Acta Polytechnica CTU Proceedings **33**:160–167, 2022
https://doi.org/10.14311/APP.2022.33.0160

A NEW PERFORMANCE TEST TO EVALUATE THE SULFATE RESISTANCE OF CONCRETE BY TENSILE STRENGTH MEASUREMENTS

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Abstract.

Concrete structures without sufficient durability can be damaged by sulfates in groundwater and from surrounding rock layers. To evaluate the performance of a concrete mixture, precise and performance-oriented test methods are a must. Therefore, a new a performance oriented concrete test procedure based on tensile strength measurements was developed considering experiences reported in international literature and recommendations of state-of-the-art reports. A vast parameter study with approx. 3850 tensile tests on ASTM briquets, 1900 flexural tensile tests on standard prisms and 2100 expansion tests on mortar flat prisms of different ages and with different storage conditions was statistically assessed. Based on the results a performance-oriented test method could be defined which considers not only the chemical, but also the physical resistance of a concrete against sulfate attack. The method was verified by 23 concretes with different cements or cement fly ash combinations and additional field tests. It could clearly be demonstrated that the results represent the performance of a practical concrete in case of sulfate attack. Furthermore, it leads much faster to an evaluation of the sulfate resistance compared to the most other practical oriented methods.

Keywords: Concrete, performance test, sulfate attack, tensile strength.

1. Introduction

If concrete structures do not have a sufficient resistance, they can be damaged by sulfates dissolved in groundwater or incorporated in surrounding rock layers. Such a sulfate attack is described as exposure class XA in the European concrete standard EN 206. To ensure a sufficient concrete resistance, minimum requirements for concrete composition, such as a minimum binder content, a maximum accepted water-cement ratio (w/c), accepted types of cement and additives as well as other protective measures, if necessary, are defined in EN 206 together with their specific national concrete standards (e.g. DIN 1045-2 for Germany).

Beside the descriptive measures the mechanisms that trigger damage because of sulfate attack have been extensively investigated in the past, e.g. in [1–10]. A lot of test methods were developed worldwide. Most of them can be used to assess the chemical resistance of cements and binders, respectively. Only few, however, allow to test concrete. Depending on the method, the test specimens are completely, partially or cyclically immersed in sulfate solutions. The concentration of the sulfate solution varies over a wide range, as does its temperature. In Germany, only test methods are applied, which evaluate the chemical sulfate resistance of a binder. These are the so-called SVA (Sachverständigenausschuss) method of the DIBt (Deutsches Institut für Bautechnik), mainly used at present and the older methods by Koch-Steinegger [11] and Wittekindt [12]. Despite intensive research and optimization, all of them show considerable scattering of the results and test artifacts. These can be attributed, to extremely high sulfate concentrations of the test solution that are not related to practical applications. Furthermore, the physical resistance - the structural density of the concrete - was deliberately neglected in these procedures. For these reasons, none of the methods has been included in German or European standardization so far [13, 14].

However, for sustainability reasons, it is a must to be able to evaluate the performance of a concrete in a precise and performance-oriented way, because new types of cements, additives and concretes with lower ecological footprints must continue to be developed in future. Also, for technical or economic reasons, it is often advisable to deviate from the normative concrete specifications. Moreover, there are current activities to shift the classical descriptive concept of concrete standardization to a performance-based concept. All these activities require a reliable test method that allows an unerring evaluation of the sulfate resistance of a concrete mix.

Variable	Unit	Variation
Binder type	–	CEM I; CEM I-SR3; CEM I +fly ash; CEM II/B-S; CEM III/A
Testing temperature	°C	5, 12, 20
Concentration of sulfate solution	mg/l	3000, 6000
Type of sulfate solution	–	Na^+, $Mg2^+$ as cation
Cement content	kg/m^3	320, 360, 400
Content of cement + fly ash	kg/m^3	$270 + 90$, $285 + 94$, $300 + 100$
Equivalent water-cement-ratio w/c_{eq} [1]	–	0.45, 0.50

[1] $w/c_{eq} = w/(c + k \cdot f)$ with c = cement content, $k = 0.4$ and f = fly ash content

Table 1. Influencing variables on the sulfate resistance and variation parameters.

2. Objectives and Realization

The main objective of the research project was to develop a practical test method for the precise and selective determination of the sulfate resistance of concrete within an appropriate test period [15, 16]. The following three essential questions were defined to achieve the objective:

1. What are the test constraints that can be used to accelerate the damage mechanism without causing test artifacts? Are the test parameters verifiable, and which damage can be recorded reproducibly and accurately?
2. Is it possible to validate the results obtained with the new testing procedure with practical construction experience and field tests?
3. Is there a limit value to differentiate between concretes with high and insufficient sulfate resistance?

The study was based on experience gained from the currently applied methods as well as the findings of the state-of-the-art reports of DAfStb (Deutscher Auschuss für Stahlbeton) [13, 17, 18] and CEN/TC 51 [14]. Furthermore, both chemical resistance of the binder and physical resistance of the concrete structure were considered, since both partial resistances are important for the durability of concrete under practical conditions. Therefore, parameters influencing the sulfate resistance of concrete were varied systematically at the beginning of the project. In addition, their effect on various parameters characterizing the microstructure was determined.

The systematic statistical evaluation of the test results made it possible to define a test procedure based on the influencing parameters considered. This was followed by verification with another setup of approx. 25 concretes made with both binders known to have high and low sulfate resistance. At the same time, several of these concretes were stored under practical conditions at two different sites for at least one year [19]. The evaluation of all results made it possible to propose an acceptance criterion for the test procedure with which the sulfate resistance of a tested concrete can be evaluated reliably.

3. Experimental setup

3.1. Statistical design of experiments (DoE)

Statistical methods of design of experiments were used intensively in order to consider a wide as possible test matrix. The influencing parameters considered in the statistical experimental design are summarized in Table 1 [16]. In addition, their range of variation is also listed in the table. The statistical software Minitab© was used for designing and analyzing the experiments.

Due to the large number of influencing parameters and the different verification levels, the experimental strategy was to create a full-factorial experimental design first and to select an optimal experimental design from it afterwards. For this purpose, individual combinations were chosen by DoE software using methods of sequential optimization and taking into account terms up to the second order. The general full-factorial experimental design contained a total of 180 experiments covering all binders and a constant w/c_{eq} at 0.45. The optimal design selected from it could be reduced to 100 experiments.

Based on the statistical analysis of the results, the original test design was adapted so that individual influencing parameters that did not show any significance were omitted and additional parameters that were deliberately not considered in the first step (e.g. w/c_{eq}) were included. A total of 120 tests series were considered in the parameter study for the development of the test method [16].

3.2. Concrete production and storage

The binders listed in Table 1 have been investigated in detail. The four commercially available cements were in line with EN 197-1, the hard coal fly ash (FA) complied with EN 450-1. Their strength development measured according to EN 196-1 is shown in Table 2.

Concrete mixtures with those binders and quarzitic aggregate having a maximum grain size of 8 mm were produced. The w/c_{eq} value followed the specifications of the optimal test design. The production of the so-called fine concretes was in accordance with EN 12390-2. Prisms with dimensions $40 \times 40 \times 160$

	Compressive strength after			
	2 d	7 d	18 d	91 d
	N/mm^2	N/mm^2	N/mm^2	N/mm^2
(2) CEM I 42.5 N	28.5	46.6	61.9	71.0
(5) CEM I 42.5 N-SR3	24.5	42.5	50.3	60.8
(14) CEM II/B-S 42.5 N	22.2	41.1	61.8	73.3
(15) CEM III/A 42.5 N	18.7	40.8	61.9	73.9
(21) FA 1[1]	24.4	40.1	51.9	65.4

[1] Combination of 25 wt.-% fly ash and 75 wt.-% CEM I 42.5 R.

Table 2. Strength development of cements and fly ash.

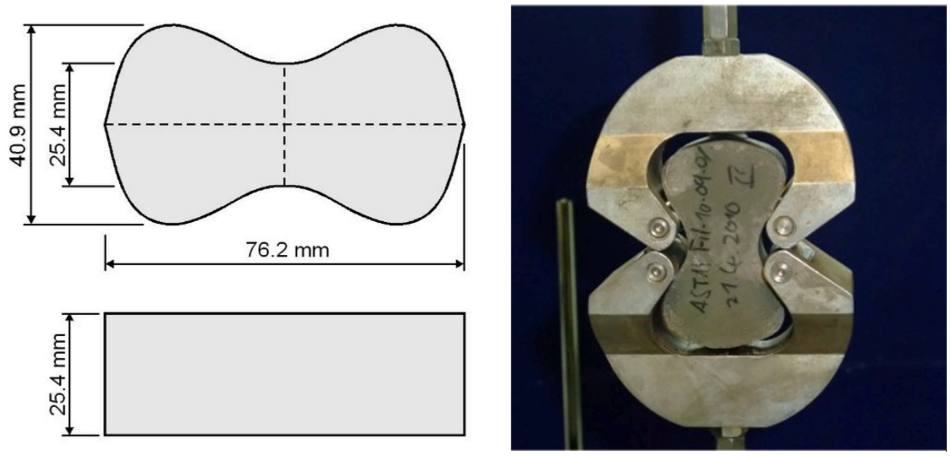

Figure 1. Concrete test specimens acc. to ASTM C307-03.

mm^3 for testing the flexural tensile strength and the dynamic modulus of elasticity and briquet specimens according to ASTM C307-03 (cf. Figure 1) for testing the tensile strength were produced as test specimens. All specimens were demolded after one day and then stored in saturated $Ca(OH)_2$ solution at 20 °C for 27 days.

3.3. Execution of tests and test parameters

The test started at a concrete age of 28 days. The specimens were stored in sulfate solution at 5, 12 or 20 °C for 181 or 273 days according to the conditions defined in the DoE. The flexural and tensile strengths as well as the dynamic modulus of elasticity were determined after 119, 181 and 273 days, where applicable.

All individual values and no mean values were always used to relate them to respective reference values. The relative values obtained this way can be compared directly with each other. Reference values were based on corresponding parameters obtained either on samples of same age but stored in saturated $Ca(OH)_2$ solution or determined before the start of sulfate storage. Furthermore, a maturity function was used for the calculation of relative bending and tensile strengths. The basis was the function described in the fib Model-Code [20]. It was adapted to account for the influence of supplementary cementitious materials on the strength development of concrete, as proposed by Vollpracht et al. [21]. Since the adapted method was originally developed for the prediction of compressive strength, its suitability was tested in advance for bending and tensile strengths [15, 16].

By using single values and not mean values, the number of results for each individual experiment of the statistical design was increased. Three single results each were used for the flexural tensile strength and the dynamic modulus of elasticity and even six single values for the tensile strength. This significantly increased the statistical certainty in the evaluation of the experimental designs.

4. Results and discussion

4.1. Suitable test criterions

The different relative test parameters were analyzed in terms of their significance for the test procedure. Damage to concrete due to sulfate attack was best characterized by the tensile strength of ASTM briquets. It was also shown that the common procedure, which uses the strength of specimens of the same age stored in a saturated $Ca(OH)_2$ solution as a reference value, gives comparatively wide scattering results. In contrast, tensile strengths based on the adapted maturity formula of the fib model-code show

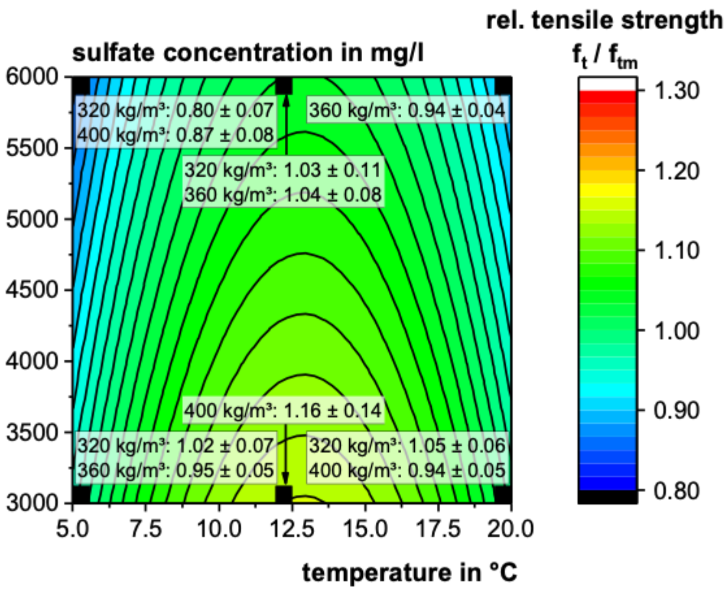

Figure 2. Contour diagram of relative tensile strength f_t/f_{tm} of concretes with (2) CEM I 42.5 after 182 days storage in Na_2SO_4 solution, reference: fib maturation function.

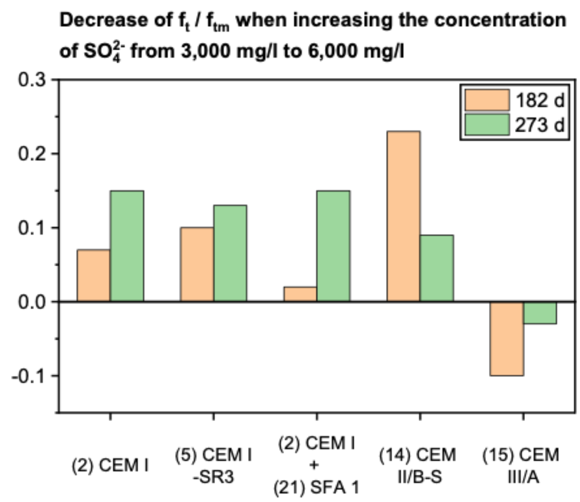

Figure 3. Mean decrease of the rel. tensile strength of the fine concretes with $w/c_{eq} = 0.50$ with a change of the sulfate concentration of the Na_2SO_4 solution from 3000 mg/l to 6000 mg/l.

significantly lower test scatter. The necessary test effort is also significantly reduced. Therefore, this relative tensile strength f_t/f_{tm} is the appropriate test parameter for the new test procedure.

4.2. Definition of the test procedure

The evaluation of the parameter study provides statistically secured information on the significant influencing parameters and their contribution to the expected relative tensile strength f_t/f_{tm}. As an example, the evaluation is visualized in a contour diagram (Figure 2) for fine concretes with Portland cement (2) CEM I 42.5 N after 182 days storage in Na_2SO_4 solution. It shows the effect of testing temperature and concentration of sulfate solution on the expected relative tensile strength. As a result, the lowest relative tensile strength can be expected if the concrete is tested at 5 °C and 6000 mg/l SO_4^{2-} concentration. The figure also contains the results of six additional tests, carried out to verify the results of the statistical evaluation.

Each single significant influencing parameter was analyzed in terms of its effect on accelerating the testing and its potential tendency towards test artifacts. For example, Figure 3 illustrates the accelerating effect when the sulfate concentration was increased. An increase of sulfate concentration from 3000 mg/l to 6000 mg/l led to a decrease of relative tensile strengths of comparable fine concretes between 0.10 and 0.15 (10 to 15 %) at a test age of 273 days, if damage occurred. At the same time, no excessive gypsum formation was observed in the pore

Concrete composition	• Fine concrete with max. aggregate size of 8 mm • w/c_{eq} ratio 10 % higher than planned for concrete formulation • Binder content as planned for concrete formulation
Test specimens	• Briquets acc. to ASTM C307-03 made from one concrete batch
Storage	• 28 d in saturated $Ca(OH)_2$ at 20 °C
Test conditions	• Test solution: Na_2SO_4 • ${SO_4}^{2-}$ concentration: 6000 mg/l • Storage temperature: 5 °C • Test duration: 273 days
Test parameters	• Relative tensile strength f_t/f_{tm} • f_t: measured tensile strength at testing • f_{tm}: tensile strength at testing, calculated by maturity function in accordance to fib Model Code • Visual assessment (cracks, spalling, etc.)

Table 3. Definition of the performance-oriented, test method for evaluating the sulfate resistance of concrete.

Figure 4. Storage site (1) in a German gypsum mine.

space of the specimens. Consequently, the increase of sulfate concentration to 6000 mg/l accelerates the test and does not cause artefacts, especially for less sulfate-resistant concretes. As a result of this evaluation the test procedure could be described. Its main features are summarized in Table 3.

4.3. Storage under practical conditions

A large number of fine concretes as well as normal concretes - the latter fulfilled the minimum requirements of DIN 1045-2 for the composition of exposure class XA2 - were stored under practical conditions at two sites. Figure 4 shows the exposure site in a gypsum mine. The laboratory results obtained with the new method will be verified by long-term tests with this real sulfate attack. Many samples were stored over a period of more than one year and inspected in regular intervals. No concrete deterioration was detected during this period as it is shown in Figure 5. It illustrates the relative dynamic modulus of elasticity of fine concretes stored in the gypsum mine. As expected, the storage time was too short to induce damage even on concrete that is known to possess insufficient sulfate resistance. The trials under practical conditions will be continued for some years.

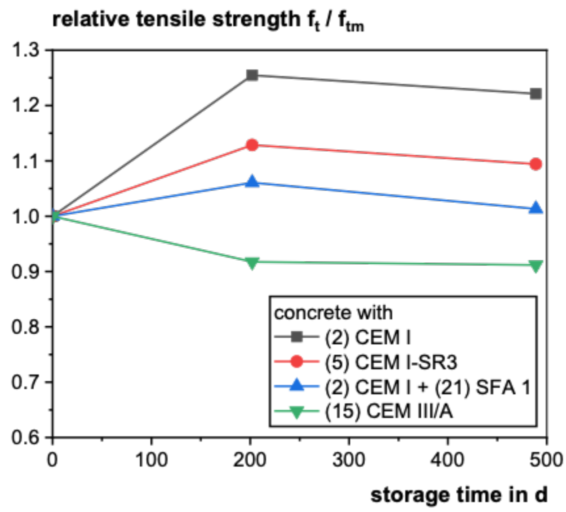

Figure 5. Rel. tensile strength of concretes after more than one year of storage under practical conditions.

4.4. Proposal of an acceptance criterion

After the definition of a new test procedure another task of the research project was to develop a proposal for an acceptance criterion for a reliable evaluation of the concrete sulfate resistance. For this purpose, 23 additional concretes with different cements and cement-fly ash combinations of different manufacturers were tested with the new test method. The relative tensile strength was determined after 119, 182 and 273 days. Figure 6 illustrates the relative tensile strengths of the 23 concretes after 182 and 273 days of sulfate storage.

After 119 days of storage no reliable statement can be made on the sulfate resistance. The first differences between concretes with different binders did occur after 182 days of storage. However, a definitive differentiation between concretes with known high or low sulfate resistance was not yet possible. Some con-

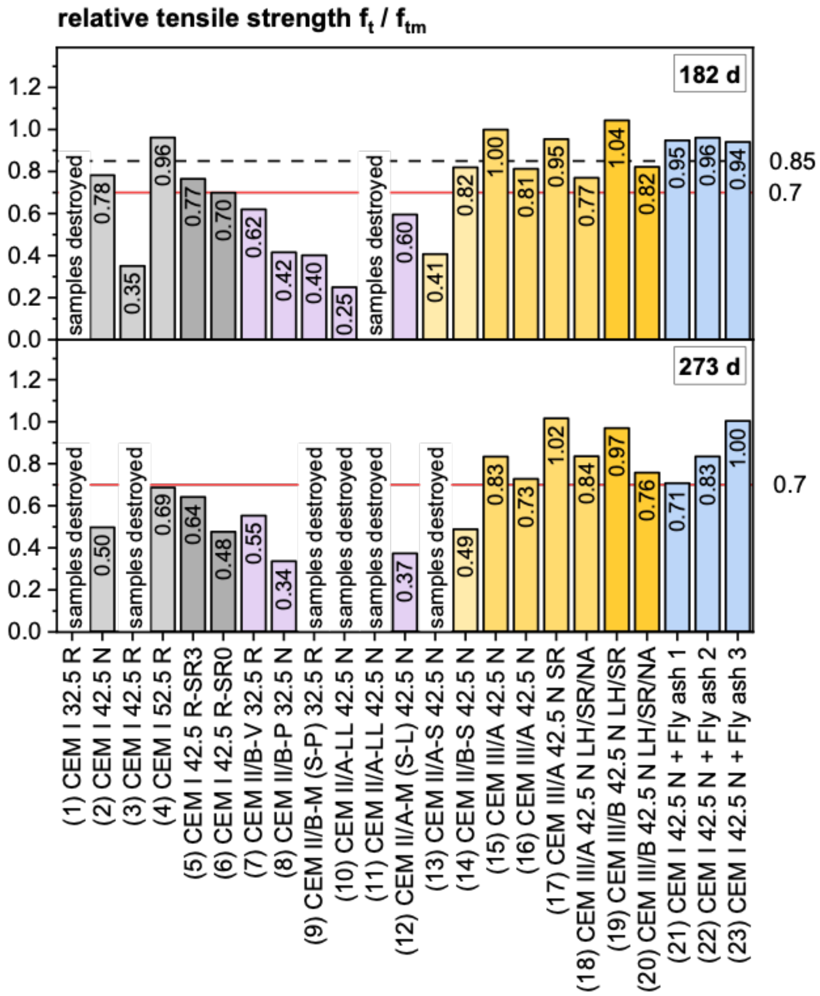

Figure 6. Relative tensile strength f_t/f_{tm} of concrete with 23 different binders after 182 and 273 days.

cretes produced with Portland cement without SR property showed residual tensile strengths comparable to those of some slag cement concretes, for which a high sulfate resistance can be expected in the light of experience. After 273 days (9 months) of sulfate storage, it was possible to make a clear distinction regarding the sulfate resistance of concrete. Concretes with blast furnace cements CEM III/A or CEM III/B and Portland cement/fly ash combinations obviously showed a high sulfate resistance with relative tensile strengths of 0.97 to 1.02. In contrast, concretes with Portland cement - including those with SR property - and Portland composite cements showed low residual tensile strengths and significant damage. The damage of concretes with CEM I-SR cements was confirmed by further tests. C_3A was determined by x-ray diffraction for all these cements. Presumably, the C_3A content is high enough to trigger a damaging ettringite reaction with the sulfate ions in the cement stone structure.

Considering the discussed results, it can be stated that a concrete has a sufficient sulfate resistance if its relative tensile strength is not lower than 0.70 (70 %) after 273 days of sulfate storage. Furthermore, two stop criteria can also be defined for the test after 182 days. Firstly, the test can already be stopped at this time if the relative tensile strength f_t/f_{tm} is lower than 0.70 (70 %), since the acceptance criterion will definitely not be reached even after 273 days. Such a concrete will have a low sulphate resistance. Secondly, the test can also be stopped at this point if the relative tensile strength f_t/f_{tm} is higher than 0.85 (85 %), because the acceptance criterion defined for a test age of 273 days is then also fulfilled with certainty. Such a concrete will have a high sulfate resistance.

5. Conclusions

The focus of the research project was the development of a concrete test procedure based on tensile strength tests, which allows a clear differentiation between concretes with and without high sulfate resistance. In the development of the test method, the recommendations of the state-of-the-art report [13] were taken into account and the tensile strength was determined as the best test parameter [16].

Based on the statistical evaluation of approx. 3850 tensile tests on ASTM briquets, 1900 flexural tensile tests on standard prisms and 2100 elongation tests on mortar flat prisms of different ages and after different pre-storage conditions, a new performance-oriented test method could be defined which was verified by 23 concretes with different cements or cement fly ash combinations.

The concluding assessment of the research project is that the newly developed performance-oriented test method

- can represent the performance of a practical concrete in case of sulfate attack,
- considers not only the chemical, but also the physical resistance of a concrete against sulfate attack,
- leads much faster to an evaluation of the sulfate resistance compared to common methods (current regulation SVA test: testing at 3000 mg $SO_4{}^{2-}$/l and 5 °C for 2 years),
- represents the damage mechanism more realistically than most conventional test methods and therefore leads to the avoidance of test artifacts, and
- could also be carried out as a "binder test" if a fixed concrete formulation is used (e.g. the limit formulation of DIN 1045-2 for exposure class XA2).

Acknowledgements

The IGF project no. 19251 N of the Research Association VDEh-Gesellschaft zur Förderung der Eisenforschung mbH was funded via the AiF as part of the program for the funding of joint industrial research (IGF) by the Federal Ministry of Economics and Energy based on a resolution of the German Bundestag. The authors would like to express their thanks for the support.

References

[1] F. Bellmann, B. Möser, J. Stark. Influence of sulfate solution concentration on the formation of gypsum in sulfate resistance test specimen. *Cement and Concrete Research* **36**(2):358-63, 2006. https://doi.org/10.1016/j.cemconres.2005.04.006.

[2] H.-G. Smolczyk, G. Blunk. Zum Verhalten von sehr jungem Beton gegen Sulfatwässer Beton-Information **12**:1-9, 1972.

[3] H. Passow. Hochofenzement und Portlandzement in Meerwasser und salzhaltigen Wässern Verlag der Tondindustrie-Zeitung GmbH (Berlin, Germany), 1915.

[4] R. Grün, H. Beckmann. Untersuchungen über das Verhalten von erhärtetem Hochofenzement gegen Sulfatlösungen und salzarmes Wasser. *Angewandte Chemie* **45**(48):739-43, 1932. https://doi.org/10.1002/ange.19320454802.

[5] J. Skalny, J. Marchand, I. Odler. Sulfate Attack on Concrete Spon Press (London, UK), 2002.

[6] W. Müllauer. Mechanismen des Sulfatangriffs auf Beton - Phasenneubildungen und Expansionsdrücke in Mörteln unter Na2SO4 Belastung (München, Germany), 2013.

[7] K. Lipus. New Approach for Testing SR Cements External Sulphate Attack Menéndez, Baroghel-Bouny (Basel: Springer International Publishing) pp. 107-120, 2020.

[8] F. Aguayo, O. J. Funez, T. Drimalas, et al. An Alternative Method to Evaluate the Sulphate Resistance of Cementitious Binders. External Sulphate Attack - Field Aspects and Lab Tests. RILEM Bookseries p. 93-105, 2020. https://doi.org/10.1007/978-3-030-20331-3_8.

[9] N. B. Tiburzi, J. Garcia, T. Drimalas, et al. Sulfate resistance of portland-limestone cement concrete systems: Linking laboratory and field performances. *Construction and Building Materials* **250**, 2020. https://doi.org/10.1016/j.conbuildmat.2020.118750.

[10] F. Herding. Mikrostrukturelle Schädigung von Beton durch Sulfatangriff unter unterschiedlichen Bedingungen, Master Thesis RWTH Aachen University (Aachen, Germany), 2020.

[11] A. Koch, H. Steinegger. Ein Schnellprüfverfahren für Zemente auf ihr Verhalten bei Sulfatangriff Zement-Kalk-Gips, **13**:317-324, 1960.

[12] W. Wittekindt. Sulfatbeständige Zemente und ihre Prüfung Zement-Kalk-Gips, **13**:565-572, 1960.

[13] DIN-Fachbericht CEN/TR 15697:2008-10, Cement - Performance testing for sulfate resistance - State of the art report, 2008.

[14] R. Breitenbücher, D. Heinz, K. Lipus, et al. Sulfatangriff auf Beton Sachstandbericht Deutscher Ausschuss für Stahlbeton Beuth-Verlag (Berlin, Germany), 2006.

[15] J. Haufe, A. Vollpracht, T. Matschei. Development of a Sulfate Resistance Performance Test for Concrete by Tensile Strength Measurements: Determination of Test Conditions. *Crystals* **11**(8), 2021. https://doi.org/10.3390/cryst11081001.

[16] A. Ehrenberg, A. Vollpracht. Entwicklung eines Performanceprüfverfahrens zur Bestimmung des Sulfatwiderstands von Beton, Schlussbericht zum IGF-Vorhaben Nr. 19251 N FEhS Institut für Baustoff-Forschung (Duisburg, Germany), 2020.

[17] H.-M. Ludwig, M. Müller, P. Nobst. rgänzende Untersuchungen zum DAfStb-Verbundforschungsvorhaben Vertiefte Untersuchungen zum Sulfatwiderstand von Beton, Abschlussbericht zum DAfStb-Forschungsvorhaben V 469/2 Bauhaus-Universität Weimar (Weimar, Germany), 2012.

[18] D. Heinz, L. Urbonas. Ergänzende Untersuchungen zum DAfStb-Verbundforschungsvorhaben Vertiefte Untersuchungen zum Sulfatwiderstand von Beton, Abschlussbericht zum DAfStb-Forschungsvorhaben V 469/1, Technische Universität München (München, Germany), 2012.

[19] J. Haufe, A. Vollpracht, T. Matschei. Performance Test for Sulfate Resistance of Concrete by Tensile Strength Measurements: Determination of Test Criteria. *Crystals* **11**(9), 2021. https://doi.org/10.3390/cryst11091018.

[20] FIB 2013 fib Model Code for Concrete Structures 2010 Ernst & Sohn (Berlin, Germany), 2010.

[21] A. Vollpracht, M. Soutsos, F. Kanavaris. Strength development of GGBS and fly ash concretes and applicability of fib model code's maturity function - A critical review. *Construction and Building Materials* **162**:830-46, 2018. https://doi.org/10.1016/j.conbuildmat.2017.12.054.