Investigation of the Factors Influencing the Bulk Density and Open Porosity Testing Results for Refractory Materials

E. Dahlem, S. Clasen, C. Dannert

This paper highlights the efforts that were undertaken within the framework of the EU FP7 ReStaR research project for the determination of testing parameters and conditions ("factors") influencing the bulk density (BD) and open porosity (oPo) testing results of refractory materials. Besides the generation for precision data, it is necessary to understand which instructions in the standard EN ISO 1927-6 for unshaped refractories and in the standard EN 993-1 for dense shaped refractories and carbon-bonded refractories are sensitive for diverging testing results and are therefore to be considered as significant factors influencing those results.

1 Summary

Screening factorial designs were used to identify those significant factors in bulk density and open porosity testing of unshaped as well as dense shaped and carbon bonded refractories. For each material, up to fifteen possible factors were identified and set to two different levels each. Accordingly, 16-trial runs, taking into account the possible factors, were carried out in laboratories with each type of refractory.

The analysis of variance method was used to analyse the testing results. For i.e. unshaped refractories, results showed that the format of the samples, the immersion liquid (water/paraffin) and the sponging material are factors that statistically significantly affect the results of BD and oPo testing. On the other hand, the method of the test piece preparation, i.e. if the sample is cut with a saw or first tested in a three-point bending test, the time of evacuation and the soaking time under water at ambient conditions were found not to influence the results of BD and oPo testing.

A second design of experiment, which contained the four most significant factors, was studied for each refractory material in four different laboratories. Hereby estimations of the repeatability and of the reproducibility of the testing standards for BD and oPo across different laboratories were obtained.

- For the EN ISO 1927-6 standard tested on a MCC unshaped refractory, a reproducibility of 0,05 g/cm^3 and a repeatability of 0,03 g/cm^3 were obtained for the BD and a reproducibility of 1,4 vol.-% and a repeatability of 0,9 vol.-% were obtained for the oPo.
- For the EN 993-1 standard tested on an alumina dense shaped refractory (not reacting with water as immersion liquid), a reproducibility of 0,07 g/cm^3 and a repeatability of 0,04 g/cm^3 were obtained for the BD and a reproducibility of 1,6 vol.-% and a repeatability of 1,2 vol.-% were obtained for the oPo.
- For the EN 993-1 standard tested on a carbon bonded dense shaped refractory (potentially reacting with water as immersion liquid), a reproducibility of 0,04 g/cm^3 and a repeatability of 0,04 g/cm^3 were obtained for the BD and a reproducibility of 0,6 vol.-% and a repeatability of 0,5 vol.-% were obtained for the oPo.

2 Introduction

The testing standard EN ISO 1927-6 for unshaped refractories and the testing standard EN 993-1 for dense shaped refractories inclusive carbon bonded refractories, which describe the BD and oPo testing methods for these refractory materials, include up to 15 factors (for example the factors quoted in Tab. 1 including also factors not explicitly described in the testing standards, e. g. the way of wringing the cloth in order to have it saturated with immersion liquid, the influence of the temperature of the immersion liquid coming into contact with the sample at the beginning of the test, the time that a sample remains in a desiccator after having been dried at 110 °C, the position of the samples in the vessel …), which can vary significantly among different laboratories. A more precise definition of these factors could lead to improved accuracy and precision of the results of BD and oPo testing and thus make the results more comparable.

A conventional method to identify the significant factors that influence the results of a testing method is to vary one factor for each experimental run while keeping all other factors constant. This method is very expensive and time consuming and fails to determine the combined effect of multiple factors on the testing result. That is why a number of factorial designs have been used to address these problems [1]. Among these, full factorial designs pro-

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vide more complete information, but this method requires numerous experiments (L^k experiments, where k is the number of factors and L is the number of levels for each factor). This makes full factorial designs impractical if a large number of factors have to be studied. However, screening designs (fractional factorial designs) offer an effective and economic means to analyse such large numbers of factors using a minimum number of experiments.

Plackett-Burman design (PBD) [2] is the most frequently used screening design because of its ability to estimate all main effects (calculated as the difference of the mean results of a number of tests with different levels of a particular factor) with the same precision. The main effect of each response is evaluated as the difference between averages of measurements made both at the high level (+1) and the low level (–1) of each factor. It is a fractional factorial design with the advantage of minimising the experimental runs, finding the most significant factors from a large number of factors. Using this design, k factors can be screened with k + 1 experimental runs.

In this study, the most significant factors which affect the accuracy and the precision of the testing method for the determination of BD and oPo according to the testing standard EN ISO 1927-6 for unshaped refractories and the testing standard EN 993-1 for dense shaped refractories including carbon bonded refractories were determined. After the identification of the most significant factors that influence the testing results when testing these three different materials, a second design of experiment, which contained the four most significant factors was studied for each material in four different laboratories in order to obtain an estimation of the repeatability and of the reproducibility of the two testing standards between laboratories.

3 Materials and methods

3.1 Refractory material description and material preparation

3.1.1 MCC75 unshaped refractory used to study the standard EN ISO 1927-6 (Tab. 1)

For this study, a bauxite MCC unshaped refractory product produced by Calderys within the context of the EU research project ReStaR [3, 4] was used. It contains Al_2O_3 (78,0 mass-%), SiO_2 (15,0 mass-%), CaO (3,1 mass-%) and Fe_2O_3 (1,0 mass-%). The material has a BD after firing at 800 °C of 2,73 g/cm³ (EN ISO 1927-6), an oPo after firing at 800 °C of 20 % (EN ISO 1927-6) and a cold crushing strength after firing at 800 °C of 120 MPa (EN ISO 1927-6) according to the data sheet of the material.

The castable was mixed in a Hobart mixer with 5,85 % of tap water at about 18 °C

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and the mixture was cast into B and D moulds thereafter. The moulds were filled, vibrated for 30 s, filled to the top and again vibrated for 300 s for format B or vibrated for 150 s for format D. The samples were set in a climate cabinet for three days (20 °C and 91 % humidity) and a further day in a drying chamber. After the first day in the climate cabinet, the samples were demoulded and returned into the climate cabinet. After drying, the samples were fired at 1400 °C for 5 h. This firing regime was chosen in order to ensure that CA_6 was formed inside the material [5] and that the material was stable against rehydration by water during the subsequent testing. It must be emphasized that the firing temperature and the firing duration are expected to significantly influence the testing results, whereas the standard definition in EN ISO 1927-6 merely speaks of "fired" samples. Especially low-fired unshaped refractories can show rehydration effects when immersed in water, which may significantly influence the testing results.

Tab. 1 Description of the materials used to study the two standards

Standard	Applicable to	Material Used in the Investigation	Material Code
EN 993-1	Dense shaped refractory (not reacting with water as immersion liquid)	High alumina product HA75 (RHI AG) (shaped)	HA75
	Carbon bonded dense shaped refractory (reacting with water as immersion liquid)	Magnesia-carbon bonded shaped refractory product (shaped)	MaCarb
		Magnesia-carbon bonded shaped refractory product MC95 (RHI AG) (shaped)	MC95/10
EN ISO 1927-6	Unshaped refractory	Bauxite MCC (Calderys)	MCC75

3.1.2 HA75 dense shaped refractory used to study the standard EN 993-1 (Tab. 1)

The high alumina product type HA75 ISO 10081-1 is a bauxite dense shaped refractory product produced by RHI AG for the need of the EU project ReStaR. It contains mainly Al_2O_3 (81,0 mass-%), SiO_2 (13,0 mass-%), TiO_2 (3,2 mass-%) and Fe_2O_3 (1,7 mass-%) (EN ISO 12677). The material has a BD of 2,75 g/cm³ (EN 993-1), an apparent porosity of 20,5 % (EN 993-1) and a cold crushing strength of 70,0 N/mm² (EN 993-5) according to the data sheet.

3.1.3 MaCarb carbon bonded dense shaped refractory used to study the standard EN 993-1 (Tab. 1)

This magnesia-carbon bonded shaped refractory product with 100 % fused magnesia contains mainly MgO and a carbon

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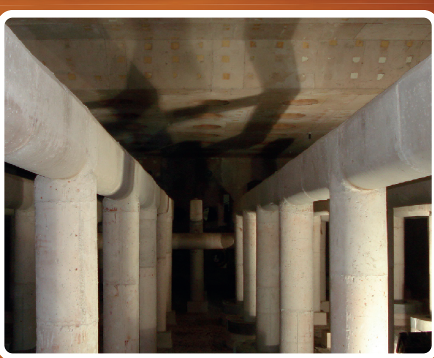

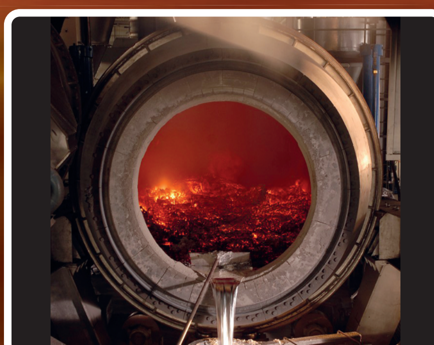

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Tab. 2 Factors and levels used for the investigation of the standard EN ISO 1927-6 using the MCC75 unshaped refractory

Factor	Label	Low Level (–)	High Level (+)
Preparation of the samples	Test	Cut with a saw	Three-point bending test
Waiting time under vacuum before filling with the immersion liquid	Tvac [min]	15	30
Vacuum level	Vac [Pa]	2500	3500
Waiting time after filling with the immersion liquid under vacuum	Wp [min]	30	180
Waiting time after filling with the immersion liquid and under atmospheric pressure	Wwp [min]	30	90
Sponging material (cloth, towel)	Sp	Linen	Leather
Format of the samples	For	B	D
Immersion liquid	Liq	Water	Paraffin (Puretol)

Tab. 3 Factors and levels used for the investigation of the standard EN 993-1 using the HA75 dense shaped refractory

Factor	Label	Low Level (–)	High Level (+)
Operator	Op	Operator 1	Operator 2
Waiting time under vacuum before filling with the immersion liquid	Tvac [min]	15	30
Vacuum level	Vac [Pa]	2500	3500
Waiting time after filling with the immersion liquid under vacuum	Wp [min]	30	45
Waiting time after filling with the immersion liquid and under atmospheric pressure	Wwp [min]	30	45
Sponging material (cloth, towel)	Sp	Linen	Leather
Geometry of the samples	Geo	Prism	Cylinder
Immersion liquid	Liq	Water	Paraffin (Puretol)
Temperature of the immersion liquid coming into contact with the sample	Temp. [°C]	15	30
Waiting time in a desiccator of the test piece after having been dried at 110 °C in the drying chamber	Texi	Necessary time to reach the room temperature	24 h
Position of the samples in the airtight vessel	Pos	Under (1st level)	Up (2nd level)
How to wring the cloth/towel saturated by the immersion liquid	Wr	Wring the cloth/towel	Sin-dry with a roller
Press direction of the samples	Press	Cut or drilled in the press direction	Cut or drilled perpendicular to the press direction
Place of extraction of the samples from bricks	Bp	Center of the brick	Side of the brick
Volume of the sample	Vol [cm^3]	50	200

content higher than 9 mass-%. The material has a BD higher than 3,03 g/cm^3, an apparent porosity smaller than 6,5 vol.-% and a cold crushing strength higher than 35 N/mm^2.

3.1.4 MC95/10 carbon bonded dense shaped refractory used to study the standard EN 993-1 (Tab. 1)

The MC95/10 is a magnesia-carbon bonded shaped refractory product produced by RHI AG/AT for the need of the EU project ReStaR. It contains mainly MgO and C (10,0 mass-%). The material has a BD of 3,05 g/cm^3 (EN 993-1), an apparent porosity of 4 % (EN 993-1) and a cold crushing strength of 35,0 N/mm^2 (EN 993-5) according to the data sheet.

3.2 Evaluation of the error of the testing equipment

The uncertainty of the testing equipment used in the scope of this investigation was evaluated for each material in order to know the level of confidence of the results and its quality. For this the software GUMsim was used. The algorithms involved are based on the current international principles/instructions of DIN/ISO/BIPM Guide to Expression of Uncertainty in Measurement (GUM) [6, 7]. In the case of the investigated testing methods for BD and oPo, the uncertainty of the testing equipment is based on the propagation on the uncertainty of the weighting for each step of the testing procedure and on the uncertainty of the density of the immersion liquid, which depends of the temperature.

It was found that the error of the testing equipment used in the scope of this investigation to measure BD was ±0,002 g/cm^3 and error of the testing equipment to measure oPo was 0,01 vol.-%.

3.3 Fractional factorial design

The fractional factorial design method was used to evaluate the relative importance of various factors on the bulk density and open porosity testing results according to the two mentioned standards. The unshaped refractory MCC75 was chosen to study the standard EN ISO 1927-6, the dense shaped refractory HA75 was chosen to study the standard EN 993-1 for the dense shaped refractory (not reacting with water as immersion liquid) and the carbon bonded dense shaped refractory MaCarb was chosen to study the standard EN 993-1 for the carbon bonded dense shaped refractory (potentially reacting with water as immersion liquid).

For the standard EN ISO 1927-6 studied with the MCC75 unshaped refractory, a total of eight independent factors were identified. The independent factors and their levels are shown in Tab. 2. The selected levels of investigation are fixed at high (+) and low (–). The responses are the bulk density (BD) and the open porosity (oPo). The experimental design analysing the seven factors at two levels was composed of 16 runs (16 different experiments) and the experiments were replicated four times, i.e. for each run four samples were tested. Furthermore, the experimental design was replicated two times, the first time with water and the second time with Puretol as immersion liquid, i.e. each sample was first tested with water as immersion liquid and then with Puretol as immersion liquid.

For the standard EN 993-1 studied with the HA75 dense shaped refractory, a total of fifteen factors were identified. The independent factors and their levels are shown in Tab. 3. The selected levels of investigation are fixed at high (+) and low (–). The responses are the bulk density (BD) and the open porosity (oPo). The experimental design analysing the fourteen factors at two levels was composed of 16 runs (16 different experiments) and the experiments were again replicated four times. Furthermore, the experimental design was replicated two times, the first time with water and the second time with Puretol as immersion liquids like described before.

For the standard EN 993-1 studied with the MaCarb carbon bonded dense shaped refractory, a total of fourteen factors were identified. The independent factors and their levels are shown in Tab. 4. The selected levels of investigation are fixed at high (+) and low (–). The responses are the bulk density (BD) and the open porosity (oPo). The experimental design analysing the fourteen factors at two levels was composed of 16 runs (16 different experiments) and the experiments were replicated four times, i.e. for each run four samples were tested.

Tab. 4 Factors and levels used for the investigation of the standard EN 993-1 using the MaCarb carbon bonded dense shaped refractory

Factor	Label	Low Level (–)	High Level (+)
Operator	Op	Operator 1	Operator 2
Waiting time under vacuum before filling with the immersion liquid	Tvac [min]	15	30
Vacuum level	Vac [Pa]	2500	3500
Waiting time after filling with the immersion liquid under vacuum	Wp [min]	30	45
Waiting time after filling with the immersion liquid and under atmospheric pressure	Wwp [min]	30	45
Sponging material (cloth, towel)	Sp	Linen	Leather
Geometry of the samples	Geo	Prism	Cylinder
Temperature of the immersion liquid coming into contact with the sample	Temp. [°C]	15	30
Waiting time in a desiccator of the samples after having been dried at 110 °C in the drying chamber	Texi	Necessary time to reach the room temperature	24 h
Position of the samples in the airtight vessel	Pos	Under (1st level)	Up (2nd level)
How to wring the cloth/towel saturated by the immersion liquid	Wr	Wring the cloth/towel	Sin-dry with a roller
Ratio of the surface area to the volume of the test piece	S/V	1,32	0,8
Press direction of the samples	Press	Cut or drilled from bricks in the press direction	Cut or drilled from bricks perpendicular to the press direction
Place of extraction of the samples from bricks	Bp	Center of the brick	Side of the brick

Tab. 5 The Round Robin Test Factorial Design Matrix for the standard EN ISO 1927-6 studied with the MCC75 unshaped refractory

Run N°	For	Liq	Test	Wp
7	D	Paraffin	Cut	3 h
1	D	Paraffin	3-point	30 min
3	D	Water	Cut	30 min
8	D	Water	3-point	3 h
2	B	Water	3-point	30 min
4	B	Water	Cut	3 h
6	B	Paraffin	3-point	3 h
5	B	Paraffin	Cut	30 min

3.4 ANOVA analysis of the factorial design

Analysis of variance (ANOVA) method [8] is based on an analysis of the variation present in an experiment. It is a test of the hypothesis that the variation in an experiment is no greater than that due to normal variation of individual characteristics and error in the measurement. The tests in an ANOVA are based on the F-ratio: the variation due to an experimental effect divided by the variation due to experimental error. This ratio is equal to 1,0 if the experimental effect is the same as the experimental error. If the F-ratio is significantly large and the subsequently obtained p-value is lower than 0,05 then the factor is considered statistically significant. The statistical significance (p-value) of a factor represents a decreasing index of the reliability of a result. The higher the p-value, the less we can believe that the factor is statistically significant on the results.

Specifically, the p-value represents the probability of error that is involved in accepting the observed result as valid. Historically, a threshold value of 0,05 is often assumed to determine a significant effect of a factor on the testing results.

3.5 Round Robin Test

A Round Robin Test is an interlaboratory test (measurement, analysis or experiment) performed independently several times. In the scope of this investigation, four laboratories throughout Europe performed the same testing methods (measurement of the BD and the oPo on refractory materials according to EN ISO 1927-6 and EN 993-1) on the same materials but with different

Tab. 6 **The Round Robin Test Factorial Design Matrix for the standard EN 993-1 studied with the dense shaped refractory HA75**

Run N°	L/d	Sp	Tvac	Wwp
7	1,5	Leather	30 min	180 min
1	1,5	Leather	15 min	30 min
3	1,5	Linen	30 min	30 min
8	1,5	Linen	15 min	180 min
2	1	Linen	15 min	30 min
4	1	Linen	30 min	180 min
6	1	Leather	15 min	180 min
5	1	Leather	30 min	30 min

Tab. 7 **The Round Robin Test Factorial Design Matrix for the standard EN 993-1 studied with the carbon bonded dense shaped refractory MC95/10**

Run N°	Geo	Sp	Tvac	Vac
7	Cylinder CCS	Leather	30 min	3500 Pa
1	Cylinder CCS	Leather	15 min	2500 Pa
3	Cylinder CCS	Linen	30 min	2500 Pa
8	Cylinder CCS	Linen	15 min	3500 Pa
2	Prism MOR	Linen	15 min	2500 Pa
4	Prism MOR	Linen	30 min	3500 Pa
6	Prism MOR	Leather	15 min	3500 Pa
5	Prism MOR	Leather	30 min	2500 Pa

equipment. The aim of the Round Robin Test is to determine the reproducibility and the repeatability of the testing methods.

The repeatability is defined as the closeness of agreement between independent results obtained with the same method on identical test material under the same conditions (same operator, same apparatus, same laboratory and after short intervals of time). The reproducibility is defined as the closeness of agreement between independent results obtained with the same method on identical test material but under different conditions (different operators, different apparatus, different laboratories and/or after different intervals of time).

3.5.1 For the standard EN ISO 1927-6 studied with the MCC75 unshaped refractory

For the Round-Robin Test the samples were fired at 800 °C, as in the data sheet of Calderys the BD and the oPo are given after firing at 800 °C. The influence of the format ["For": (–) B; (+) D], of the immersion liquid ["Liq": (–) water; (+) Paraffin], of the method of the test pieces preparation ["Test": (–) three-point bending test; (+) cut in two-halves with a saw] and of waiting time with immersion liquid under vacuum ["Wp": (–) 30 min; (+) 3 h] on the BD and oPo results was analysed. Other parameters were kept at fixed levels ("Wwp" to 30 min, "Vac" to 2500 Pa, "Tvac" to 15 min and a linen towel was used as sponging material).

The four influencing factors quoted previously were tested in four different laboratories in order to determine the most significant factors influencing the BD and oPo testing results of unshaped refractory material and in order to obtain an estimation of the repeatability and of the reproducibility of the BD and oPo testing methods. For each run, each laboratory used four different samples and each sample was tested only once (Tab. 5).

3.5.2 For the standard EN 993-1 studied with the dense shaped refractory HA75

In the Round-Robin Test the influence of the ration of the longest to the shortest dimension of the samples ["L/d": (–) 1; (+) 1,5], of the kind of sponging material ["Sp": (–) linen; (+) leather], of the time of vacuum pressure ["Tvac": (–) 15 min; (+) 30 min] and of the time that the sample is immerged in the immersion liquid after the vacuum pressure has been removed ["Wwp": (–) 30 min; (+) 180 min] on the BD and oPo testing results was analysed.

Furthermore the effect of the position of the extraction of the samples from bricks ("Bp") was analysed. Thus, for each run of the factorial design, three samples were extracted from the border of a brick and for each run one sample was extracted from the middle of a brick. Other parameters were kept at fixed levels ("Vac" to 2500 Pa and "Wp" to 30 min). In order to reduce the standard deviation between the results of different laboratories, only water was used as immersion liquid for the tests. The four influencing factors quoted previously were tested in five different laboratories in order to determine the most significant factors influencing the BD and oPo testing results of dense shaped refractory material and to obtain an estimation of the repeatability and of the reproducibility of the testing methods and results. For each run, each laboratory used four different samples and each sample was tested only once (Tab. 6).

3.5.3 For the standard EN 993-1 studied with the carbon bonded dense shaped refractory MC95/10

In the Round-Robin Test the influence of the sample geometry ["Geo": (–) cylinder CCS; (+) prism MOR], of the kind of sponging material ["Sp": (–) linen; (+) leather], of the time of vacuum pressure ["Tvac": (–) 15 min; (+) 30 min] and of the vacuum pressure ["Vac": (–) 2500 Pa; (+) 3500 Pa] on the BD and oPo testing results was analysed. Accordingly, two sample geometries were used: the prism MOR with the dimensions 150 mm × 25 mm × 25 mm and the cylinder CCS with a diameter of 50 mm and a height of 50 mm.

Furthermore the effect of the position of the extraction of the samples from bricks ("Bp") was analysed. Thus, for each run of the factorial design, three samples were extracted from the side of a brick and for each run one sample was extracted from the centre of a brick. Other parameters were kept at fixed levels (paraffin as immersion liquid "Liq", "Wp" to 30 min and "Wwp" to 30 min).

The four influencing factors quoted previously were tested in four different laboratories in order to determine the most significant factors influencing the BD and oPo testing results of carbon bonded dense shaped refractory material and in to obtain an estimation of the repeatability and of the reproducibility of the testing methods and results. For each run, each laboratory used four different samples and each sample was tested only once (Tab. 7).

4 Results

4.1 Fractional factorial design

4.1.1 Results for the standard EN ISO 1927-6 studied with the MCC unshaped refractory

The eight factors shown in Tab. 2 were examined using a fractional factorial design. The main effects (effect of a single independent variable on a dependent variable – ignoring all other independent variables.) F-ratios and the p-values were calculated from the BD and oPo testing results using the statistical software OptiVAL, version V.3.3.2.4, from the company Quodata.

A p-value of less than 0,05 for four factors "For", "Liq", "Sp" and "Vac" indicated that these factors are statistically significant for the testing results of bulk density and open porosity testing.

The influence of the format ("For") of the samples on the testing results was found to be significant: A higher bulk density result was obtained with the format B than with the format D and a lower open porosity result was obtained with the format B compared to the format D. Furthermore, results from testing with format B showed higher precision compared to the results from testing with format D. For each geometry B and D, a lower bulk density result was found with Puretol than with water as immersion liquid ("Liq"). Regarding the sponging material ("Sp"), using a linen towel lead to better reproducibility of the testing results when it was saturated with a liquid compared to any other kind of towel. Finally, the vacuum pressure ("Vac") was found to be an influencing factor on the testing results and a higher precision of the testing results was obtained with a vacuum pressure of 2500 Pa compared to 3500 Pa.

4.1.2 Results for the standard EN 993-1 studied with the dense shaped refractory HA75

The fifteen factors shown in Tab. 3 were examined using a fractional factorial design. The main effects (effect of a single independent variable on a dependent variable – ignoring all other independent variables.), F-ratios and the p-values were calculated for the BD and oPo testing results as described before. A p-value of less than 0,05 for four factors "Bp", "Liq", Geo" and "Wwp" indicated that these factors are statistically significant for the BD testing results. Likewise, the factors "Bp", "Wwp", "Tvac" and "Vol" were found to be statistically significant for the oPo testing results.

The influence of the place of extraction of the samples (internal or external part of the brick, ("Bp"] on the BD testing results was found to be significant. A higher BD result and lower oPo result were obtained when the sample had been extracted from the middle of a brick. Furthermore lower BD testing results were obtained with Puretol than with water as immersion liquid "Liq". The BD testing result were also found to be higher for a small sample geometry "Geo". Moreover the waiting time with the immersion liquid and under atmospheric pressure "Wwp" increased the BD testing results and their standard deviation and decreased the oPo results when the waiting time was increased.

4.1.3 Results for the standard EN 993-1 studied with the carbon bonded dense shaped refractory MaCarb

The fourteen factors shown in Tab. 4 were examined using a fractional factorial design. The main effects (effect of a single independent variable on a dependent variable – ignoring all other independent variables.), F-ratios and the p-values were calculated for the BD and oPo testing results as described before. A p-value of less than 0,05 for four factors "Press", "Bp", S/V" and "Tvac" indicated that these factors are statistically significant for the BD testing results. Likewise, the factors "Press", "Bp", S/V", "Tvac" and "Vac" were found to be significant for the oPo testing results.

The influence of the place of extraction of the sample (internal or external part of the brick, "Bp") on the BD testing results was found to be significant. Higher BD results were obtained when the sample had been extracted from the middle of a brick. Moreover the BD testing result was found to increase when the sample had been extracted perpendicular to the press direction ("Press"). Furthermore lower oPo results were obtained when a longer waiting time under vacuum before filling with the immersion liquid ("Tvac") had been used.

4.2 Round Robin Test

4.2.1 Results for the standard EN ISO 1927-6 studied with the MCC75 unshaped refractory

The BD and oPo testing results from each of five laboratories were collected in one factorial design and were again analysed using the software OptiVAL. For each run of the factorial design, testing results from testing of twenty samples were analysed (four from each of the five laboratories). From the analyses it was found that:

- A general deviation of the testing results between the laboratories was present due to a problem of sample preparation.
- The factor "For" showed a significant influence on the testing results due to the sample preparation. The format B was chosen, as testing results showed better reproducibility.
- The factor "Liq" showed a statistically significant influence on the testing results. For this unshaped refractory, paraffin increased the reproducibility of the testing results.
- The factor "Test" showed a statistically significant influence on the testing results. Using samples that had previously been tested in a three-point-bending test, BD and oPo results had better repeatability compared to using samples that had been cut in two halves with a saw.
- The factor "Wp" was not found to be significant. Thus a waiting time of vacuum pressure with the immersion liquid of 30 min is deemed enough.

Utilising the software PROLab from Quodata, a statistical analysis of the round-robin test was carried out. According to the statistical analyses, the combination of factors in run number 2 (Tab. 5) gives the best reproducibility and repeatability of the BD and oPo testing methods. That is when samples of the format B after having previously been tested in a three point bending test are used, the samples are immersed in water as immersion liquid and are kept only 30 min under vacuum pressure immersed in water. A reproducibility of 0,05 g/cm^3 and a

Tab. 8 Comparison of the results and the standard deviation of the results for the BD and the oPo testing from samples extracted from the side or from the centre of dense shaped refractory HA75 bricks (SD = standard deviation)

	BD [g/cm³]	BD SD [g/cm³]	oPo [vol.-%]	oPo SD [vol.-%]
All samples	2,73	0,02	20,2	0,5
From the side	2,73	0,02	20,3	0,5
From the centre	2,74	0,01	20,0	0,3

Tab. 9 Comparison of the results and the standard deviation of the results for the BD and the oPo from samples extracted from the side or from the centre of the carbon bonded dense shaped refractory MC95/10 brick (SD = standard deviation)

	BD [g/cm³]	BD SD [g/cm³]	oPo [vol.-%]	oPo SD [vol.-%]
All samples	3,04	0,02	3,7	0,4
From the side	3,04	0,02	3,7	0,5
From the centre	3,05	0,01	3,6	0,2

repeatability of 0,03 g/cm³ for the BD and a reproducibility of 1,4 vol.-% and a repeatability of 0,9 vol.-% for the oPo can then be obtained.

4.2.2 Results for the standard EN 993-1 studied with the dense shaped refractory HA75

First, in order to determine only the error due to the factors by avoiding the addition of the error due to the inhomogeneity of the material, the same samples were tested for each run of the factorial design (Tab. 6) in one laboratory (at Forschungsgemeinschaft Feuerfest e.V. in Germany). Four samples with the ratio L/d = 1 and four samples with the ratio L/d = 1,5 were tested. As results of these two factorial designs it was noted that the maximum standard deviation of the BD testing result from one sample is 0,003 g/cm³ and the maximum standard deviation of the oPo testing result is 0,1 vol.-%, which was considered to be very low.

The testing results of five laboratories were then collected in one factorial design and analysed using the software OptiVAL as described before. For each run of the factorial design, testing results from testing of twenty five samples were analysed (four from each laboratory). From the analyses it was found that:

- A general deviation of the testing results between the laboratories was present due to the use of different testing equipment.
- The factor "Sp" was found not to significantly affect the testing results but a worse repeatability was observed when the leather towel was used. Thus, the use of the linen towel as sponging material is recommended.
- The factor "Tvac" was found not to significantly affect the testing results. Thus a waiting time of 15 min is deemed enough.
- The factor "L/d" was found not to significantly affect the testing results.
- The factor "Wwp" was found not to significantly affect the testing results. So a waiting time of the sample in immersion in liquid under atmospheric pressure of at least 30 min is deemed enough.

Another calculation was made to analyse the influence of the position where samples are extracted from a brick (results obtained from samples extracted from the side of a brick and results obtained from samples extracted from the centre of a brick, Tab. 8). The results showed that the standard deviation is lower if the tested samples are extracted from the centre of a brick. The BD was found to be higher and the oPo lower when obtained from samples from the centre of a brick. Utilising the software PROLab from Quodata, a statistical analysis of the round-robin test was carried out. It was noted that there were only small differences in the results between all runs (Tab. 6) for the reproducibility and the repeatability of the testing methods according this study. Thus, for a practical issue, the combination of factors as used for the run number 2 can be recommended. That is when samples with a ratio L/d of 1 are being used, the waiting time under pressure without liquid is being fixed to 15 min, the samples are kept at least 30 min immersed in water under atmospheric pressure, and the excess water is removed using a saturated linen towel. A reproducibility of 0,07 g/cm³ and a repeatability of 0,04 g/cm³ for the BD and a reproducibility of 1,6 vol.-% and a repeatability of 1,2 vol.-% for the oPo can then be obtained.

4.2.3 Results for the standard EN 993-1 studied with the carbon bonded dense shaped refractory MC95/10

The testing results of five laboratories were collected in one factorial design and analysed using the software OptiVAL as described before. For each run of the factorial design, results from testing of twenty samples were analysed (four from each laboratory). From the analyses it was found that:

- A general deviation of the testing results between the laboratories was present due to the use of different testing equipment.
- The factor "Sp" was found not to significantly affect the BD and oPo testing results, but an increase of the standard deviation was observed when the leather towel was used. The use of the linen towel as sponging material is thus deemed more suitable.
- The factor "Tvac" was found not to significantly affect the testing results. Thus a waiting time of 15 min is considered enough.
- The factor "Geo" was found not to significantly affect the testing results.
- The factor "Vac" was found to statistically significantly influence the standard deviation of the testing results. A higher vacuum pressure of 2500 Pa is recommended, as it is believed to allow a better filling of micropores.

Another calculation was made to analyse the influence of the position where samples are extracted from a brick (results obtained from samples extracted from the side of a brick and results obtained from samples extracted from the centre of a brick (Tab. 9).

The results showed that the standard deviation is apparently lower if the tested samples are extracted from the centre of a brick. The BD was found to be slightly higher and the oPo slightly lower when obtained from samples from the centre of a brick.

Utilising the software PROLab from Quodata, a statistical analysis of the Round Robin Test was carried out. Only small differences in

Tab. 10 Summary of the reproducibility and repeatability obtained for both standards EN ISO 1927-6 for unshaped refractory and EN 993-1 for dense shaped refractory

		Reproducibility		Repeatability	
		Best Combination	Worst Combination	Best Combination	Worst Combination
Standard EN ISO 1927-6 using the unshaped refractory MCC75	BD [g/cm³]	0,05	0,15	0,03	0,03
	oPo [vol.-%]	1,4	3,9	0,9	1,2
Standard EN 993-1 using the dense shaped refractory HA75	BD [g/cm³]	0,07	0,07	0,04	0,05
	oPo [vol.-%]	1,6	1,9	1,2	1,3
Standard EN 993-1 using the carbon bonded dense shaped refractory MC95/10	BD [g/cm³]	0,04	0,08	0,04	0,06
	oPo [vol.-%]	0,6	1,8	0,5	1,1

the results between all runs (Tab. 7) for the reproducibility and the repeatability of the testing methods according the study were noted. Thus for a practical issue the combination of factors as used in the run number 2 can be recommended. That is when CCS cylinders are being used, the waiting time under vacuum pressure without immersion liquid is fixed to 15 min, the samples are under 2500 Pa vacuum pressure, and to remove the excess liquid of the samples a saturated linen towel is being used. A reproducibility of 0,04 g/cm³ and a repeatability of 0,04 g/cm³ for the BD and a reproducibility of 0,6 vol.-% and a repeatability of 0,5 vol.-% for the oPo can be obtained in this way.

5 Discussion

Recommendations can be given to achieve high reproducibility and high repeatability of the BD and oPo testing methods concerning refractory materials.

Generally for both testing standards involved, it can be applied that:

- The time waited under vacuum before filling with the immersion liquid should be at least 15 min.
- The waiting time with the immersion liquid under vacuum should be 30 min.
- The waiting time with the immersion liquid and under atmospheric pressure should be at least 30 min.
- The vacuum pressure should be set to 2500 Pa. A lower vacuum pressure will increase the standard deviation of the testing results between results obtained in

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different laboratories. If water is used as immersion liquid, its temperature should be held below and/or equal to 20 °C in order to attain the maximum vacuum pressure of 2500 Pa. It is recommended to always wait until the water boils to ensure the maximum vacuum is attained for a given temperature.

- The time that a sample should be placed in a desiccator after having been dried at 110 °C in order to cool down ("waiting time", between 2 h and 24 h) showed no significant impact on the testing results. The current testing standards suggest placing the test piece, for instance, in a desiccator until it has cooled to room temperature. As no significant impact of the waiting time on the testing results were found, in order to save time, the samples can be put in the desiccator at the end of the day before being tested the next morning for example.
- The position of the samples in the airtight vessel showed no significant impact on the testing results. The whole vessel with several layers of samples can be used to study the BD and the oPo without significant effect on the testing results.
- A linen towel should be chosen as sponging material as using a leather towel resulted – in some laboratories – in a higher standard deviation of the oPo and BD testing results. One main reason is thought to be that the leather towel showed some difficulties to store a constant level of moisture.

Further recommendations are derived from the investigations when using the standard EN ISO 1927-6 with unshaped refractories:

- The format B needs to be privileged for this test as problems with sample preparation of the smaller format D influences the BD and oPo testing results.
- The immersion liquid can be water for samples fired at sufficient high temperature in order to reduce the standard deviation of testing results. The use of paraffin as immersion liquid is often uncomfortable and more expensive. Furthermore, commercial paraffins may show variations in viscosity, leading to different degrees of infiltration of the material when tested in different laboratories, which leads to a higher standard deviation of the testing results between the laboratories.
- The castable bars B can be used after having been tested by the modulus of rupture test before being tested for BD and oPo. For samples simply prepared by cutting with a saw, a lower reproducibility in the BD and oPo results was achieved.

A number of recommendations are derived from the investigations when using the standard EN 993-1 with dense shaped refractories:

- Cylinders with a format of 50 mm × 50 mm can be chosen to be first tested for the BD and oPo then secondly tested for cold crushing strength.
- Despite a lower standard deviation was found if the samples are extracted from the centre of the brick, it is not possible to favour the position of extraction of the samples. That is why the samples should be extracted randomly from bricks. Furthermore the side or the centre of the brick shall not be privileged, as an inhomogeneity between both locations may exist due to the pressing process of bricks.
- Whenever possible, the immersion liquid should be water in order to reduce the standard deviation between results from different laboratories.

Further recommendations are derived from the investigations when using the standard EN 993-1 with carbon bonded dense shaped refractories:

- Cylinders with a format of 50 mm × 50 mm can be chosen as this is the standard geometry for cold crushing strength.
- Despite a lower standard deviation was found if the samples are extracted from the centre of the brick, it is not possible to favour the position of extraction of the samples. That is why the samples should be extracted randomly from bricks. Furthermore the side or the centre of the brick shall not be privileged, as an inhomogeneity between both locations may exist due to the pressing process of bricks.

6 Conclusion

Factorial designs for the two testing standards (EN ISO 1927-6 for unshaped refractory and EN 993-1 for dense shaped refractory) were effectively applied for the screening of the factors that influence the results of the BD and the oPo testing results on three different refractories (Tab. 1). A second design of experiment, which contained the four most significant factors was studied for the two testing standards in four or five different laboratories to evaluate precisely the effects and possible interactions of the influencing parameters/factors and estimate the repeatability and the reproducibility of those standards, which are summarised in the Tab. 10.

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