus, the target viscosity 2000 mPas is fulfilled, when the value is inside the interval 3425 +/- 614 mPas (at a shear rate of 100 s⁻¹ at 20 °C).

This uncertainty is taken into account during the data analysis and is plotted also in the so called **Pareto-diagram** (see Figure 1.23). This is a column diagram with the absolute values (is the non-negative distance to zero) of each main effect and interaction. Mostly the values are classified and the prefixes are also shown in the diagram. The limit of the confidence interval is plotted as a line. In most cases the double value of the standard deviation (2s), which is close to the confidence value with 95 % probability) is plotted. Dealing with many variables the geometric mean of the standard deviation of all main effects and interactions is favoured. Beside this way of illustration several other possibilities, which are described in Chapter 3.2 can be found. Very often a relative scale is used with so called **standardizes effects.** The effect will be divided by the standard deviation (see Figure 2.17).
Questions about the significance of effects are independent of all experimental design methods. It is anyway crucial for an efficient progress in projects. The target of a project can only be fulfilled in an efficient way, if the target value can be detected by an objective and validated method (the accuracy of the method is known, see Appendix 1 and 4). Unavoidable fluctuations and disturbances have to be taken into account already during target definition. Thus, discussions whether an alternative coating system has a better resistance as the reference are based on valid facts. The difference to the reference material has to be only compared with the precision of the measurement method given by the confidence interval - see Appendix 4. In many cases the confidence interval is larger than the supposed effect or the difference. Already Aristotle said: It is the mark of an educated man to look for precision in each class of things just so far as the nature of the subject admits. In such situations, system knowledge and experience is important to give statements of high quality. Also very often used reference materials or standards cannot help to interpret the effects. All results, also from the reference material, have the same fluctuations. However, such experiments with standards are valuable to check the plausibility of an experimental series! In addition, be aware, that a statistical significant effect is no assurance that it is large enough to have any practical value. Significance is no matter of blind trust. These principles are also relevant regarding technical specifications of products. The variance of the measuring method should consume only a certain amount of the tolerance limit of the product specification. Please consider for details regarding measurement system analysis further literature.\cite{38, 39}. 

Figure 1.22: Uncertainty of statements in case of effects\cite{18} 

Figure 1.23: Pareto-diagram for the example for the filler concentrate. The dotted line represents the upper level of the confidence interval
**Basic principle:** Not every difference between two values is an effect. Every measurement is imperfect and scientists must judge frequently whether a numerical difference is a manifestation of the indeterminated errors inevitable in all measurements. All statements have to take into account the unavoidable fluctuations of experimental data (replications of the measurement, calculation of the mean and the confidence interval). The difference between two values is significant, when the averages separated by at least two times of the value of the confidence interval. However, not every statistically significant difference between two values is large enough to have any practical value. This has to be proofed in a second step.

**Basic principle:** Significance is in general relevant for any target formulation or the quality control of products. Experimental results seldom agree with just one value. Thus, tolerance limits of the product specifications has to be defined. In addition, the variance of the measuring method should consume only a certain amount, ideally only 20 %, of the specified tolerance limit. Otherwise, there is no free space for other fluctuations like from the production process, e.g. due to quality fluctuations in case of raw materials. In addition, a precision is faked which can never be fulfilled: A factory can never produce more precise than measure the specifications.

The approach to analyze data by confidence intervals is also valid for the one-factor-at-a-time method. However, the necessary replications of measurements have different influence depending on the experimental design. If it is necessary to assure a measurement by 4 replicated experiments, in case of the one-factor-at-a-time method \(3 \cdot 4 = 12\) experimental runs has to be done (see Figure 1.24a). With every additional factor the number of runs is increasing by 4. In case of the DoE-approach only 8 runs are necessary, because the design is replicated twice and in each data point two measured values are available (see Figure 1.24a). Nevertheless at each level of each factor 4 experiments exist. Thus the average main effects are even with a lower number of experimental runs as precise as those from the single-factor experiment. If a ratio of the number of experimental runs of both methods is calculated, the DoE-approach is \(12/8 = 1.5\) times more efficient than the one-factor-at-a-time method (see Figure 1.25). In case of three fac-

**Figure 1.24:** Comparison of the one-factor-at-a-time method (always at the left side) with the design of experiments approach (always at the right side). The values in the data points represent the number of necessary experiments (multiple experiments): a) two factors b) three factors
tors the ratio is $16/8 = 2$, because in case of the one-factor-at-a-time method 16 experimental runs are necessary and in case of DoE only one run per each data point (in sum 8 experiments) has to be done (see Figure 1.24b and 1.25). In this case at each level of each factor 4 pairs of values are available and the statistical validation is equal. This calculation can be done for any number of factors and is shown in Figure 1.25.

**Basic principle:** With regard to experiments, which always need to be carried out multiple times, the statistical DoE approach is much more efficient than the one-factor-at-a-time method.

### 1.7 Models – pictures of reality

Each experiment provides some results. Once collected, these data are investigated using data analysis, because in most cases correlations between the data are assumed. This cause and effect correlations, relating the changes in the factors to the changes in the responses, enable a conscious control of the system and an extraction of in-depth information. To make a statement about this issue, we need a (mathematical) model, which represents the basic mechanisms [8]. It enables also afterwards a precise prediction about measured data or an optimization. Main effects and interactions are very often not enough meaningful.

Models are like pictures for us. They are not reality, but approximate representations of some important aspects of reality. A classic example is the landscape or an electric toy train. Think about atomistic models. In this case the word is already included in the definition. Models can be derived from well-established theoretical fundamental laws of science (like the Ohm’s law or the free fall), or have only an empirical background. In the second case, which is much more important in practice (because the system is too complex), correlations between input and output (like viscosity as a function of filler and surfactant content) elucidate how factors influence the response of a system work. Design of experiments produces always an empirical model, which illustrates on a quantitative base the correlation between factors and the response. This is an essential advantage regarding the simple collection of data points. To know the influence of the pH-value of the binder on the effect of the thickener is much more valuable than only the knowledge about the certain value of the optimized pH condition. This compression can never give a clear explanation of the basic mechanisms behind the issue. The system is like a black box and a description of the correlation between input and output takes place (see Figure 1.26). But how is it possible to determine the correlation of the system?
If all data are available, a straight line or even a curve can be drawn through the cloud of data points by a lineal. This line characterizes the correlation between the factor and the response and is called „best fit straight line“. To work with this correlation much more efficient and to perform also predictions, usually it is tried to describe this line with a mathematical equation.

From the perspective of mathematics, the simplest model is the **linear model** with the so called **equation of a line** (see Figure 1.27 and Equation 1.10). In case of one factor, the equation is:

\[ y = a_1 \cdot x + a_0 \]

where:
- \( y \) ... response
- \( x \) ... factor
- \( a_1 \) ... slope of the line
- \( a_0 \) ... axis intercept

In case of more factors, the model is extended to:

\[ y = a_0 + a_1 \cdot x_1 + a_2 \cdot x_2 + a_3 \cdot x_3 + \cdots + a_n \cdot x_n \]

with:
- \( y \) = response
- \( x_1 \) to \( x_n \) = factors
- \( a_1 \) to \( a_n \) = slope of the line respective model parameters regarding each factor
- \( a_0 \) = intercept of the line

The determination of the model equation can be done by the so called **regression analysis** (see Chapter 3.3 and Appendix 7). This tool is offered by many software packages including EXCEL.

Main effects and interactions can be used to describe the effect of the factors on the response. Thus, it is obvious, that they can also be used to derive the linear model. But how can this be done?

The main effect is the average difference between the response at the high (+1) and the low (-1) level of the effect. The distance between the two levels is in this case 2. Thus, the slope is given by half of the main effect (see Figure 1.28). The intercept \( a_0 \) can be calculated from the average of all data points. Therefore, design of experiments offers the possibility to calculate directly the model parameters (at least for a two factorial design (see Chapter 3.3)).
Linear models can only describe easy and not complex systems. There is no degree of freedom. $1 + 1$ is in a linear model always 2. However, sometimes the whole is more than the sum of each part (interactions, non-linear behaviour, see Chapter 1.4.2.2). Regarding the mathematical description in experimental design three very often used types of models can be distinguished (described for two factors, see Figure 1.29).

**Equation 1.12:** \[ y = a_0 + a_1 \cdot x_1 + a_2 \cdot x_2 \]  (linear model)

**Equation 1.13:** \[ y = a_0 + a_1 \cdot x_1 + a_2 \cdot x_2 + a_3 \cdot x_1 \cdot x_2 \]  (linear model with interaction)

**Equation 1.14:** \[ y = a_0 + a_1 \cdot x_1 + a_2 \cdot x_2 + a_3 \cdot x_1 \cdot x_2 + a_4 \cdot x_1^2 + a_5 \cdot x_2^2 \]  (quadratic non-linear model)

In case of two-factorial designs these models can also be derived directly from the DoE-approach.

**Basic principle:** Design of experiments offers in contrast to the classical approach (for both, one-factor-at-a-time or intuitive method) the calculation of an empirical model. The model describes the system with a mathematical approach. Real effects are estimated by the model coefficients. This opens up the possibility to optimize the system and generate predictions about certain parameter settings by simulation. Thus the system knowledge is increasing (success factor system knowledge). However, models are not reality, but approximate representations of some important aspects of reality.

Looking once more on the example of the filler concentrate, the linear model including an interaction of Equation 1.15 can be derived from the main effects and the interaction (Table 1.6). The intercept $a_0$ is calculated from the average value of all data points, which are registered in Table 1.4.
Equation 1.15: Viscosity (in mPas) = $a_0 + a_1 \cdot x_1 + a_2 \cdot x_2 + a_3 \cdot x_1 \cdot x_2$

$$= 4558 \text{ mPas} - 1373 \frac{\text{mPas}}{\%} \cdot \text{Surfactant (in \%)}$$

$$+ 2928 \frac{\text{mPas}}{\%} \cdot \text{Filler (in \%)} - 643 \frac{\text{mPas}}{\%^2} \cdot \text{Surfactant (in \%)} \cdot \text{Filler (in \%)}$$

The model is shown in Figure 1.30, perfectly illustrating the principle of interaction (see Chapter 1.5.2). The slope on both edges is completely different. This means that an increase of the surfactant concentration at the lower level of the filler content has a significant other effect as at the higher concentration level of the filler. An extreme example is shown in Figure 1.29b. In both Figures 1.29 and 1.30 systems are illustrates three-dimensional as so called response surfaces to see the impact of inputs on the response. A two-dimensional

<table>
<thead>
<tr>
<th>Identification</th>
<th>Identification in the model</th>
<th>Main effect and interaction</th>
<th>Model parameter (= Main effect/2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Main effect (Surfactant)</td>
<td>$a_1$</td>
<td>-2745 mPas</td>
<td>-1372.5 mPas</td>
</tr>
<tr>
<td>Main effect (Filler)</td>
<td>$a_2$</td>
<td>5855 mPas</td>
<td>2927.5 mPas</td>
</tr>
<tr>
<td>Interaction (Filler · Surfactant)</td>
<td>$a_3$</td>
<td>-1285 mPas</td>
<td>-642.5 mPas</td>
</tr>
</tbody>
</table>
Design of experiments – systematic mania?

plot or map (contour plot) is also used very often and illustrates the response by contour lines (see Figure 2.16).

One picture due to fitting a model is worth a thousand numbers. Beside the well-arranged overview, Figure 1.29 shows also the much deeper insight about the correlations and mechanisms of the system in the whole experimental area. In addition, due to this step it is possible to clarify important further questions like

- the optimal recipe regarding viscosity (see Figure 1.32) and transparency,
- the range of operation conditions which can be expected as highly robust against viscosity fluctuations – see Chapter 4.3.

In principle the chosen generated design of experiments and the chosen model are intimately linked. It has to be taken into account that a two-factorial plan analyses only two data points (low and high level). Unfortunately through two data points only a linear model with a straight line can be drawn, because another model is not clearly definable. With three data points it is possible to create a non-linear model (see Figure 1.31). However, be careful with very simple models based on two or three data points (see Figure 1.31a and b). There is a high chance to run the risk of overseeing non-linear behaviour.

Models have always only a local explanatory power. Inside the analyzed area interpretations are possible. An extrapolation, meaning a prediction of values outside the analyzed area is forbidden (see Figure 1.31c and 3.6).

Every model has to be analyzed on its plausibility. Later on systematic methods will be shown about this topic (see Chapter 3). Especially additional data points show the explanatory power of the model quite good. Some basic principles of modelling are summarized in Chapter 3.3.3.

All previous statements did not take the fluctuation of the data into account. It was assumed that the model represents reality. However, in practice there is a certain fluctuation due to disturbances, errors, etc. on the response. Ultimately, also the model parameters have an uncertainty. Repeating the experimental design for the same conditions, each time ones
obtains slightly different model parameters. Also in this case an average value and a confidence interval of the model parameters can be calculated which contain the noise (see Chapter 3.3).

In case of the filler concentrate additional data points show, that even although the interaction is taken into account a substantial deviation from the linear model takes place. Finally the system is described by a quadratic model (see Figure 1.32). In addition in Figure 1.32 the area is marked, in which the target value of the viscosity is fulfilled. Experimental design for quadratic models will be described in Chapter 2.4 of the book.

Additional data points open up also the possibility to integrate additional parameters, like dispersing time or other parameters (e.g. sequence of the additive addition or the temperature). By this approach the last open questions about the influence of these parameters can be answered. DoE offers an easy and systematic approach for that, because the designs can always be expanded (see Chapter 2).

Thus, in contrast to the one-factor-at-a-time method design of experiments offers the calculation of a model. Therefore, the correlation between input factors and the response can be derived. **Models**

- compress date perfectly,
- opens up the possibility to make predictions about or simulations of certain situations without a big effort,
- show, which adjustment of the parameters ends up in an optimized operation point and
- show compromise solution in case of conflicting targets.
Thus, design of experiments opens great opportunities to optimize systems. A typical example for such an approach would be the reduction of raw material cost in parallel to keep the performance at the same level or even an increase in performance. In analogy to additional factors also additional target values (e.g. transparency) can be included. Thus, in the already identified area with filler concentrates which have an optimized viscosity, the recipe with the highest transparency can be identified. This approach can also be used for further measurements like storage stability of the viscosity and is called multiple response optimizations (see Chapter 4.2).

**Basic principle:** Regarding modelling some basic principles has to be taken into account. The generated design of experiments and the chosen model are intimately linked. Not every design can be used for every model. Be careful with very simple models based on two or three data points. There is a high chance to run the risk of overseeing non-linear behaviour. However, plausibility has to be checked always and the experimental design has to be adapted to the model.

### 1.8 Overview, possibilities, benefits and limits

Statistical experimental design analyzes systematically the effect of some factors on a response. The methodology joins already in the preliminary planning and is also present during data analysis. The experimental effort is adjusted to the request (maximal information with a minimal effort) and an optimized ratio between the amount of relevant information and the number of experiments is realized. As a consequence time to market is reduced and also the R&D costs. With the global aim to improve the system knowledge, with the aim to make an optimization or even a preventive analysis of influencing factors and disturbances to avoid errors and design robust products and processes, DoE may increase at the project start the number of experiments in contrast to the classical approach. Especially in case of unknown systems in both approaches an intuitive experimentation is necessary to get some experience, to know the limits, etc. Without this knowledge it would be impossible to structure and define the questions for a selective application of the DoE-tool. On the other hand during data analysis information is compressed into a model. This describes how to affect the response by each factor. Thus precise and quantitative predictions about correlations in the system are possible and optimizing steps can be initiated. Finally this ensures the sustainable success with robust products and processes that can be widely used. Problems can be solved very fast and there is a perfect base for further developments (e.g. with a platform strategy). Already the huge and for all people available and documented system knowledge based on facts is of inestimable value. Thus, very often a considerable effort has to be overcome. However, in the long term this gives the best return.

Design of experiments can also be understood as philosophy, which is as package highly cross-linked with supplemental up- and downstream tools like project management, quality management, benchmarking, Quality Function Development (QFD), Failure Mode and Effects Analysis (FMEA), platform strategy, etc. Thus, design of experiments is an integral element of quality planning and quality management in R&D, construction and process planning. Finally, this element perfectly suits to the principles of lean-development [37, 38]. This mindset has the potential to revolutionize product and process design in many kinds. Due to planning the odds of success are increasing. Planning reduced the reaction time in case of external occurrences and there is a learning effect for the next time. However, planning can never predict the future, but its improving the ability to estimate certain aspects and to react in case of changes much faster and flexible.