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Tests on Rinsing Methods for Non-refillable PET Bottles

Preliminary tests were conducted in order to develop a standard contamination, which can be used to analyse the mechanical effects of rinsing. These showed that a 70 % mustard ethanol suspension, dried slightly, is suitable to fulfil this task. In a systematic decision-making process, the conductivity measurement was selected as the method to determine the residual contamination. The performed measurement systems analysis confirmed the suitability of the used Conductometer for this measuring task. The design of the experimental plan as well as the evaluation and graphical display of the results was realized with the software Visual X-Sel® 10.0 in order to attain a large amount of information with a minor effort.
The process of rinsing is used to remove dust particles. Due to the nature and quantity of the dirt, the mustard suspension does not represent a realistic contamination. That is why the rinsing procedure rather resembles a cleaning process than a pure rinsing out process. Therefore an additional test series with the powdered dye Allurared AC as standard contamination was conducted. The percentage of rinsed-out material is calculated based on the original and residual contamination in order to obtain a meaningful value for the effectiveness of rinsing. The target size for mustard as standard contamination is the cleaning rate and for Allurared AC it is the rinsing rate.
The previously reported worse performance of the germ reduction tests of ribbed bottles was confirmed. The maximum cleaning and rinsing rate is reached by the lower setting of the number of ribs (0 ribs) and the lowest tenside concentration as well as the upper setting of air and water pressure. During the experiment with Allurared AC a direct relation between the remaining quantity of water and the rinsing rate was assumed. This could not be confirmed since no correlation between these two parameters was observed.
In conclusion mustard as standard contamination is suitable for the investigation of the mechanical effects of rinsing and for spray shadow tests. Allurared AC can be used in case fast results are demanded or to estimate real rinse programmes or bottle shapes.

Descriptors: rinsing, PET bottles, conductivity measurement, contamination

1 Introduction

PET was launched as a bottle material about 25 years ago. This started a significant and far-reaching change within the beverage industry. This change is still ongoing because PET provides a form of packaging which is significantly lighter and can be moulded into far more varied forms than glass. These are properties that enable effective promotional communication and which also serve to differentiate products from those of fellow competitors. The result is that the consumer can choose from among an increasing variety of different types of PET packaging. Innovation encourages further innovation. New generation PET bottles which have an oxygen barrier can even be used for bottling fruit health drinks, (flavored) mineral water, and beer. This results in yet further increases in the popularity of PET as a packaging material.

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Tables and figures see Appendix

In order to reduce the cost of materials, non-refillable PET bottles with increasingly thin walls are coming onto the market. Non-refillable PET bottles are produced in a wide range of shapes in order to fulfill the mechanical requirements of the beverage industry in terms of stacking strength, pressure resistance and handling rigidity.

The shape has a decisive influence on the rinsing process both for non-refillable and refillable PET bottles. Tests at KHS have confirmed the differences in rinsing behavior resulting from rinsing ribbed PET bottles in comparison with smooth PET bottles.

2 Objectives

The term rinsing does not denote bottle washing in the normal sense, but simply rinsing out the bottles. This means that different conditions need to be incorporated in a test method for the rinsing process than for cleaning techniques in a bottle washer. In the latter case, dirt must adhere so firmly to the bottle that measurable residues are still present even after an intensive washing process. For this reason, substances that can be virtually burned into the surface are used. A wide range of combinations of substances has already been tested for this. Bottlers often use their own mixtures.

Whatever the substances used, until now measurement methods have not been adequate to quantify the remaining dirt more precisely. This is where our tests come in. What is required is to find a type of soiling[1] for a rinser that only adheres slightly to the bottles, which can be easily rinsed out leaving a residue that can be detected following the rinsing process. What makes this more difficult is that it should also be applicable to PET bottles with a highly hydrophobic surface. Due to the lower surface energy, it is difficult to create a layer of dirt that adheres evenly. To date, there is no known standard soiling substance for rinsing PET bottles that fulfils the requirements mentioned.

With the aid of statistical experimental design (DoE = Design of Experiment) a large quantity of information can be obtained with relatively little experimental effort. It is therefore possible to identify both the effects of the factors on the dependent variable as well as interactions between the individual factors.

3 A brief digression on rinsing

The term 'rinse' denotes the rinsing of bottles before they are filled. This is necessary particularly for new bottles that do not go through the bottle washer. The task of a rinser is to remove dust and other dirt particles from the bottles, because foreign bodies can have a detrimental effect on the shelf life of the product and can lead to release of the carbon dioxide when bottling carbonated beverages. Rinsing media include water (cold or hot), air, sterile air, ionized air, ozonized water, disinfectants, and saturated steam. The chosen method depends entirely on the nature of the application.

Rinsing times range from three to ten seconds, depending on the bottle size and rinsing concept. Figure 1 shows the processing sequence for rinsing PET bottles to be aseptically cold-filled after rinsing.

4 A brief digression into statistical experimental design

The aim of statistical experimental design (DoE = Design of Experiment) is to obtain the desired information with the minimum of time and expense. Only one parameter is varied at a time using the classical method of one-factor-at-a-time. This makes it easy to attribute the cause of a difference in the results, but the experimental expense quickly increases dramatically. Modern experimental design recommends investigating all conceivable combinations. Each result is used to calculate several effects. In addition, interactions between two factors can be identified. Consequently, more information is gathered with fewer individual experiments and this can be verified statistically. An effect is demonstrated if the results of a factor deviate significantly from one another for different levels of that factor. An interaction between two factors means that the effect of one factor is dependent on the value of the other.

Those influencing variables that are likely to be important are selected and comprise the factors which are varied in the experi-ment. The values taken by the factors in the experiment are called the factor levels. The results of an experiment are described by dependent variables. These can be measured values or parameters that are calculated from one or several measured values. It is possible to determine several dependent variables from one experiment. Experimental design covers the following steps:

- Description of the initial situation;
- determination of the objective of the experiment and the dependent variables figures and factors;
- setting up the experimental design;
- carrying out the experiments;
- evaluation of the results and their interpretation;
- verification, documentation, and further actions [3].

Kleppmann gives detailed explanations of these procedures in his book.

The aim of D-optimal (D comes from "Determinante") experimental designs is to clearly describe the desired effects and interactions with minimal effort. This focuses on the following advantages: the number of levels of each separate factor, the interval between levels, the mathematical model and the distribution of sample points in the n-dimensional experimental space can be selected as desired.

In contrast to fractional factorial designs, there is no mixing of interactions. In addition, the design can be extended with new influencing variables. Particular levels and combinations which are not feasible or do not make sense can be excluded [5]. Unfortunately, there is still a disadvantage in that, in contrast to full factorial designs in which all combinations of factor levels are investigated, the experimental design is not orthogonal. However, usually only minor deviations occur. In addition, it can only be realized using suitable software [3]. Figure 2 shows a full factorial and a D-optimal experimental design with the factors A, B, and C. Orthogonality means that no correlation exists between the factors, i.e. these can be varied independently of one another. All full factorial designs are orthogonal. However, D-optimal designs with a central point in the middle of the experimental space are not.

5 Materials and methods

The following laboratory equipment is required for the experiments: dispersion device, analytical balance, conductometer, spectrophotometer, pycnometer, refractometer, rheometer.

The suitability of fat, starch, mustard, pectin and Allura red AC powder (Sigma Aldrich) were tested as standard soiling substances for PET bottles to evaluate the effectiveness of rinsing.

Various methods for determining the residual soiling were tested. These methods are electrical conductivity, turbidity, density, viscosity, refractive index, and extinction.

5.1 Measurement systems analysis

All measurement systems display a certain degree of uncertainty. The analysis of results therefore shows random and systematic deviations. These are caused by imperfections in the measuring systems and methods and influences from the environment and operators. Measurement systems analyses define the extent of these uncertainties and allow a statement as to whether a test device is suitable for a particular measuring task [4]. The following processes can be distinguished:

- Accuracy and capability indices C_g and C_{gk},
- repeatability and reproducibility precision and total scatter range with operator effect and
- repeatability and reproducibility precision without operator effect.

For procedure number 2, four methods are described:

- The Range Method (RM);
- the Average and Range Method (ARM);
- the range and standard deviation method;
- the ANOVA method (Analysis of Variance) [1].

In this study, the average and range method (ARM) is used to evaluate the measurement system capability. This requires two testers to measure ten objects at least twice. These values are used to calculate the repeatability and reproducibility (R&R). In order for a new measurement system to be declared capable, $\% R\&R \leq 20\ \%$ must apply, or for systems already in use, $\% R\&R \leq 30\ \%$ [5]. The evaluation is carried out using Visual-XSel® 10.0 software (see also Fig. 5).

6 Preliminary investigations

6.1 Selection of soiling substances

As a result of the findings from the preliminary investigations it was decided to use both mustard and Allura red AC® as the standard soiling substances. The decision was easy to make because both substances offer the ideal properties for detection. The other soiling substances tested such as fat, starch, and pectin were either difficult to apply to the hydrophobic surface of the PET bottles or were difficult to rinse off. It goes beyond the scope of this work to describe the entire test series. For this reason, only the two substances mentioned above are presented.

6.2 Determination of residual soiling

Determination of the residual soiling for the Allura red AC® dye was carried out using extinction measurement, because of the considerable effort involved in detecting minor residues of this kind using other methods. Deciding on a method for mustard was more difficult, as several measurement systems are available for detecting this substance. The suitability of electrical conductivity, turbidity, density, viscosity, and refractive index were tested. Calibration curves using the relevant measuring system were established in advance. Figure 3 shows two such curves for water/mustard suspensions. A non-linear curve, as obtained with the refraction index, results in elimination of this method.

We selected a method for determining residual soiling using a systematic decision-making process (see Fig. 4). Weightings of times three for linear dependency, times two for accuracy, and times one for measurement effort were used for the decision criteria. Due to the non-linear dependency and/or greater effort involved with the refraction index, viscosity, turbidity, and density measurement systems, the decision was made in favor of conductivity for the mustard system, whereas for Allura red, extinction was the best detection method.

6.3 Measurement systems analysis

A measurement systems analysis was carried out for all the measuring instruments used. The result of the conductometer is shown in Figure 5 as an example. The repeatability and reproducibility for this instrument is 13.82 %. It is therefore well below the limit value of 30 %. Consequently, the conductometer was declared suitable for determining conductivity with the defined tolerance of 0.02 mS/cm. This result is also confirmed by the variation coefficient of 1 %.

7 Experimental design

In a brainstorming session, the various influencing and dependent variables in a rinsing process were compiled and the experimental design established. The decision as to which influencing variables should be varied and which kept constant in the tests was based on the importance of accuracy/reproducibility and the amount of effort required. When selecting the dependent variables, the following aspects should be considered: relevance, quantifiability, completeness, and dissimilarity [3]. In our case, the chosen factors were number of ribs on the bottle, the pressures of the air and water rinsing media, and the surfactant concentration level. Two factor levels (smooth and ribbed) were set for the number of ribs and three factor levels for each of the other factors (see Table 1). The remaining influencing variables such as rinsing program, media temperature, and nozzle design were kept constant.

As the aim of the rinsing process is to remove dust particles, the Allura red AC® dye in powder form is a realistic soiling substance in contrast to the mustard. Because of the type and quantity of soiling, the rinsing process for mustard resembles cleaning more than a simple rinsing process which is why the dependent variable is given as a cleaning rate here. For Allura red AC® as the standard soiling substance, the rinsing rate is the dependent variable. A D-optimal experimental design was set up for both systems using the statistical program Visual-XSel® 10.0 (see Table 1). This involves 20 tests each with triple determinations.

The individual tests are arranged in groups. Random variation within these blocks should be as small as possible and each factor level combination should occur with equal frequency in order to minimize chance variation. The tests are also run randomly (in random sequence). This prevents the assessment of the factor effects being distorted by a trend or another unidentified variation in the results [3].

8 Standard soiling substances

Two different standard soiling substances have been used in order to evaluate the effectiveness of a rinsing process for PET bottles. These are mustard and Allura red AC® (see Fig. 1). As described in Part 1, fat, starch, and pectin are not suitable for this purpose and were therefore eliminated after comprehensive tests.

After rinsing, the residues of the standard soiling substances previously added to the bottles were rinsed out using a defined amount of water. These samples were used to determine the electrical conductivity in the case of mustard and the extinction in the case of Allura red AC®. These results represent the residual soiling RC respectively RE. The initial soiling IC or IE was determined in the same manner using reference bottles. The cleaning rate *CR* or rinsing rate *RR* can be calculated from these values using the following equations:

$$CR = \frac{IC - RC}{IC} \cdot 100\ \% \quad [\%] \tag{1}$$

→ applies only to mustard or other equally strongly adhesive soiling substances

$$RR = \frac{IE - RE}{IE} \cdot 100\ \% \quad [\%] \tag{2}$$

→ applies only to Allura red AC or other dust-like soiling substances

with:

CR = cleaning rate (–)
IC = initial conductivity (mS/cm)
RC = residual conductivity (mS/cm)
RR = removal rate (–)
IE = initial extinction (–)
RE = residual extinction (–)

9 Evaluation of the experimental design

9.1 Cleaning and rinsing rates

The rinsing tests using mustard as the standard soiling substance produced cleaning rates of 61.0 % to 96.5 %. This equals a range of variation of 35.5 %. With Allura red AC® as the standard soiling substance, the rinsing rates lie between 98.70 % and 99.99 %, corresponding to a range of variation of only 1.29 %. If the range of variation is large, then the individual factorial effects are more strongly developed and are easier to identify. Figure 2 shows the cleaning and rinsing rates for all factors. In the case of cleaning rate, the air pressure effect is greatest (11.5 %), followed by the number of ribs (8.0 %), water pressure (5.5 %) and surfactant concentration level (3.0 %). Rinsing rate is dependent primarily on the number of ribs (0.55 %) and only to a minor degree on air and water pressure (both 0.05 %), while no significant effect is demonstrable for the surfactant concentration level. There is an interaction between the number of ribs and the water pressure for both the cleaning rate and rinsing rate. The maximum calculated is obtained with the lower setting for number of ribs and surfactant concentration and the upper setting for air and water pressures.

The gradient of the graph shows the magnitude of the effect!

Figure 7 illustrates the gradient of each factor and their effects on the dependent variable. The steeper the gradient, the greater the effect. This is decisive for interpreting the data. If the gradient is negative, then the factor has a negative affect on the dependent variable.

The bottle shape plays a decisive role effect for rinsing. Cleaning and rinsing rates are inversely proportional to the number of ribs. If the bottle has a smooth surface, the water can flow off evenly and rinse away the dirt. The ribs present an obstacle to this process by slowing down the rinsing medium and therefore reducing the mechanical cleaning effect. Between the ribs are dead areas where rinse water does not penetrate well. In addition, the surface area of ribbed bottles is larger. In order to obtain the same cleaning effect for ribbed bottles as for smooth ones, a larger quantity of rinse medium would be necessary i.e. it would be necessary to lengthen the rinsing times or increase the pressure. Under identical conditions, it is obviously impossible to obtain the same results as with a smooth bottle. During the tests with Allura red AC it was apparent that numerous water drops remained on the ribs. Although the dye particles were mostly removed from the surface of the bottle, they were not completely rinsed out of it. This led to a considerable reduction in the rinsing rate.

The cleaning and rinsing rates are proportional to the air and water pressure, although the effects are of differing magnitude. Higher pressure in the rinsing medium increases turbulence which accelerates the removal of the dirt. An increase in pressure also produces an increase in the flow rate. With constant rinsing times, this means that more water is injected into the bottles, enabling a larger quantity of soiling to be removed. However, from a particular point onwards this is counterproductive because the water flow gurgles in the bottle neck and cannot run out.

The higher the surfactant concentration level of the water used for rinsing, the lower the cleaning rate. Surfactants are used in practice to reduce the surface tension. This should improve the wetting of the PET and therefore the effectiveness of rinsing. Because the entire surface is soiled with mustard in these tests and at the start of the rinsing process there is no contact between the bottle and the rinsing medium, this effect does not occur. The surfactant used causes slight foaming. This foam reduces the rinsing effect and also the power of the jet from the nozzle. This is a common effect with high amounts of foam.

Using surfactants to lower the surface tension results in a reduction in drop size [7]. According to Loncin [6], the kinetic energy and thus the effectiveness per kilogram of cleaning solution is lower for smaller drops. When rinsing out dye particles, no significant effect of surfactant concentration could be determined. This can be explained by the fact that the negative effects of additional surfactant described are compensated by the improved wetting.

9.1.1 Interactions

An interaction between two factors means that the effect of one is dependent on the value of the other. Figure 5 shows the significant interactions. The blue lines refer to the number of ribs and the red ones to the water pressure. (+) indicates the upper factorial level in each case and (–) the lower one. For the cleaning rate it can be seen that the effect of water pressure for smooth bottles is 10 times as high as for a bottle with 18 ribs. The cause of this interaction could be the decline in mechanical cleaning power on the ribs and the dead areas between them. This means that an increase in the water pressure for ribbed bottles only produces a minor improvement in the cleaning effect. The rinsing rate is not dependent on water pressure in smooth bottles. One explanation might be that, as a result of the lower degree of soiling, the optimum for rinsing out the dye particles is already achieved at the lower water pressure level and therefore no further improvement can be made. In the case of ribbed bottles, increasing pressure produces a better rinsing result. Because a higher pressure is necessary for the same rinsing rate, the optimum must therefore lie at a higher level.

The rinsing tests reveal that the dirt is rinsed out from top to bottom. The largest amount of residual soiling always occurs in the lower part, when looking at the bottle upside down in the rinsing position, while the upper part (the base) is mostly visually clean (see Fig. 11). When the jet hits the base of the bottle (approximately in the middle of the bottem), this has the greatest mechanical cleaning energy and can remove a large amount of dirt. When running down the wall of the bottle this energy declines and so no longer has the same effect.

9.2 Conclusion and comparison of methods

The test series using mustard as the standard soiling substance produces more marked differences in the mechanical effects of rinsing than that using Allura red AC®. In addition, a visual assessment of the rinsing results can be carried out, allowing critical areas on the bottle to be identified. Please note: mustard is certainly not a realistic soiling substance, but in order to be able to evaluate the results of the tests, this soiling must be so persistent that a measurable residual soiling always remains in the bottle. One drawback is that the bottles must be prepared six days before the tests can be carried out. However, it would be possible to shorten the drying time if the rinsing program were to be reduced. One advantage of Allura red AC® is that bottles can be prepared quickly and easily, after which the tests can be carried out immediately or at a later date. Furthermore, the dye particles constitute a realistic soiling versus mustard. Mustard is suitable for examining the mechanical effects of rinsing and for carrying out spray shadow tests. On the other hand, Allura red AC® could be used for evaluating different bottle shapes and real rinsing programs. A positive result would then require that an as yet undefined limit value not be exceeded. In addition, Allura red AC® could be used if quick results are required.

The cleaning rate (mustard) is calculated according to equation 1, the rinsing rate (Allura red AC) according to equation 2. These are listed in the chapter on standard soiling substances. No target values are given because the results depend on many different factors (e.g. rinsing program, bottle size, and bottle shape).

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Footnotes

1 Let us call it that from now on, because dirt cannot be removed by a rinser.

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Appendix

Table 1 D-optimal experimental design

Test no.	No. of ribs[1]	Air pressure[2]	Water pressure[3]	Surfactant conc.[4]	
1	18	7	6	400	
2	18	5	6	0	
3	1	6	5	0	group I
4	1	5	4	0	
5	18	5	6	0	
6	1	7	4	200	
7	1	5	6	400	
8	18	7	4	400	group II
9	1	5	5	400	
10	18	5	4	0	
11	18	5	4	200	
12	18	6	4	200	
13	1	6	4	400	group III
14	1	7	6	400	
15	18	5	6	400	
16	18	7	4	0	
17	18	7	5	200	
18	1	7	6	0	group IV
19	18	7	6	0	
20	18	6	5	200	

Footnotes
1) –
2) kPa
3) kPa
4) ppm

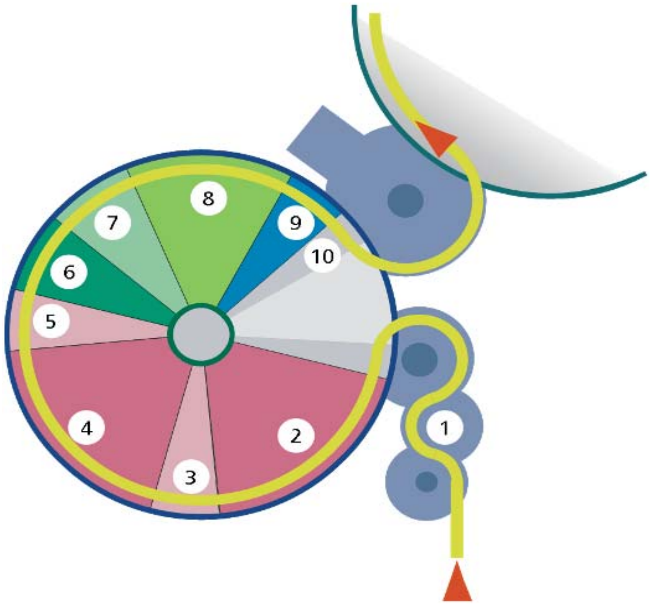
Table 2 Work instructions for standard soiling

Mustard

- Produce a homogenous ethanol/mustard suspension (mustard concentration 70 %) using a dispersion device
- Pour the suspension into the bottles (20 g per 0.5 L, 35 g per 1.0 L, 50 g per 1.5 L bottle)
- Close the bottles and shake vigorously until the entire bottle interior is equally soiled
- Place the open bottles in a refrigerating cell (7°C) for 6 days
- The bottles are ready for rinsing
- After rinsing, Add 50 mL of bidest. water to the bottles and shake 40 times to loosen the remaining soiling
- Pour the suspension into an Erlenmeyer flask
- Using an additional 50 ml of bidest. water, rinse the bottles again, shake 20 times and empty into the flask
- Measure the conductivity (subtract the conductivity of the water used from the measured value)
  → Residual soiling *RC*
- Determine the initial soiling *IC* using reference bottles

Allura red AC®

- Allura red AC is an azo dye (2-naphthalenesulfonic acid disodium salt)
- In using as Food dye E129
- Record the empty weight of the bottles
- Add 50 mg of Allura red AC to the bottles
- Seal the bottles and evenly distribute the dye
- The bottles are ready for rinsing
- After rinsing, Add enough bidest. water to the bottles until total weight = 25.0 g + empty weight
- Shake the bottles carefully to remove dye particles which are still adhering to the walls of the bottle
- Fill a 5 cm cuvette excluding gas bubbles and place in the holder of the spectrophotometer
- Measure the extinction at 500 nm (if the measuring range is exceeded, use a 1 cm cuvette and multiply the measured value by a correction factor of 5)
  → Residual soiling *RE*
- Determine initial soiling *IE* using reference bottles (dilute a sample 1:100 and multiply the measured value by a correction factor of 100)



1 Exterior disinfection
2 PAA (+ sterile air)
3 Reaction period
4 PAA (+ sterile air)
5 Reaction period
6 Sterile water
7 Drip-out
8 Sterile circulation water
9 Sterile air
10 Drip-out

Fig. 1 Sequence of a rinsing process before aseptic cold filling [KHS AG]

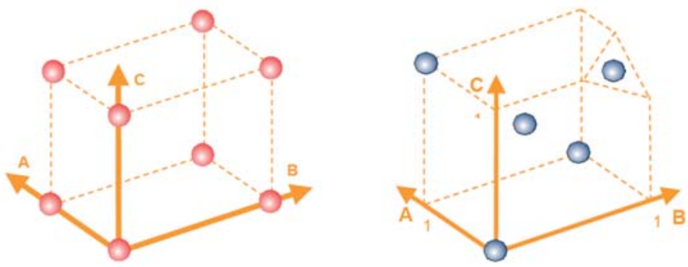


Fig. 2 Full factorial (left) and D-optimal experimental design (right)

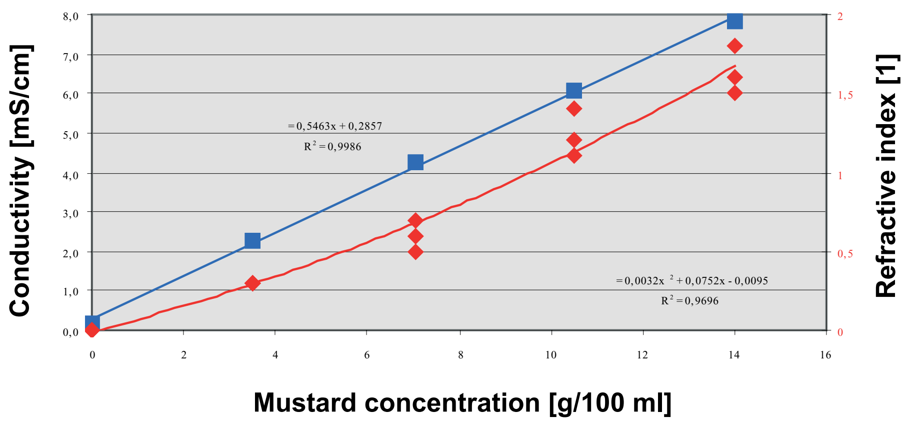


Fig. 3 Calibration curves for water/mustard suspensions

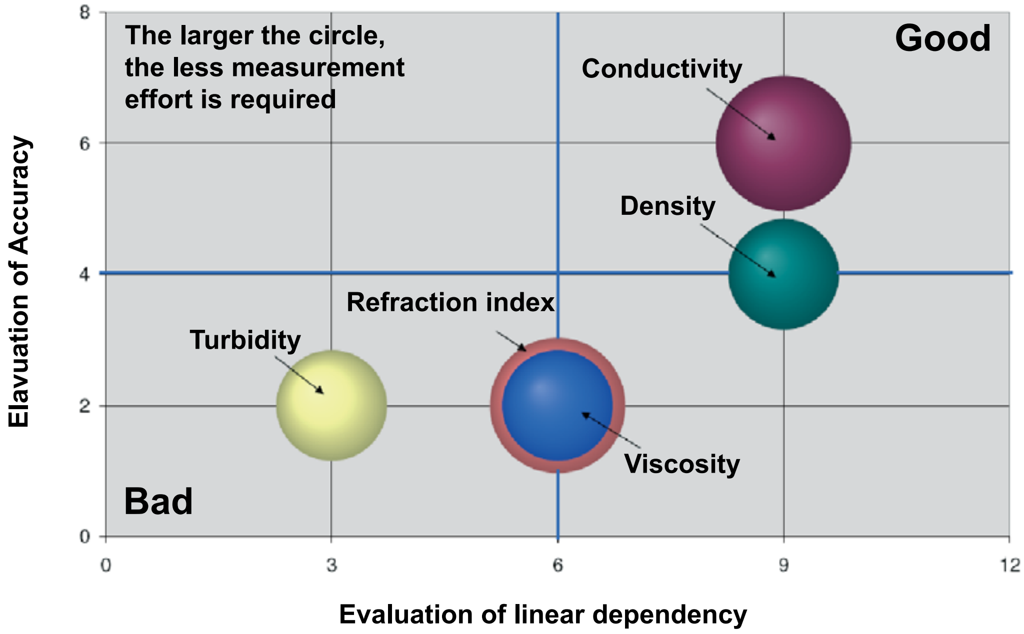
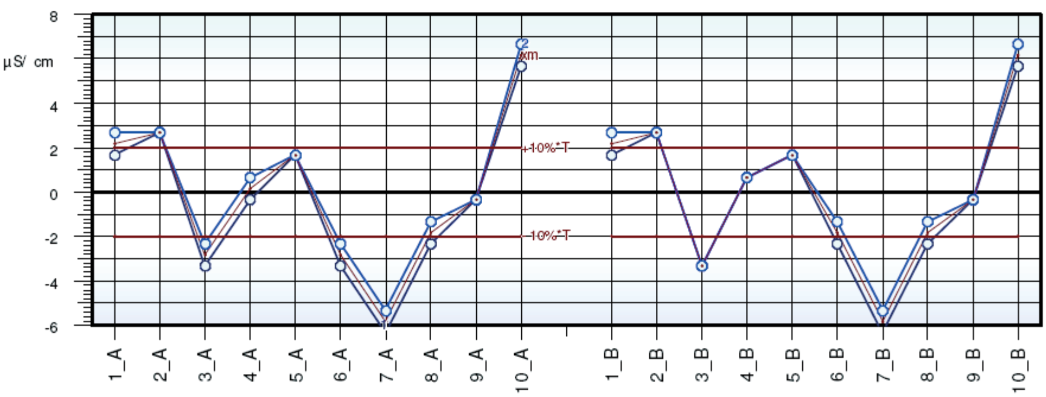


Fig. 4 Results of suitability of test systems with residual soiling using mustard

	A-Prüfer A			B-Prüfer B		
	1	2	R	1	2	R
1	519	520	1	519	520	1
2	520	520	0	520	520	0
3	514	515	1	514	514	0
4	517	518	1	518	518	0
5	519	519	0	519	519	0
6	514	515	1	515	516	1
7	511	512	1	511	512	1
8	515	516	1	515	516	1
9	517	517	0	517	517	0
10	523	524	1	523	524	1



R&R 2,7647
Cgp 2,506
Cgpk 2,485

Measurement:

→ **System capable!**
***%R&R* < 20**

% R&R 13,82

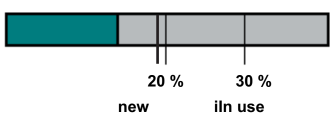


Fig. 5 Results of the measurement systems analysis

Fig. 6 Bottles with standard soiling substances: mustard (left) and Allura red AC® (right)

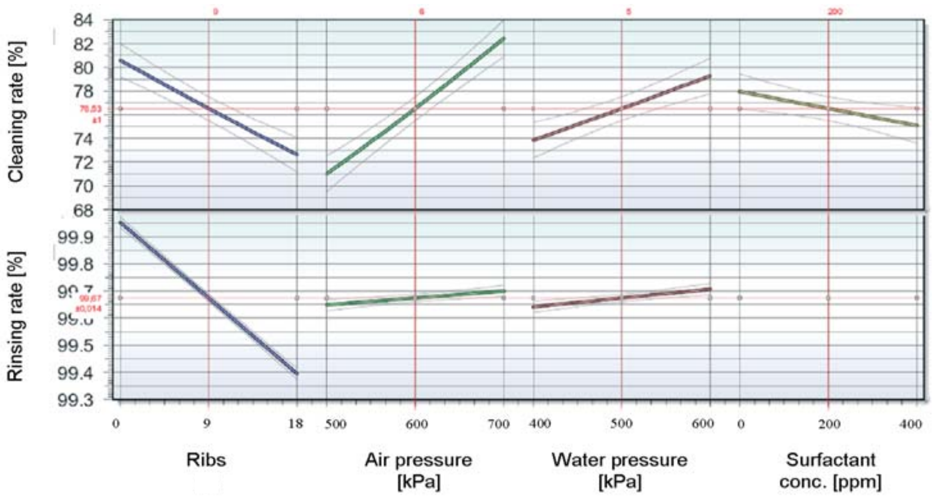


Fig. 7 Graph of cleaning and rinsing rates

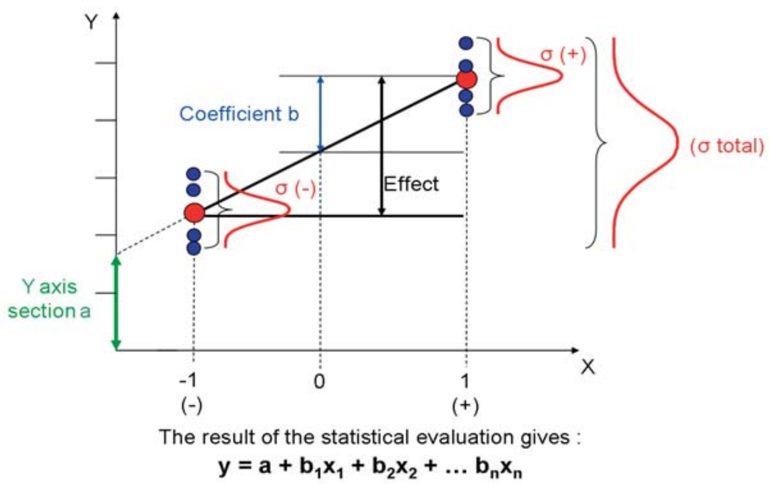


Fig. 8 The gradient of the graph shows the magnitude of the effect

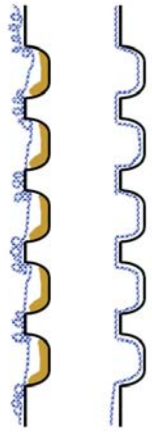


Fig. 9 Run-off and rinsing behavior in ribbed bottles. Right, normal flow behavior of the rinse liquid under ideal laminar flow. Left, disruption to flow on the ribs and location of residual soiling

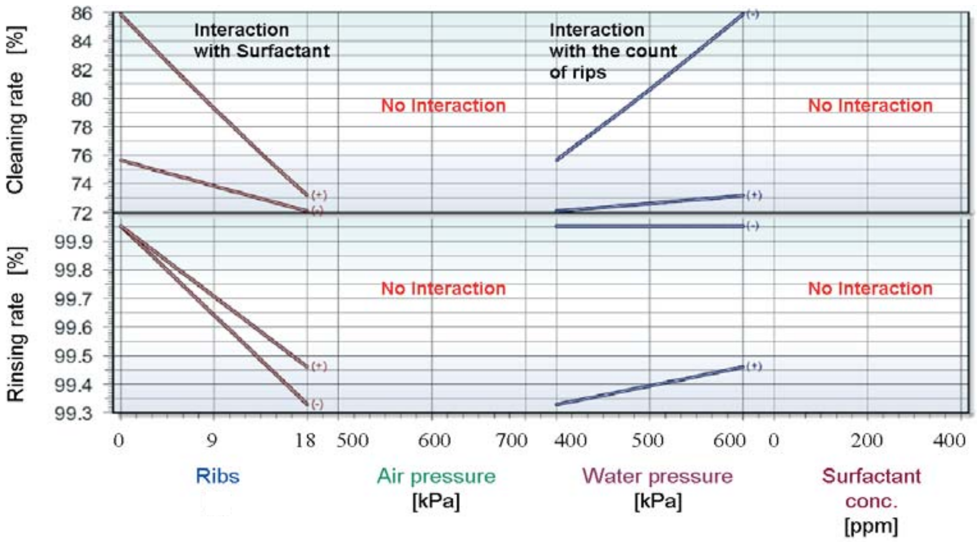


Fig. 10 Interaction of cleaning and rinsing rates

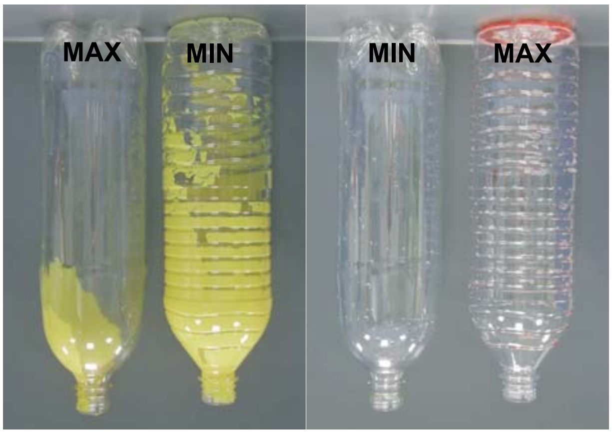


Fig. 11 Bottles rinsed with minimum and maximum cleaning rate levels (left) and rinsing rate levels (right). The mouth down is the orientation of a bottle in a rinser