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Characterization data of reference cement CEM I 42.5 R used for Priority Program DFG SPP 2005 “Opus Fluidum Futurum - Rheology of reactive, multiscale, multiphase construction materials”

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Article Title

Title: Characterization data of reference cement CEM I 42.5 R used for Priority Program DFG SPP 2005 "Opus Fluidum Futurum - Rheology of reactive, multiscale, multiphase construction materials"

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Abstract

A thorough characterization of starting materials is the precondition for further research, especially for cement, which contains various phases and presents quite a complex material for fundamental scientific investigation. In the paper at hand, the characterization data of the reference cement CEM I 42.5 R used within the priority program 2005 of the German Research Foundation (DFG SPP 2005) are presented from the aspects of chemical and mineralogical compositions as well as physical and chemical properties. The data were collected based on tests conducted by nine research groups involved in this cooperative program. For all data received, the mean values and the corresponding errors were calculated. The results shall be used for the ongoing research within the priority program.

Keywords: Portland cement; Characterization; DFG SPP 2005

Specifications Table

Subject area	Ceramics and Composites
More specific subject area	Building materials; Cement
Type of data	Table; Image; Graph; Figure

How data was acquired	XRD; SEM; EN 196-1: 2016; EN 196-2: 2013; EN 196-3: 2016; EN 196-6: 2018; EN 196-11: 2018; EN 1097-7: 2008; ISO 13320: 2009; ISO 9277: 2010
Data format	Raw; Analyzed
Parameters for data collection	Chemical composition; Phase contents; Density; Specific surface area; Particle size; Calorimetry; Water demand; Setting time; Mechanical strength
Description of data collection	Firstly a thorough characterization on CEM I 42.5 R sample was made by in total 9 research groups. Then the data were collected and compared in this paper. Furthermore, the mean values and the corresponding errors were calculated based on the collective data.
Data source location	Seven universities, one research institute, and one company as shown in Table1
Data accessibility	The data is included in this article
Related research article	The data presented here, as a result, will be cited by the upcoming research articles by members of DFG SPP 2005 in which the CEM I 42.5 R is used

Value of the Data

- The aim was to characterize the CEM I 42.5 R sample as the basis for further research in the DFG SPP 2005 priority program.
- A common characterization test by seven universities, one research institute and one company was conducted to collect data for the CEM I 42.5 R sample.
- For all properties, the mean values and the corresponding errors were calculated based on the collective data from the test results.
- The data present the basis for other research work involved in the DFG SPP 2005 priority program.
- All different research groups involved in the DFG SPP 2005 priority program can use these data for their research work.

1. Data

Table 1 lists the universities, research institute, and cement company involved in the characterization of the CEM I 42.5 R and the abbreviations are explained, respectively. Figure 1 shows selected SEM pictures of cement particles with different magnifications.

Table 1 Universities, research institute and the company involved in the characterization

Acronym	Affiliation
BAM	Bundesanstalt für Materialforschung und -prüfung
BUW	Bauhaus-Universität Weimar
FAU	Friedrich-Alexander Universität Erlangen-Nürnberg
Heidelberg	HeidelbergCement AG
KIT	Karlsruher Institut für Technologie
TUB	Technische Universität Berlin
TUBS	Technische Universität Braunschweig
TUDD	Technische Universität Dresden

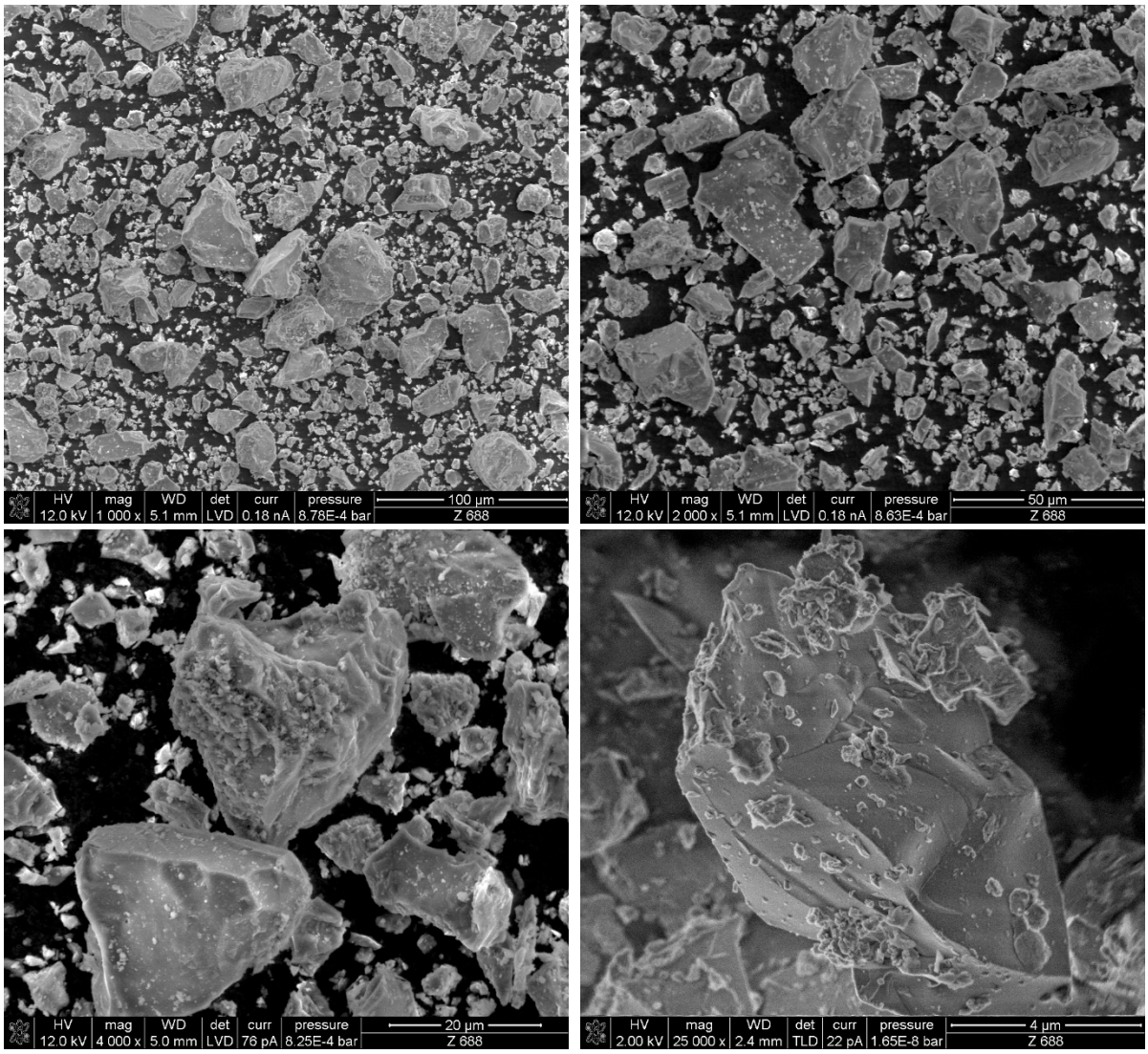


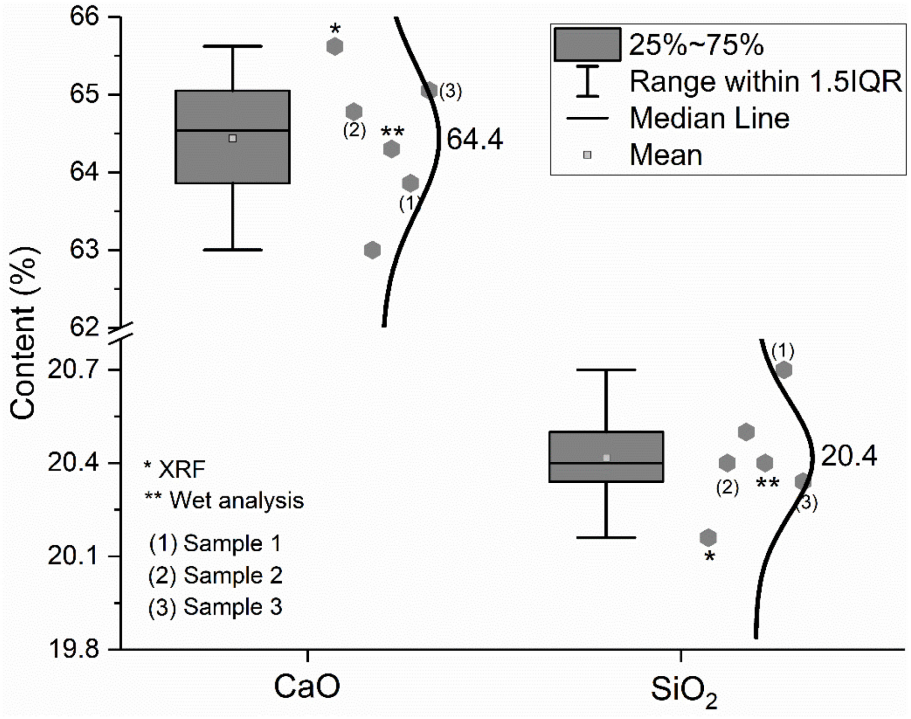
Fig. 1. SEM pictures of CEM I 42.5 R with different magnifications.

1.1 Characterization data of oxide composition and phase contents

In Figure 2 the oxide composition (CaO, SiO_2, Al_2O_3, Fe_2O_3, SO_3, MgO, K_2O, Na_2O, TiO_2 and P_2O_5), insoluble residue as well as the loss on ignition (LOI) of CEM I 42.5 R measured by the different participating groups according to EN 196-2: 2013 [1] are shown. It should be mentioned that the data denominated as (1) to (3) were measured by one research group from one single batch but different bags. In Figure 2(b) SO_3* means the value obtained by the X-ray fluorescence analysis (XRF) and SO_3** indicates the value captured by the wet chemistry method. The same meanings of * and ** are also suitable for the other data shown in Figure 2. Unless otherwise stated, the oxide composition shown in Figure 2 is measured based on XRF analysis. Furthermore, due to the quite low content of Cl^- of 0.02 wt.% only, the amount of Cl^- is not included in Figure 2.

In the legend of the figures of this paper, IQR means the interquartile range, namely the range between 25^{th} and 75^{th} percentiles (as shown in the area in the grey box). The specific explanation could be found on the website [2]. The error bar shows the range within 1.5 times of IQR. The median line indicates

the 50th percentile and the mean value is calculate based on data from all the samples within the 1.5 IQR range and does not include outliers.



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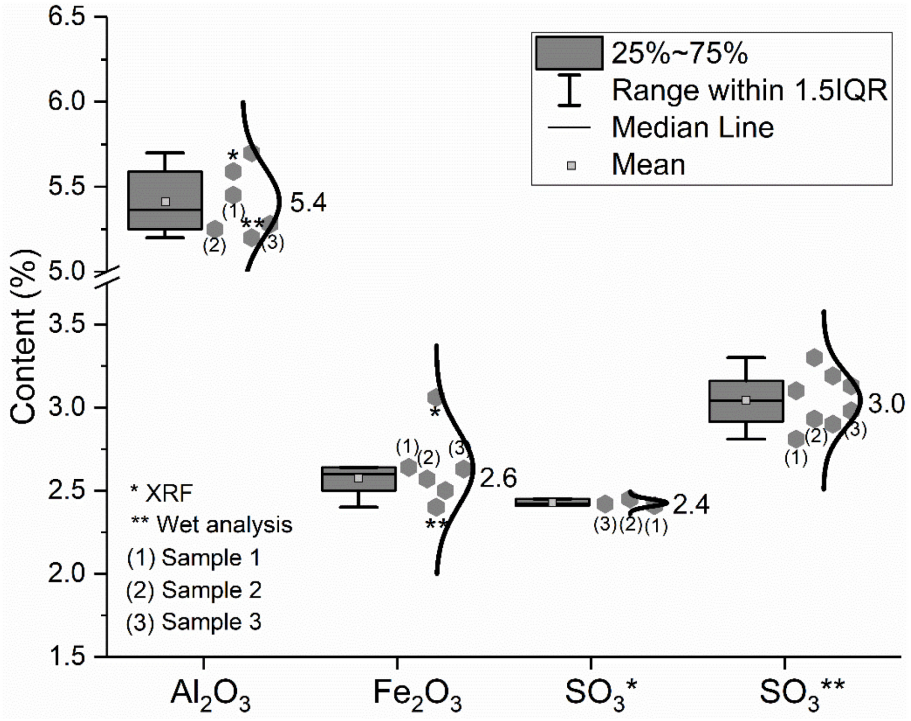

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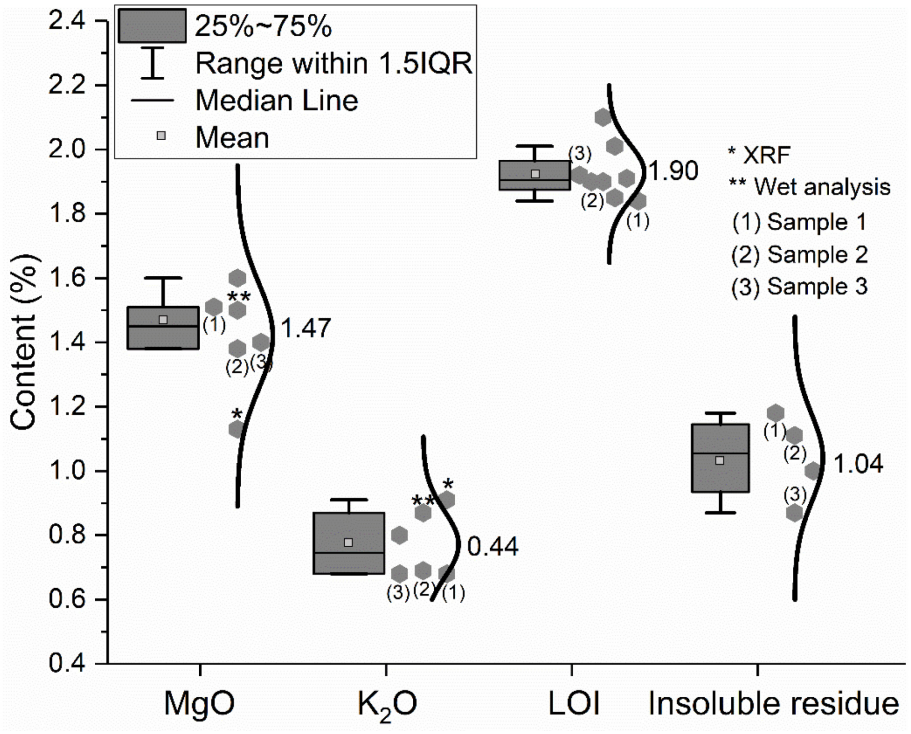

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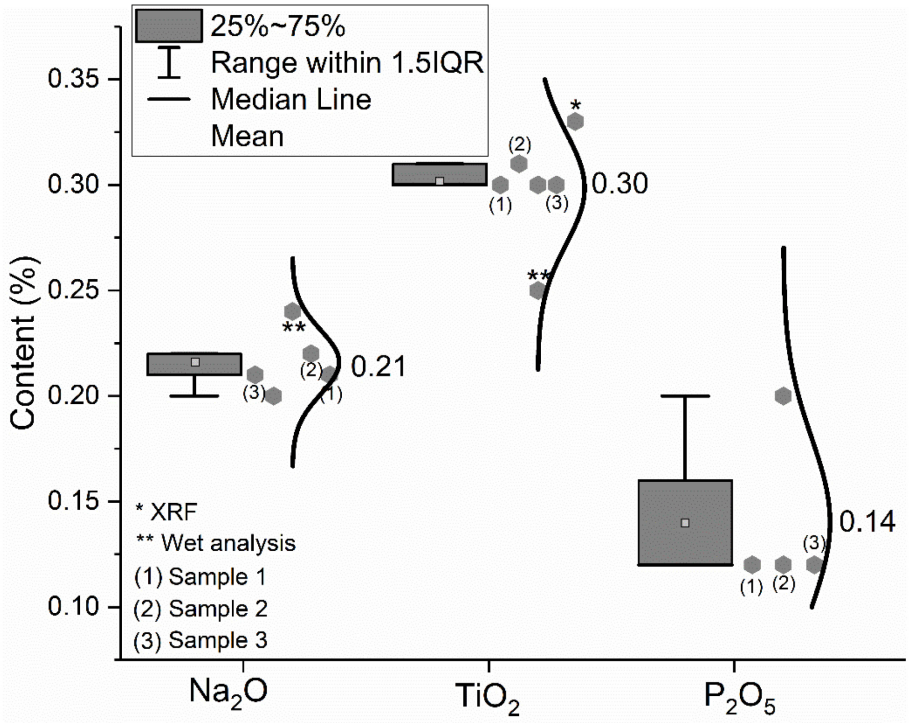
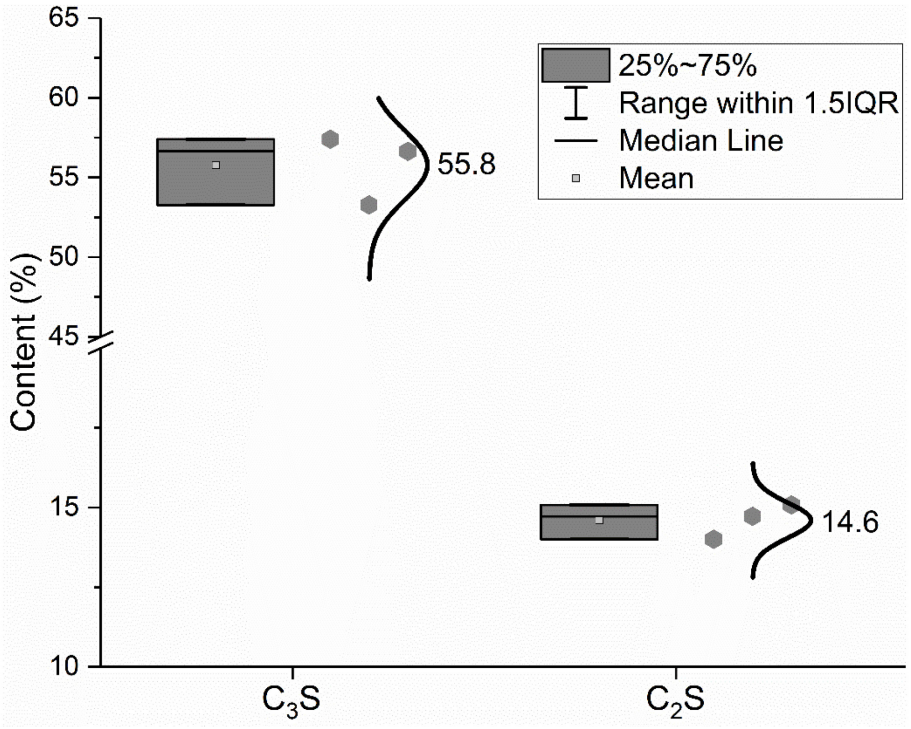


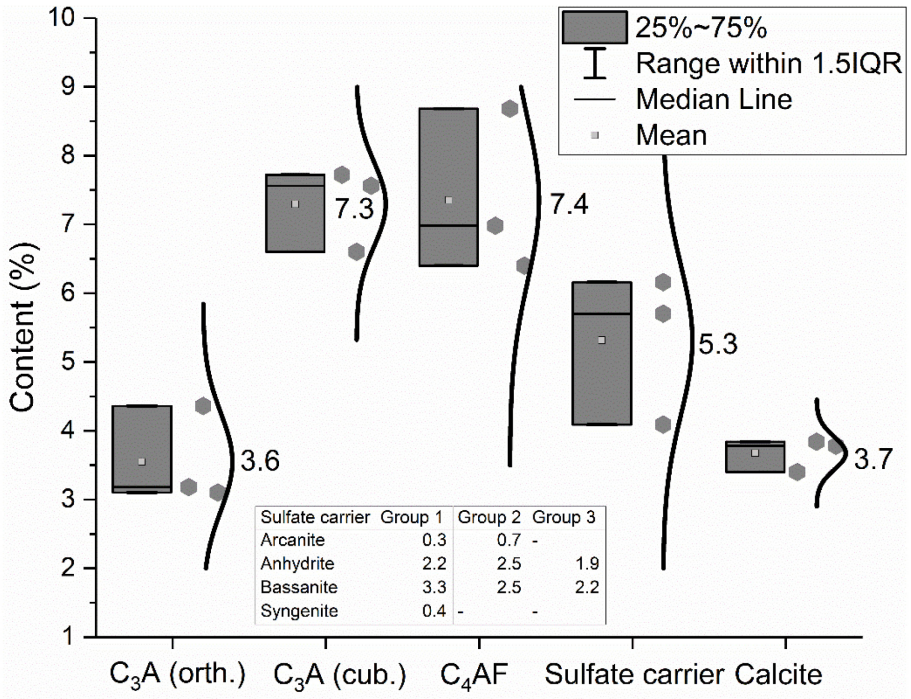
(d)

Fig. 2. Oxide composition of CEM I 42.5 R; (a) CaO and SiO_2; (b) Al_2O_3, Fe_2O_3 and SO_3; (c) MgO, K_2O, loss on ignition and insoluble residue; (d) Na_2O, TiO_2 and P_2O_5.

Figure 3 shows the phase contents of CEM I 42.5 R based on the results from three different groups through the method of powder-XRD combined with quantification of the patterns according to the Rietveld refinement method [3].



(a)



(b)

Fig. 3. Phase contents in CEM I 42.5 R; (a) C_3S and C_2S; (b) C_3A, C_4AF, sulfate carrier and calcite.

1.2 Characterization data of physical properties

The ture density of the CEM I 42.5 R was measured by Helium pycnometer method according to standard EN 1097-7: 2008 [4]. Results are shown in Figure 4. The same experiment was conducted by different groups, as shown by the hexagon, and then the mean value was calculated.

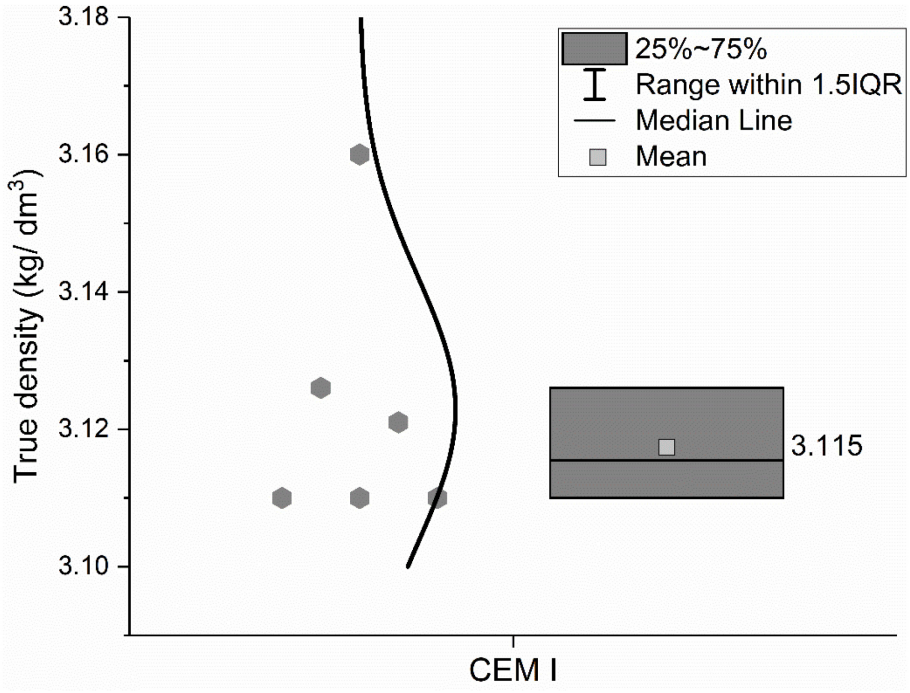


Fig. 4. True density of CEM I 42.5 R.

The specific surface area of the CEM I 42.5 R was measured by the Blaine method according to EN 196-6: 2018 [5] and the results are shown in Figure 5.

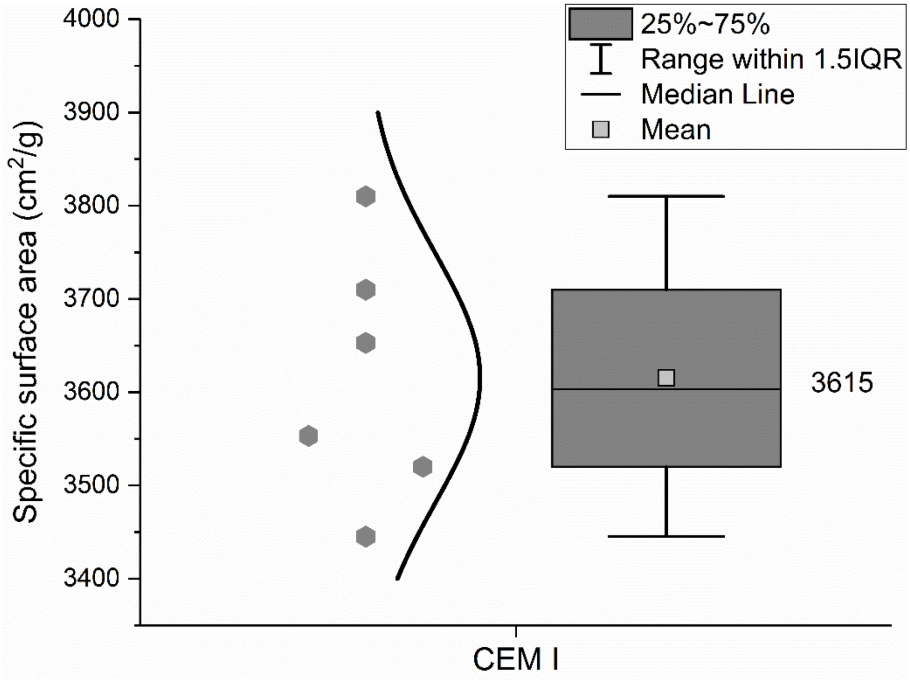


Fig. 5. Specific surface area of CEM I 42.5 R measured by the Blaine method.

The specific surface area of the CEM I 42.5 R was measured by the BET method as well according to ISO 9277: 2010 [6]. Results are shown in Figure 6. The numbers in brackets indicate the values from the same sample but different pre-treatment methods that were conducted by one same group.

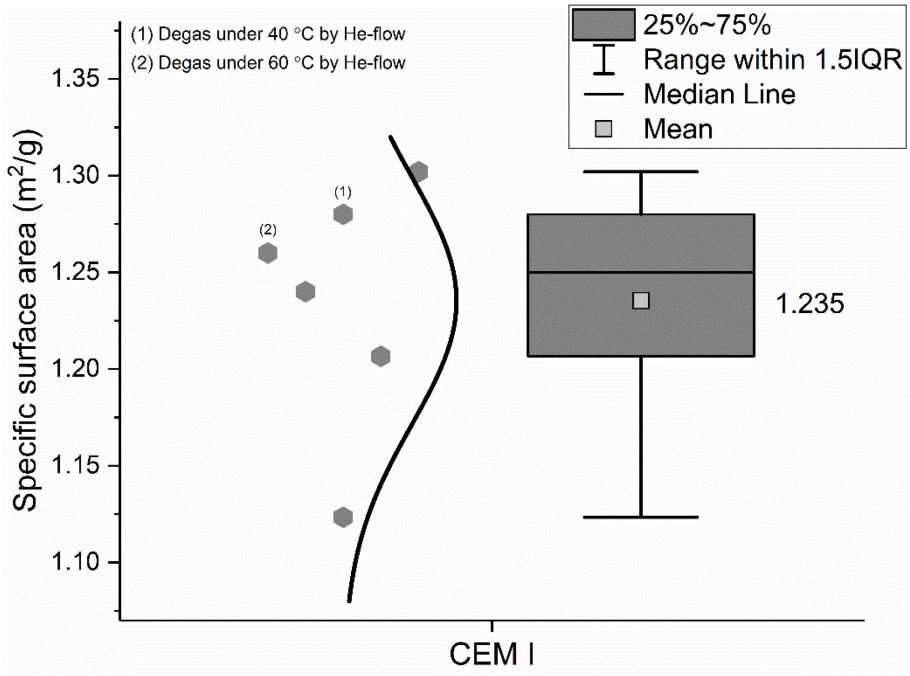
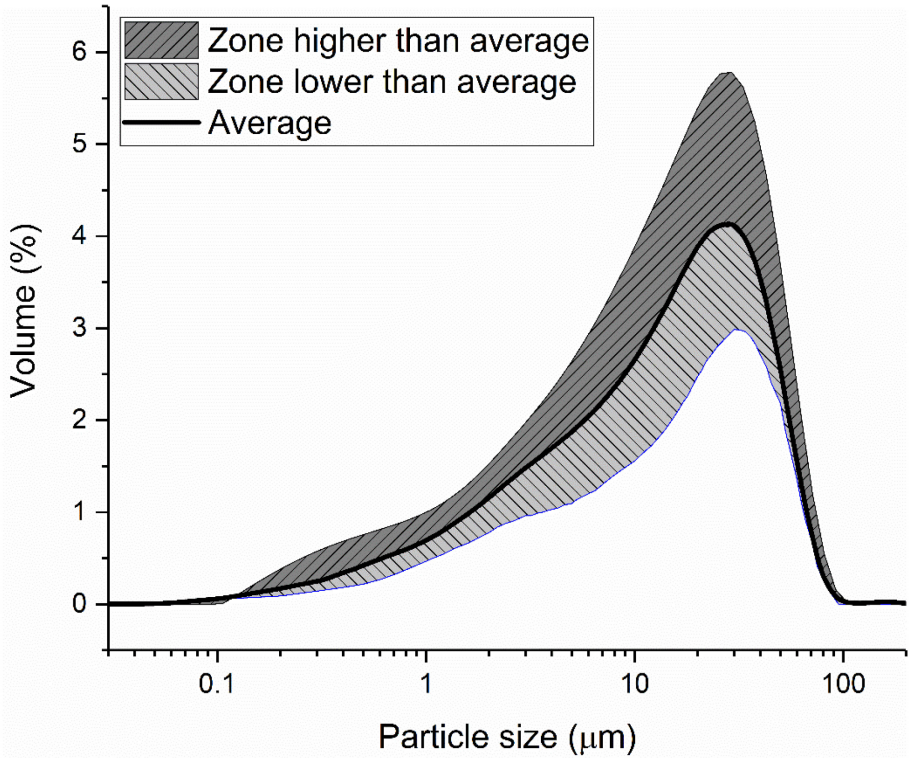
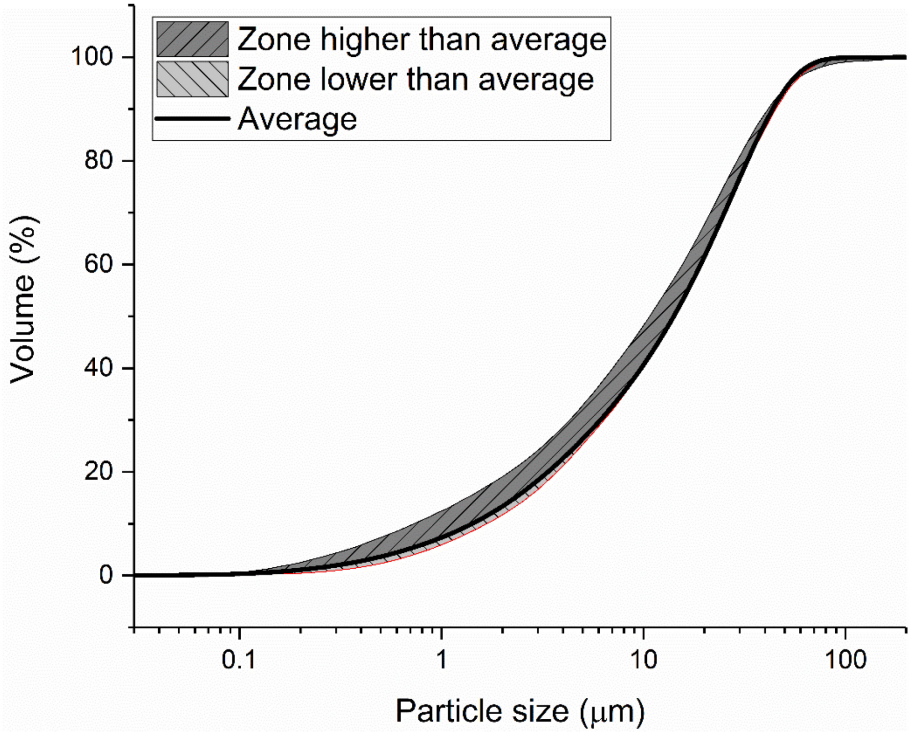


Fig. 6. Specific surface area of CEM I 42.5 R measured by the BET method.

Laser diffraction was applied to measure the particle size distribution (PSD) of the cement by eight different groups according to the method described in ISO 13320: 2009 [7]. The average distribution line was calculated, as shown in Figure 7. The shadow areas below and above this average line indicate the scope of the testing results. The characterized particle size distributions of the cement (d(0.1), d(0.5) and d(0.9)) are shown in Figure 8.



(a)



(b)

Fig. 7. Particle size and distribution of CEM I 42.5 R measured by laser diffraction method; (a) differential curve; (b) Integration curve.

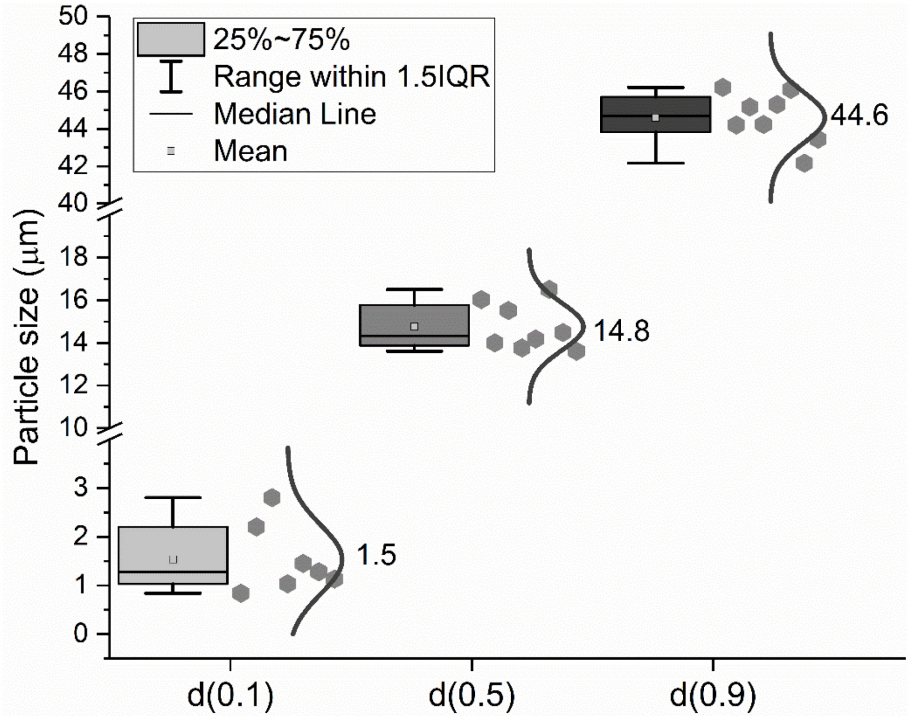


Fig. 8. Particle size distribution of CEM I 42.5 R at d(0.1), d(0.5) and d(0.9).

1.3 Characterization data of other properties

Water demand, as well as initial and final setting time were measured according to the standard EN 196-3: 2016 [8]. Flexural and compressive strength were measured according to the standard EN 196-1: 2016 [9]. The results are shown in Figures 9-11.

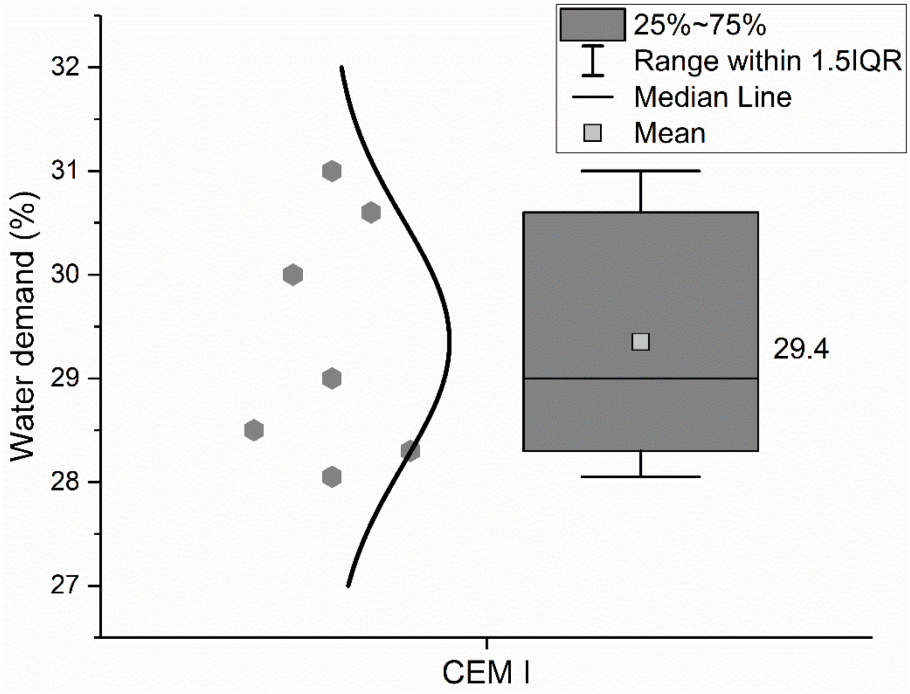


Fig. 9. Water demand of CEM I 42.5 R.

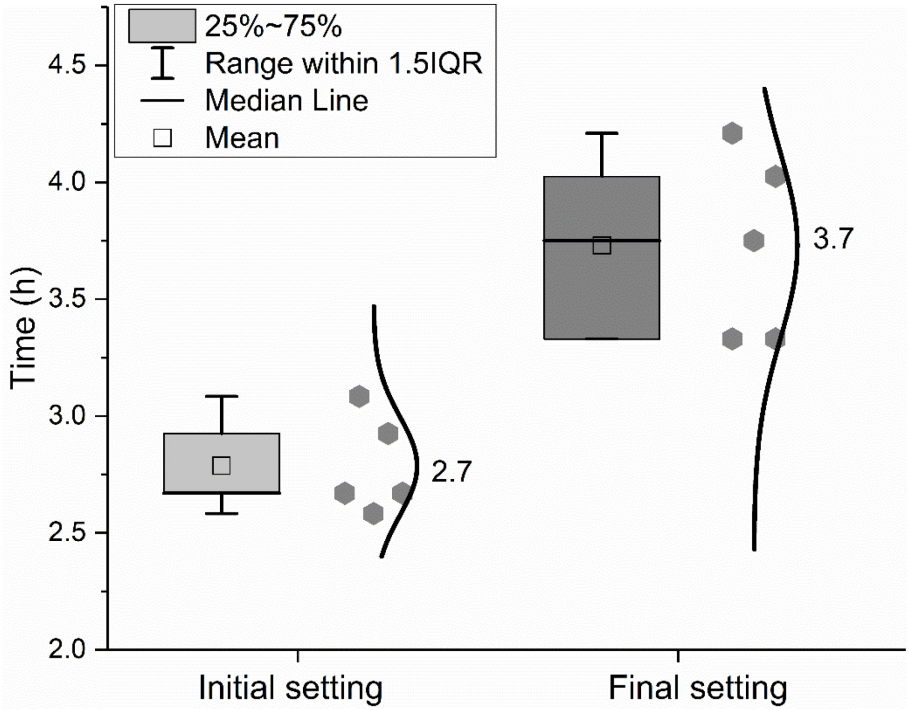
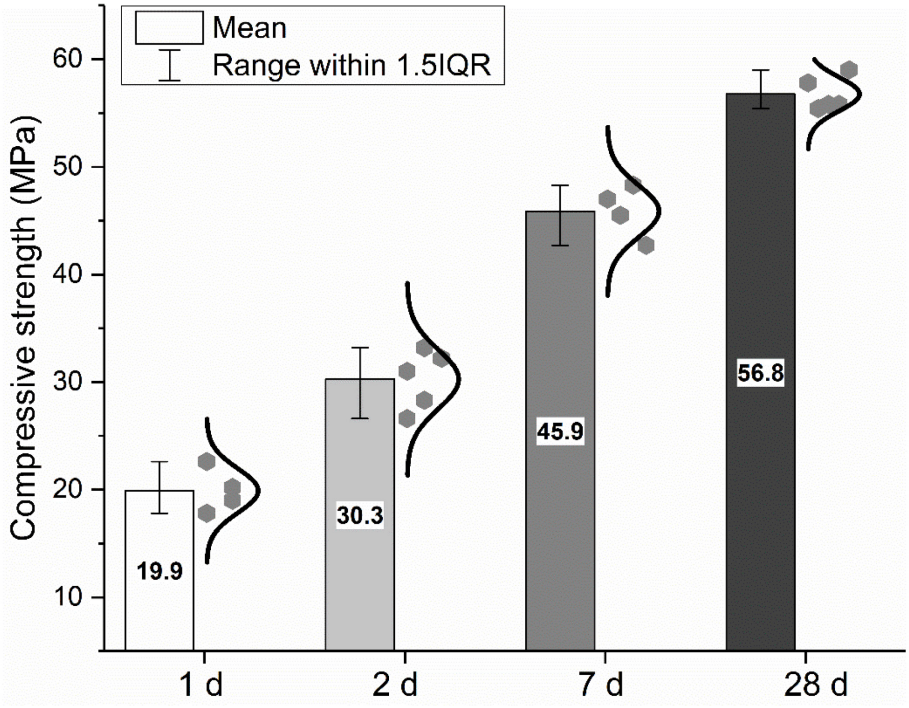
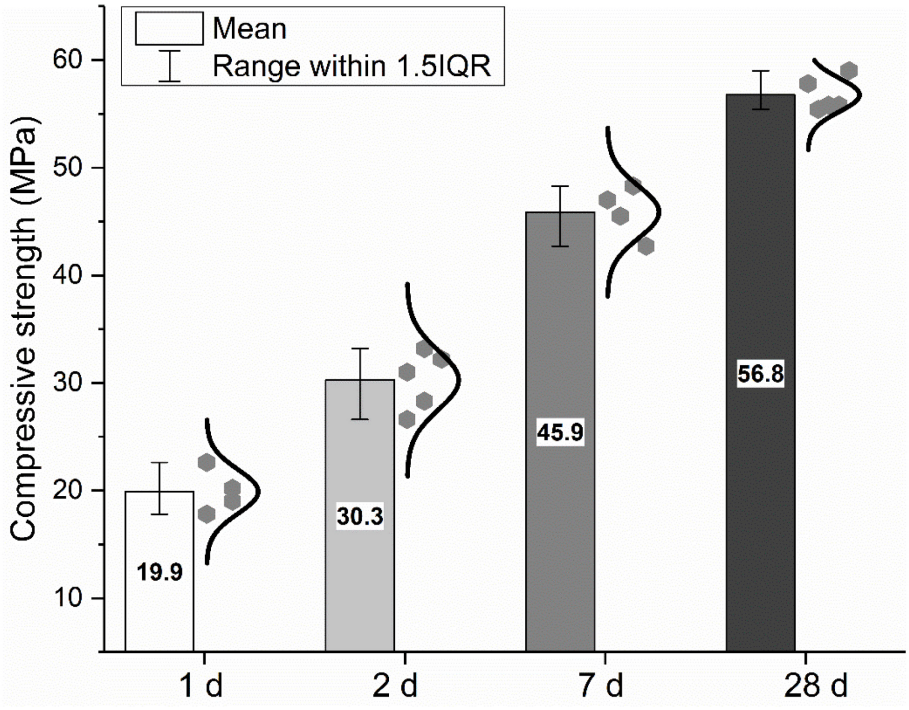


Fig. 10. Initial and final setting time of CEM I 42.5 R.



(a)



(b)

Fig. 11. Mechanical strength of hardened cement mortars after curing for certain time; (a) Compressive strength; (b) Flexural strength.

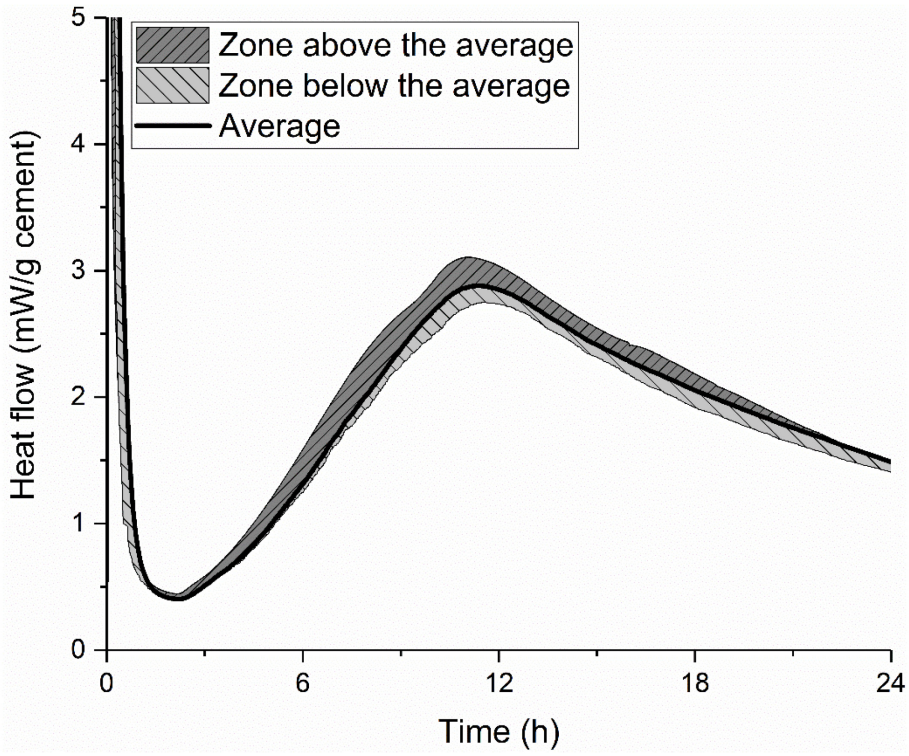


Fig. 12. Calorimetry curve of cement paste with water to cement ratio of 0.434 at the temperature of 20 °C.

The cement hydration with a water to cement ratio of 0.434 at the temperature of 20 °C was characterized independently by three different groups according to the method described in EN 196-11: 2018 [10]. The results are shown in Figure 12. The shadow areas below and above the average line indicate the scope of the test results.

2. Experimental Design, Materials, and Methods

All samples analyzed in this campaign stemmed from the same batch of cement production. The sample amount delivered to the different research groups were between a few kilograms up to several tons. The material was stored in closed containers, and the various groups took a representative sample from their own sub-batch.

For the characterizations of the CEM I 42.5 R, EN 196-2: 2013 was applied for the assessment of the oxide composition, insoluble residue and loss on ignition. Density was measured according to EN 1097-7: 2008; specific surface area by the Blaine method was measured according to EN 196-6: 2018 and by BET based on ISO 9277: 2010. Water demand and setting times were tested based on EN 196-3: 2016; flexural and compressive strength were obtained following EN 196-1: 2016. Isothermal heat flow calorimetry was measured according to EN 196-11: 2018. Particle size distribution was evaluated based on ISO 13320: 2009. For the other characterization methods of the CEM I 42.5 R, the specific experiment design and methods are explicated below.

SEM images were recorded on uncoated cement powder with a Nova NanoSEM 230 (FEI, Netherlands) equipped with a field-emission gun (Shottky emitter). For lower magnification, a low-vacuum-detector (LVD) applying 12 kV acceleration voltage and 0.9 mbar was used. For higher magnification, a through the lens detector (TLD) at 2 kV and 22 pA electric current was used under high vacuum conditions.

For the characterization of phase contents, powder-XRD combined with quantification of the patterns was used. In different research groups, different XRD devices with different analysis software were used. In one research group, XRD was performed in a Siemens D5000 with operation parameters given elsewhere [11]. Rietveld refinement was performed with the software Profex (3.12.1). In the software package, the fundamental parameters approach for Rietveld refinement was applied [12]. In another research group, the software package of Bruker Topas V5.0 was used for Rietveld refinement. In the software package, the fundamental parameters approach for Rietveld refinement was implemented [13]. Additionally, an external standard [14] was applied in order to estimate the amorphous content of the CEM I 42.5 R, which was found to be negligible.

Acknowledgments

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References

[1] EN 196-2, Method of testing cement - Part 2: Chemical analysis of cement.
[2] https://www.originlab.com/doc/Origin-Help/Create-Box-Chart, 2019 (accessed 05 July 2019).
[3] H.M. Rietveld, A profile refinement method for nuclear and magnetic structures, J. Appl. Cryst. 2 (1969) 65–71. https://doi.org/10.1107/S0021889869006558.
[4] EN 1097-7: 2008, Tests for mechanical and physical properties of aggregates. Determination of the particle density of filler. Pyknometer method.
[5] EN 196-6: 2018, Method of testing cement – Part 6: Determination of fineness.
[6] ISO 9277: 2010, Determination of the specific surface area of solids by gas adsorption - BET method.
[7] ISO 13320: 2009 - Particle size analysis - Laser diffraction methods.
[8] EN 196-3: 2016, Method of testing cement – Part 3: Determination of setting times and soundness.
[9] EN 196-1: 2016, Methods of testing cement - Part 1: Determination of strength.
[10] EN 196-11: 2018, Methods of testing cement - Part 11: Heat of hydration - Isothermal Conduction Calorimetry method.
[11] F. Bellmann, D. Damidot, B. Möser, J. Skibsted J, Improved evidence for the existence of an intermediate phase during hydration of tricalcium silicate, Cem. Concr. Res. 40 (2010) 875–884. https://doi.org/10.1016/j.cemconres.2010.02.007.
[12] N. Döbelin, R. Kleeberg, Profex: a graphical user interface for the Rietveld refinement program BGMN, J. Appl. Cryst. 48 (2015), 1573–1580. https://doi.org/10.1107/S1600576715014685.
[13] R.W. Cheary, A. Coelho, A fundamental parameters approach to X-ray line-Profile Fitting, J. Appl. Cryst. 25 (1992) 109–121. https://doi.org/10.1107/S0021889891010804.
[14] D. Jansen, Ch. Stabler, F. Goetz-Neunhoeffer, S. Dittrich, J. Neubauer, Does Ordinary Portland Cement (OPC) contain amorphous phase? A quantitative study using an external standard method, Powder Diffraction Journal, 26 (2011) 31–38. https://doi.org/10.1154/1.3549186.

Appendix: Average values and the standard deviation calculated based on the results from different groups.

Table 2 Oxide composition of CEM I 42.5 R and the corresponding standard deviation.

	CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	K_2O	Na_2O	TiO_2	P_2O_5	Mn_2O_3	SO_3*	SO_3#	LOI	Cl^-	Insoluble residue	Sum
Composition (wt.-%)	64.4	20.4	5.4	2.6	1.4	0.77	0.22	0.29	0.14	0.07	2.7	3.11	1.87	0.02	1.04	100.12
Standard deviation	0.85	0.16	0.19	0.21	0.15	0.09	0.01	0.02	0.04	0.02	0.35	0.24	0.05	0.003	0.12	0.25

* Measured by XRF

Analysis by other methods

Table 3 Phase contents of CEM I 42.5 R and the corresponding standard deviation.

	C_3S	C_2S	C_3A (orth.)	C_3A (cub.)	C_4AF	Anhydrite	Bassanite	Arcanite	Calcite	Quartz	Periclase	Sum
Composition (wt.-%)	55.8	14.6	3.6	7.3	7.4	2.2	2.7	0.5	3.7	0.9	0.4	99.5
Standard deviation	1.79	0.45	0.58	0.50	0.97	0.27	0.45	0.23	0.19	0.21	0.11	0.50

Table 4 Physical properties of CEM I 42.5 R and the corresponding standard deviation.

	Density (kg/ dm^3)	Specific surface area* (cm^2/g)	Specific surface area # (m^2/g)	Particle size (μ m)		
				d(0.1)	d(0.5)	d(0.9)
Average value	3.115	3615	1.235	1.5	14.8	44.6
Standard deviation	0.0068	122.6	0.0584	0.66	1.03	1.29

* Measured by Blaine method

Measured by BET method

Table 5 Other properties of CEM I 42.5 R and the corresponding standard deviation.

	Water demand (wt.-%)	Setting time (h)		Compresive strength (MPa)				Flexural strength (MPa)			
		Initial	Final	1 d	2 d	7 d	28 d	1 d	2 d	7 d	28 d
Average value	29.4	2.7	3.7	19.9	30.3	45.9	56.8	4.6	5.8	7.5	8.1
Standard deviation	1.09	0.19	0.36	1.77	2.46	2.08	1.40	0.24	0.43	0.53	0.63