Report.

Research projects of the FEhS-Institute **2020**

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BRINGING TOGETHER THE PROCESS INDUSTRY TO DISCUSS EUROPEAN NON-TECHNICAL BARRIERS TO INNOVATION

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ABSTRACT

Increasing innovation for sustainable growth in Europe is widely acknowledged by public authorities, academia and industry as a means to increase European global competitiveness. The HARMONI project connected all SPIRE industrial sectors (steel, minerals, non-ferrous metals, engineering, chemicals, ceramics, cement and water) in order to identify non-technical barriers to innovation currently facing the European process industry. The three key challenges that were identified are 1) regulation and regulatory processes, 2) barriers to transferability of innovation and 3) standards and standardization process. By identifying common barriers shared by different heavy industry, the European authorities and industry can be given priorities to enhance the competitiveness in Europe. This article presents tool boxes developed during the HARMONI project which may be used to address barriers to cross-sectorial transfer and the current standardization needs of the process industry in order to help the industry to implement its innovations better.

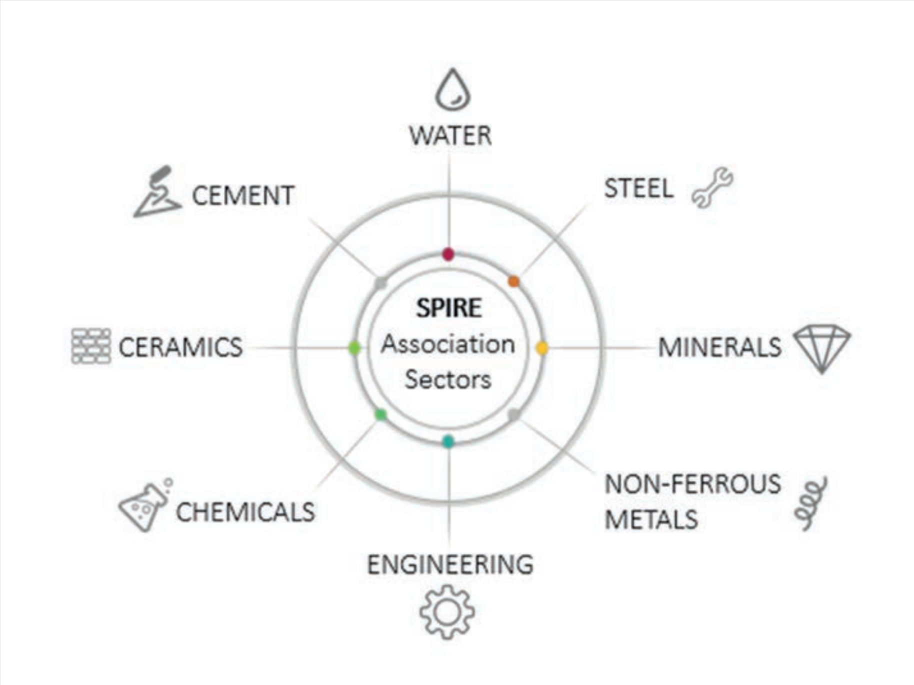

Figure 1: Sectors of SPIRE network

INTRODUCTION

The European process industry is technologically sophisticated, innovation-orientated and has high transferability potential for innovation. However, it is trailing its major world competitors in research and development [1]. For Europe to be competitive in the global market it must focus on innovation and barriers which are blocking innovation. European cross-sectional cooperation can help identify common non-technical bottlenecks. The HARMONI project (Aug 2017 to Oct 2019, www.spire2030.eu/harmoni) assembled all SPIRE (Sustainable Process Industry through Resource and Energy Efficiency) sectors: steel, minerals, non-ferrous metals, engineering, chemicals, ceramics, cement and water in order to better understand the needs of the European process industry with respect to non-technical barriers to innovation (Figure 1).

The HARMONI project tried to address why companies in the European process industry struggle to implement innovations in the market and how to develop solutions to foster innovation activities within the industry. The three key challenges identified are 1) regulation and regulatory processes, 2) barriers to transferability of innovation and 3) standards and standardization process.

The non-technical issues with respect to regulation and regulatory process identified during the HARMONI project have overlapping interest in the various European process industry sectors: holistic approach, access to public funding, circular economy, waste recycling, plastic recycling and carbon capture and utilization [2]. The issues have been identified through literature review, phone interviews with various stakeholders and workshops. The groups targeted for input include industry representatives, associations from each sector and related project coordinators. A discussion with authorities was started and some feedback obtained. These issues in relation to the steel industry were previously presented during the 10th European Slag Conference [3].

This article presents barriers to cross-sectorial transfer and the current standardization needs of the process industry.

CROSS-SECTORIAL SOLUTIONS TRANSFER

Social, institutional and technical innovations are considered as the main drivers of structural change and international competitiveness [4] [5]. The identified key barriers to innovation transferability were structured into "barriers" and "missed opportunities".

In terms of the barriers, the key categories identified are:

- Lack of incentives; taxation
- Lack of harmonization
- LCA and misestimating in life cycles
- Financial barriers: funding, financing and investment decisions
- Bureaucratic barriers

In terms of the missed opportunities, the key categories identified are:

- Cross-sectorial cooperation and partnerships
- Role of the industrial associations for innovation transfer
- Research
- Information sharing and communication; knowledge management
- IT-based challenges

Potential key areas of improved knowledge transfer	Potential key areas of technology transfer	Social, political, economic & industrial trends
a. Accelerated deployment of the R&D&I opportunities identified within SPIRE b. Access to funding and new business models, e.g. for circular economy c. Cluster initiatives to leverage outreach d. Innovation management (high exchange of approaches, Best Practices, lessons learned) e. Management attitude & corporate culture f. New materials / surfaces g. Process modification and refinement h. Logistics i. Licensing in different life cycle phases j. Skills & training k. Smart Specialisation	a. Construction technologies b. Information technology, IT applications and software, IT-security technology and monitoring applications c. Key Enabling Technologies, especially - Nanotechnology - Advanced materials - Advanced manufacturing d. Production technology including mechanical engineering and machinery e. Testing facilities (DIN e.V. & DKE 2018) f. Resource and energy efficient process industry* - Feedstock - Processes and process intensification, incl. ISy - Market application - Waste2Resource - CO_2 to fuel/chemicals	These trends form a third type of transfer since they require deepened knowledge exchange & cooperative learning a. Digital transformation & Industry 4.0 b. Enhanced use of optimization technique c. Pro-environmental activities - Circular economy - Energy management - CO_2 valorisation - Renewable energies - Waste recycling d. Lightweight products e. Measuring devices f. Monitoring technologies g. Industrial symbiosis (IS)

Table 1: Identified key areas of potential transfer opportunities

- Utilizing digital tools
- Inadequate change management and technology adoption
- Role of standardization for innovation transfer

There are various approaches on how to foster innovation activities in companies. A very important way of doing so is innovation transfer, and HARMONI dealt with questions about how to measure and improve the cross-sectorial transfer of innovations. Through extensive cooperation with the industry, it was possible to define a methodology for companies to assess their cross-sectorial transferability and a holistic set of transferability indicators across the value chain. Stakeholders are provided with the most relevant channels to transfer innovation and potential key areas of transfer opportunities.

Twenty-nine critical success factors were identified through comprehensive research. The identified key areas of potential transfer opportunities are shown in Table 1.

Results from SPIRE projects were evaluated for transferability potential to other sectors based on the key areas identified. These results are presented in Deliverable 5.3 [6]. Table 2 shows an example of how the results are presented based on innovation area, technology, application, scaling-up interest, current TRL, innovation area 2, sector, replication sector and initiative. With this tool it is relatively easy to find potential technologies that can be transferred to another sector.

Access to the tool box can be found at www.spire2030.eu/HARMONI#edit-group_outcomes HARMONI Final Booklet.

STANDARDIZATION

The companies and associations in the process industry actively participate in standardization on European and international level [7]. Standards which are developed by the industry are a good way for the industry to set a standard for their innovation. Standardization can help stimulate innovation by

Innovation area	Technology	Application	Scaling-up interest?	Current TRL	Innovation area 2 (if any)	Sector	Replication sector	Initiative
ENERGY FLEXIBILITY MANAGEMENT	ENERGY MANAGEMENT SYSTEM	Monitoring of main variables related to energy and regulating them	PULP & PAPER	6	Digitalisation of plant and plant operations	Non-ferrous metals	Steel (metallurgy, casting), chemicals, cement, glass, refining	SusPire
		Modular framework for seamless data management in the ERMS		5	Digitalisation of plant and plant operations	Chemicals (Polymers), Others	Refining (TRL 6-8)	SYMBI-OPTIMA
	FLEXIBILITY PLATFORM	DSS for optimising waste streams valorisation		5		Chemicals, Minerals (Magnesite), Steel, Others	-	BAMBOO
		Optimisation of demand-side management and distributed generation		4	Process technology for Industrial Symbiosis	Chemicals (Polymers), Others		SYMBI-OPTIMA

Table 2: Identified key areas of potential transfer opportunities

providing solutions for free global trade of goods and services [8]. Standardization limits barriers by facilitating an exchange of goods, processes, and services. It promotes dissemination and application of innovations, lowers R&D risks and costs, assures quality, assures environmental protection and improves communication and information exchange [8].

The organizations responsible for global standardization are the International Organization for Standardization (ISO) [9] together with the International Electrotechnical Commission (IEC) [10]. The International Telecommunications Union (ITU) [11] is the United Nation's specialized agency for information and telecommunication technologies. Many of ISO's members belong to regional standardization organizations. ISO has recognized regional standards organizations representing Africa, the Arab countries, the area covered by the Commonwealth of Independent States, Europe, Latin America, the Pacific area, and the South-East Asia nations.

In Europe, standardization is conducted by the European Committee for Standardization (CEN) [12], the European Committee for Electrotechnical Standardization (CENELEC) [13] and the European Telecommunications Standards Institute (ETSI) [14].

At European level, different standardization documents are available. Each of these represents a different level of consensus [7]:

- The European Standard (EN) aims at developing (Figure 2) a normative specification reflecting the current state of technology and knowledge. Every CEN member is obligated to acquire the EN and to withdraw national standards which are in conflict with or duplicate EN standards.
- Other products of European standardization include the European Technical Specifications (CEN/TS), which aim to aid market development and growth for products or methods that are still in the development and/or trial phase, and the European Technical Reports (CEN/TR), which provide speci-

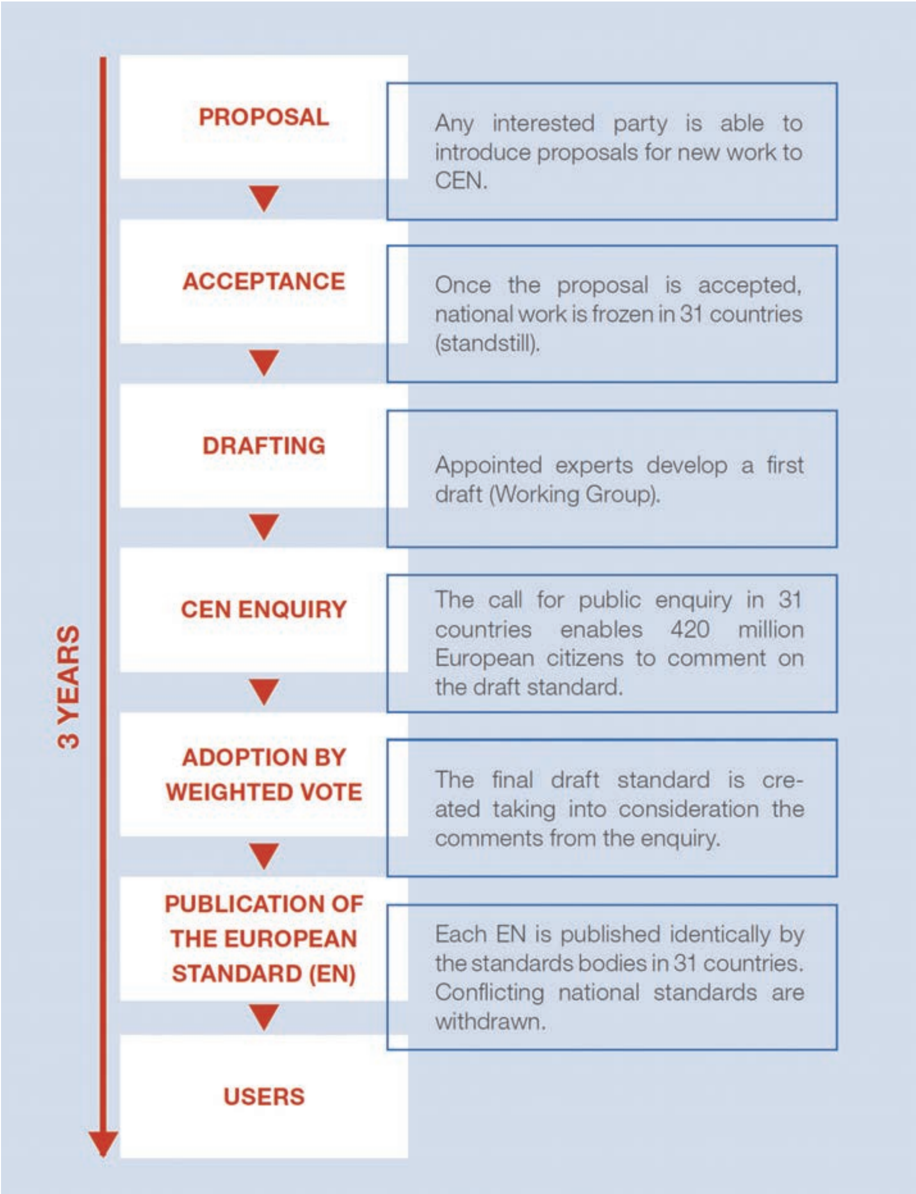

Figure 2: Development of an EN [15]

fications of a recommendatory and explanatory nature.

- Special specifications, which are developed with the rapid consensus of expert stakeholders (no full consensus needed), can be found in CEN Workshop Agreements (CWA).

While the process industry actively takes part in the standardization process due to the benefits, the HARMONI project identified common perceived bottlenecks [7]. Examples include:

- Time to market
- Absence of resources to participate
- Excessively complex decision-making process
- Participation costs too high
- Missing information about the process
- IPR issues/drainage of know how
- No access to the standardization process
- Irrelevance to an organization

The HARMONI project identified the needs with respect to standardization of the process industry and gave suggestions based on them. In addition, it was noticed that a large part of the standardization work of the past years focused on aspects of waste management and waste prevention instead of the circular economy itself. Often, processes are not considered as a circle, but in a linear manner. Questions of material recycling, recyclability, reuse and reprocessing are also usually dealt with within the framework of a specific industry and/or product group. To date, there are no formal standards that concentrate on the concept of recycling management in its entirety. However, this area is gaining momentum due to the great importance of this topic and the standardization processes [16].

During the HARMONI project, a standardization tool box was developed to help the process industry participate in the standardization process. It can be found at https://www.spire2030.eu/sites/default/files/users/user740/harmoni_standardisation_toolbox.pdf.

SUMMARY

In order for the European process industry to better implement innovations on the global market, key issues were identified by the HARMONI project as non-technical barriers, such as cross-sectorial solutions transfer and standardization.

With respect to cross-sectorial solutions transfer, a tool box has been compiled for companies as an easy way to pinpoint potential technologies that can be transferred to other sectors. The tool box is based on research conducted in the project to identify twenty-nine critical success factors based on, for example, innovation area, technology, application, scaling-up interest, current TRL, innovation area 2, sector, replication sector and initiative.

In terms of standardization, a tool box was developed to help the process industry participate in the standardization process. While most of industry already participates in the standardization process, common perceived bottlenecks were identified during the HARMONI project, e.g. time to market, absence of resources to participate, excessively complex decision-making process, participation costs too high, missing information about the process, IPR issues/drainage of know-how, no access to the standardization process and irrelevance to an organization. The tool box addresses these concerns and helps to guide participants towards better involvement in the process.

ACKNOWLEDGEMENT

This project has received funding from the European Union's Horizon 2020 research and innovation programme under Grant Agreement No 768755. <<<

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COMPARISON OF DIFFERENT METHODS FOR DETERMINING THE SURFACE OF COARSE AGGREGATES

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ABSTRACT

CEN/TC 351 "Construction products: Assessment of release of dangerous substances" provided Technical Specifications for horizontal test methods, which are necessary for the implementation of the Basic Requirement No 3 "Hygiene, Health and the Environment" in the Construction Products Regulation (CPR) into harmonized Product Standards. Based on the European Commission's (EC) mandate amendments, the Product Committees must integrate this basic requirement in the product standards for the European internal market as a further performance characteristic in future. With the publication of CEN/TS 16637-2, a "Dynamic Surface Leaching Test (DSLT)" was made available to the CEN Technical Committees. It relates to an index test method to evaluate surface-related release from construction products. The DSLT is an appropriate test method for larger aggregates such as armourstone (EN 13383-1) and railway ballast (EN 13450) due to the surface-related release scenario. To date no standards are available for the determination of the surface of aggregates. In contrast to geometrically simple products, such as paving stones, it is not easy to calculate the surface area of aggregates with irregular side areas. The DSLT mentions the aluminium-foil method for the determination of the surface of products with irregular sides. Contactless 3D measurements by laser scanning or computed tomography system (CT) could be an alternative. This paper presents a comparison of these methods.

Figure 1: Construction of an embankment from aggregates; armourstone (top layer) and grain filters

INTRODUCTION

The largest quantities of construction products on navigable waterways are aggregates. They can be used as concrete aggregate for solid structures or for revetments of navigable waterways. In the tidal area of rivers (Figure 1), the intended use of aggregates can be as armourstone (top layer) and grain filter (bottom layer).

In terms of structural aspects, the geometrical properties of the aggregates are important parameters to assess the functionality of these construction products for the revetment to be erected. Within the meaning of EN 13383-1 [1], armourstone used in hydraulic structures and other civil engineering works can be natural, manufactured as slag material or recycled. For example, grain size classes for armourstones according to [1] are defined with square-hole sieves, and the shape must be determined by the length-to-thickness ratio. The percentage of pieces of armourstone with a length-to-thickness ratio greater than 3 must be tested by using straight laths and a carpenter's rule or a tape-measure, or by using a calliper. In accordance with the Construction Products Regulation (CPR), Regulation (EU) No 305/2011 [2], the Basic Requirement No 3 (hygiene, health and the environment) must also be reflected in the harmonised construction product standards throughout Europe. In future, manufacturers must also indicate in their declarations of performance (DoP), whether dangerous substances can possibly be released. With the publication of the CEN/TS 16637-2 [3] of the CEN/TC 351 "Construction Products – Evaluation of the Release of Dangerous Substances", the horizontal test method – the "surface leaching test (DSLT)" – was made available to the CEN Technical Committees for the implementation of Basic Requirement No 3. It relates to an index test method to evaluate surface-related release from monolithic, plate- or sheet-like products. Armourstone [1] or railway ballast [4] can be considered as monolithic. In situ tests of single stones in water are carried out in which water samples are taken at fixed times and then analyzed. A status report on leaching test methods developed by CEN/TC 351 was already presented at the 8th European Slag Conference in Linz 2015 [5]. The surface of the samples is needed twice for the DSLT [3]. Firstly, the liquid volume to surface area-ratio (L/A) must be calculated. For monolithic products, the test is carried out at an L/A ratio of (80 ± 10) l/m²; for sheet- or plate-like products, lower L/A ratios ≥ 20 may be applied. Furthermore, the concentration of released substances after 64 days must be expressed in mg/m² to provide a surface-related specification of the release of dangerous substances. To date no standard test methods are available for the determination of the surface or

Figure 2: Unfavourably formed armourstone with a length-to-thickness ratio > 3

contactless measurements of irregular aggregates.

TEST METHODS AND MEASURING DEVICES FOR GEOMETRIC PROPERTIES

Up to now the product standards for aggregates require only contact measuring methods such as the determination of the shape or size. For instance, armourstone in accordance with EN 13383-2 [6] requires labour-intensive manual measurements to determine the length-to-thickness ratio. Figure 2 shows the measuring with a carpenter’s rule on a revetment of a river.

Thanks to the new horizontal Dynamic Surface Leaching Test (DSLT) [3], the surface determination will be an important geometric property in the future. According to [3], the geometric area of very irregular test pieces must be determined using the aluminium-foil method. Figure 3 shows the steps of the procedure:

A stone must be wrapped in aluminium-foil and the foil which is in contact with the lateral surface

Figure 3: Aluminium-foil method for surface determination, tear down of foil pieces

Figure 4: Measuring by a hand-held scanner

Figure 5: Measuring by laser scanner

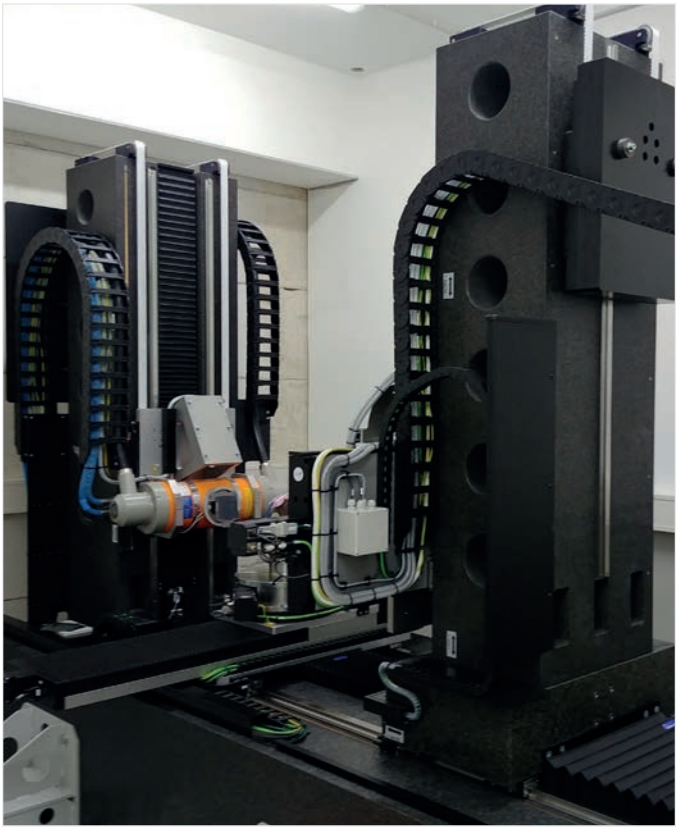
must be torn down and weighed. The surface of the sample can be calculated using the mass per unit area of the foil.

A comparison of several different methods for determining the surface – one of them being the aluminium-foil method – was published back in 2012 [7]. The aluminium-foil method showed the best results with regard to the statistical uncertainty, compared to a 3D laser scanning method. However, this comparison was presented only for small samples with a mass of less than 2 kg. This method was described as being fast, inexpensive and reliable.

More accurate scanners have since been developed, and the use of industrial computer tomography based metrology (CT) is more widespread and less expensive. The hand-held scanner uses the photogrammetric measuring system (Figure 4).

A portable laser scanner (Figure 5) has a more modern measuring technique with greater range accuracy. In the non-contact 3D scanners used here, the light acts as a measuring medium based on reflection and absorption. Optical sensors are able to detect and evaluate visually accessible areas of an object.

A greater point density is recorded, and the result can be promptly compared with a reference model. Contact measurement technology is currently reaching its limits when it comes to measuring speeds or measuring surface structures in the millimetre range. An important component for non-contact measuring systems is a powerful evaluation software for the analysis of large amounts of data, e. g. for a complete 3D model. A hand-held portable scanner of an older design (Artec MHT 3D, works with photogrammetry) and the current model of a laser scanner (Leica P30) were used. Terrestrial laser scanners work on a reflector-less distance measurement with simultaneous determination of two solid angles. Local three-dimensional coordinates can be derived.



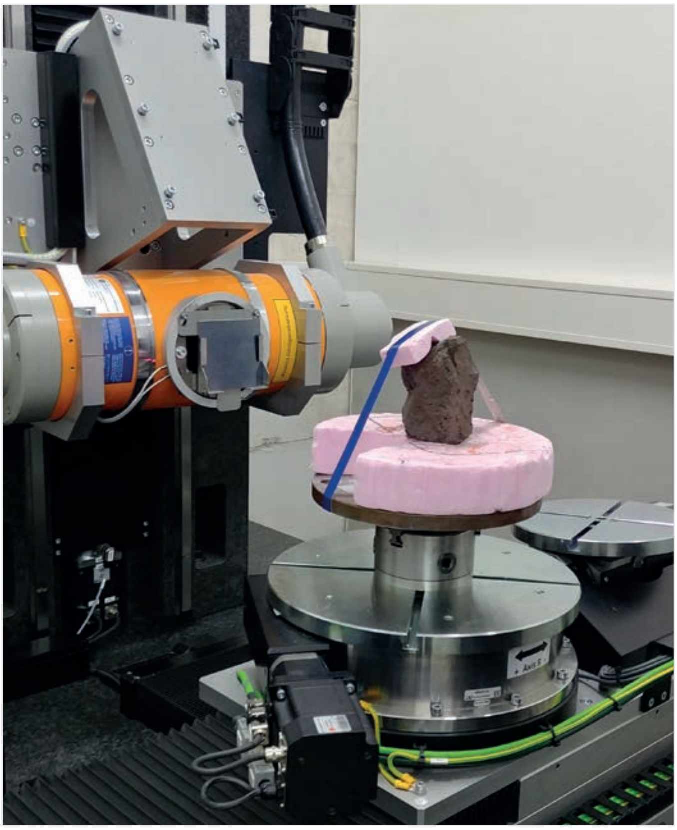


Figure 6: Measuring by CT: Macro CT system (left) and armourstone sample (right)

Discrete points are not specifically observed with terrestrial laser scanners. Rather, the environment of the measuring object is recorded at high speed in defined steps. The measurement result of the laser scanner is a point cloud. In addition to the 3D coordinates, the user receives an intensity value per point that describes the reflectivity of the measured object. Essential characteristics of a scanner are the range, the resolution, the beam divergence and the measuring accuracy. The range of a laser scanner is determined by the manufacturer, depending on the laser power and quality of the receiving optics. The resolution of the measurement depends on the selected step size for the two deflection angles. The smaller the step size, the finer the spatial resolution of the measurement object. With regard to beam divergence, the sampling rate should be tuned to one another. No sampling rate should be selected that is smaller than the spot size. The scan station P30 has a range accuracy of 1.2 mm + 10 ppm over the entire range. It also has a 3D position accuracy of 3 mm at 50 m. The beam divergence is > 0.23 rad and the laser spot size at the front window is less than 3.5 mm.

In contrast to medical use for CT in non-destructive testing, the sample rotates. A 3D volume is reconstructed from a series of 2D X-ray projection. High resolution CT can be performed for greater samples such as armourstone by macro-focus technology (Figure 4). An application for CT on construction products with irregular side areas is described in [8]. The resolution of the 3D CT data is described in voxel, and the size of this data directly depends on the sample size and the pixel size of the detector. A CT measurement of an armourstone can be performed in 30 min (Figure 6). Further details of the measuring methods used are described in [9].

MEASUREMENTS AND RESULTS

In order to be able to evaluate the accuracy of the methods by comparing with calculations, measurements were first carried out on geometrically simple test specimens of natural stone and concrete (Table 1). The selection of specimens was made due to their different colours and pattern. In order to test resolution

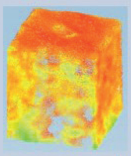
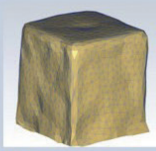
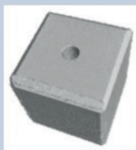
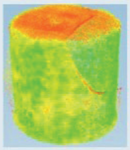
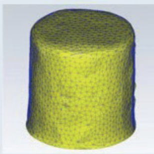
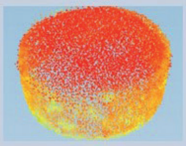
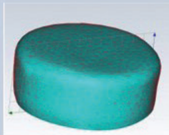
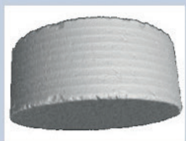
Calculated		Laser scanner		CT	
Sample 1: granite **surface [cm²] = 206.5** **volume [cm³] = 201.8**					
		surface [cm²] = 208.7 **volume [cm³] = 205.3**		**surface [cm²] = 209.11** **volume [cm³] = 195.50**	
Sample 2: concrete **surface [cm²] = 117.8** **volume [cm³] = 98.2**					
		surface [cm²] = 116.3 **volume [cm³] = 96.2**		**surface [cm²] = 151** **volume [cm³] = 98.26**	
Sample 3: rhyolithe **surface [cm²] = 72.4** **volume [cm³] = 40.9**					
		surface [cm²] = 60.6 **volume [cm³] = 33.7**		**surface [cm²] = 70.85** **volume [cm³] = 39.89**	

Table 1: Samples with regular dimensions, comparison of methods

Test method	Dolomite		Copper slag	
	surface [cm²]	volume [cm³]	surface [cm²]	volume [cm³]
Aluminium-foil method	546.4		315.3	
Archimedes volume		641.1		235.0
Laser scanner Leica P30	559.1	601.5	239.2	239.8
Hand-held scanner Artec MHT 3D	467.2	646.9	227.3	234.2
CT	668.0	641.5	535.5	243.7

Table 2: Comparison of results for dolomite and copper slag [8]

of the measuring systems, a cube with chamfer and bore as well as a cylinder were chosen. The surface and volume of cubes and cylinders can be easily calculated. Contactless 3D measurements were carried out by laser scan and CT. The granite cube was calculated without bore and chamfer. It must firstly be pointed out that the laser cannot detect the chamfer and the bore of the cube. The surface of the cube was calculated without bore and therefore the CT result of the surface is slightly larger and the volume slightly lower. For the concrete cylinder, the CT shows a slightly larger surface due to roughness of the lateral surface and the pores. In summary it can be stated that the CT shows a better match with the calculations than the laser scanner.

Figure 7: Copper slag (left) and dolomite (right) [8]

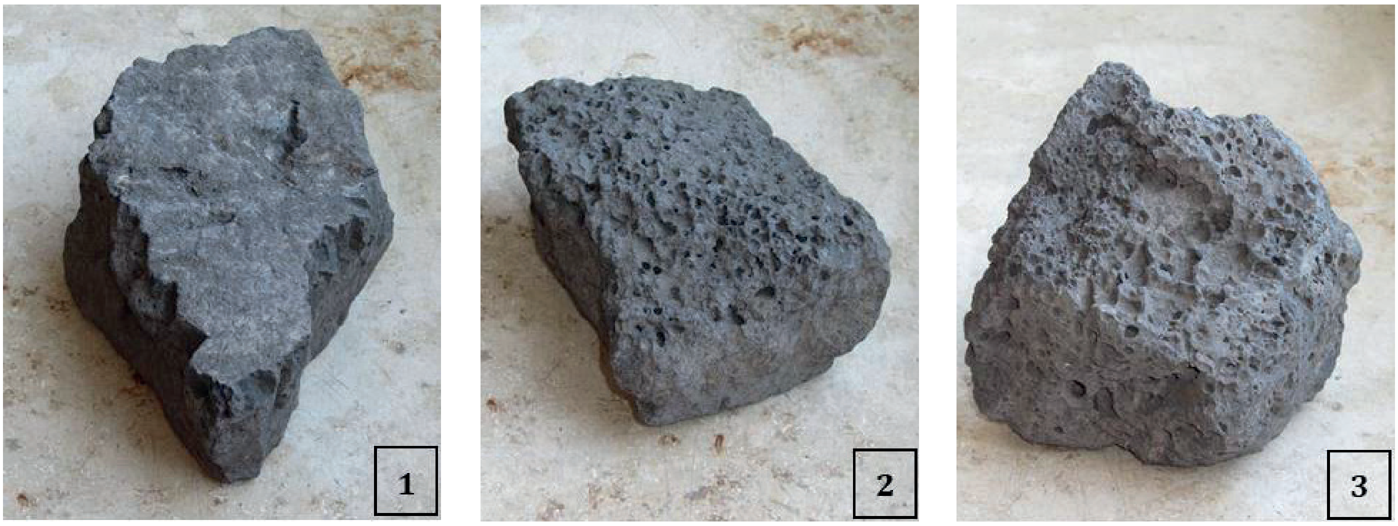


Figure 8: Steel slag samples, homogeneous (1), partly cavities (2) and cavities (3)

In order to assess the applicability of the methods for irregularly shaped samples, a volume determination by immersion weighing according to Archimedes was performed as a reference method. The determination of particle density was carried out in accordance with [6] and the Archimedes volume was calculated by the density. To assess the application limits of the scanners, samples with different colours and textures were selected (Figure 7). The copper slag is homogeneous, dark and partly metallic glossy. By contrast, the dolomite is cream-coloured and bright. Table 2 shows the measurement results.

The dolomite can be detected relatively well by the scanner systems due to the surface colour. As a result of the accuracy of these measuring devices, smaller structures such as the fixing wire of the samples are not visible. When comparing the CT results of the copper slag sample with the other measuring methods, it can be seen that the non-contact scanners show the largest differences. This is probably due to the glossy areas, which are difficult to capture by the scanner because of the reflective beam reflection.

Further measuring tasks were differently shaped and porous steel slag samples (Figure 8). The specimens differed in surface texture and cavity.

Neither the aluminium-foil method nor the scanners were able to detect the cavities. Therefore, the greatest deviation from the CT is found in the sample with the

Sample		Homogeneous 1	Partly cavities 2	Cavities 3
Weight	g	1605	1431	1147
Aluminium-foil method				
Surface	cm²	400	360	400
Archimedes volume	cm³	482	419	401
	cm²/cm³	0.83	0.86	1.0
Laser scanner Leica P30				
Surface	cm²	342	333	344
Volume	cm³	443	436	439
	cm²/cm³	0.77	0.76	0.78
Hand-held scanner Artec MHT 3D				
Surface	cm²	346	359	407
Volume	cm³	422	339	484
	cm²/cm³	0.82	1.06	0.84
CT				
Surface	cm²	663	680	1233
Volume	cm³	448	413	411
	cm²/cm³	1.48	1.65	3.0

Table 3: Results for steel slag

Sample	CT volume [cm³]	CT surface [cm²]	Archime--des volume [cm³]	Aluminium-foil method [cm²]	Laser volume [cm³]	Laser surface [cm²]
Copper slag 1	234.75	256.64	231.90	263.6	230.07	219.81
Copper slag 2	160.86	518.56	164.30	278.2	190.12	218.64
Copper slag 3	302.86	944.46	309.00	385.5	338.26	319.53
Copper slag 4	1922.08	2921.68	1897.40	981.8	1880.61	834.21

Table 4: Copper slag, results of different surface determination methods

greatest porosity (Table 3). A further comparison was made with copper slag samples. Here, too, the specimens differed in surface texture, metallic glossy areas and cavity. The results are shown in Table 4.

Only sample 1 shows an approximate match for the surface between the CT and aluminium-foil method. Sample 3 is a copper slag with large continuous cavities. Therefore, the greatest deviation from the CT is found in this sample. The cavities could not be found in the point clouds of the laser scanner and therefore the results also differ from the CT. Sample 4 shows the largest deviation for the surface. Here, there is an almost threefold deviation.

CONCLUSION AND DISCUSSION

On the basis of the European Products Regulation (CPR) [2] and Basic Requirement No 3 (hygiene, health and environment), the possible release of regulated dangerous substances must be considered for harmonised standards in the future. A horizontal test method for leaching [3] was developed and the expression of the results must be surface related. For products with irregular side areas such as aggregates, it is not possible to calculate the surface in an easy way. Therefore, standardized test methods are necessary. The paper shows the application of the test methods of aluminium-foil method, optical scanner and computed tomography (CT) in comparison. Today CT is less expensive and service providers can be found all over in Europe.

In the case of irregularly shaped aggregates, the error potential of CT systems can be evaluated by comparison with volume determination according to Archimedes. Metallic glossy or porous samples, e.g. aggregates made of slag, can be a problem for optical scanner systems. With CT it was easy to detect the real surface of these materials.

The error possibility / measuring accuracy of the aluminium-foil method depend on the size and kind of stone. This method is not useful for stones with cavities, such as slag materials or shell limestone and also material with coarse-grained mineral structure. Railway ballast can be a problem for this method due to its size.

In one sample, an almost threefold deviation was found between CT and the aluminium-foil method. The comparison with the calculation of geometrically simple specimens and the volume determination by immersion weighing has shown that CT is the appropriate method for the surface determination of aggregates. It must be considered that the DSLT [3] requires the surface twice and therefore an inaccuracy and errors in the measurement method can significantly affect the leaching result. <<<

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EUROSLAG CONFERENCE 2021

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EUROSLAG CONFERENCE 2021

The 11th EUROPEAN SLAG CONFERENCE will take place next year at the Maritim Hotel in Cologne from 5th to 8th October 2021! Two years after we last met in this context in Thessaloniki, Greece. During this time, the slag value chain has further developed so that the need for discussion of the issues is great. Under the title "The Transformation of the steel industry and its effects on the slag value chain", an ambitious programme with lectures, three sessions and discussions awaits all participants in the Rhineland metropolis. We are looking forward to a great conference.

FURTHER INFORMATION FROM

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A NEW PERFORMANCE TEST METHOD TO EVALUATE THE SULPHATE RESISTANCE OF CONCRETE

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INTRODUCTION

Concrete structures can be damaged by sulphates in groundwater and from surrounding rock layers if they do not have a sufficient resistance. This attack is described as exposure class XA in DIN EN 206-1. Minimum requirements for concrete composition, such as a minimum binder content, a maximum accepted water-cement ratio (w/c ratio), accepted types of cement and additives as well as other protective measures, if necessary, are described in DIN 1045-2, the German application document for DIN EN 206-1. The mechanisms that trigger damage because of a sulphate attack have been extensively investigated, e.g. in [1-6].

A number of different methods is available worldwide for evaluating the sulphate resistance of cement and concrete. Depending on the method, the tests are carried out on cements, mortars or concretes that are completely, partly or cyclically immersed in sulphate solutions of various concentrations. The test temperatures also vary widely. In Germany, common test methods for evaluating the sulphate resistance of binders are the so-called SVA (Sachverständigenausschuss) method of the DIBt (Deutsches Institut für Bautechnik), the Koch-Steinegger method [7] and the Wittekindt method [8]. Despite intensive research, all of them show considerable scattering of the measurement results and test artifacts, which can be attributed, for example, to extremely high sulphate concentrations of the test solution that are not related to practical applications. In addition, the physical resistance – the structural density of the concrete – was deliberately neglected in these procedures. For this reason, none of the methods has been included in German or European standardization so far [9, 10].

For reasons of sustainability, new types of cements, additives and concretes with lower ecological footprints will continue to be developed. 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 with which the sulphate resistance of a concrete can be unerringly evaluated.

OBJECTIVES AND REALIZATION

The main objective of the research project, which was carried out in close co-operation with the Institute for Building Materials Research at the RWTH Aachen University (ibac), was the development of a practical test method to determine the sulphate resistance of concrete in a precise and selective manner within an appropriate test period [11]. For this purpose, 3 essential questions had to be answered:

(1) What are the test constraints that can be used to accelerate the

	Influence variable	Unit	Variation
1	Binder type	-	CEM I; CEM I–SR3; CEM I + FA; CEM II/B-S; CEM III/A
2	Testing temperature	°C	5, 12, 20
3	Concentration of sulphate solution	mg/l	3000, 6000
4	Type of sulphate solution	-	Na^{+}, Mg^{2+} as cation
5	Cement content Content of cement + fly ash *	kg/m³	320, 360, 400 270 + 90, 285 + 94, 300 + 100
6	Equivalent water-cement-ratio w/c_{eq}	-	0.45, 0.50

** Practical setting off the fly ash to the w/c_{eq} with k = 0.4*

Table 1: Influencing variables on the sulphate resistance and variation parameters

	No.	CEM I 42.5 N (2)	CEM I 42.5 N-SR3 (5)	CEM II/B-S 42.5 N (14)	CEM III/A 42.5 N (15)	FA 1 * (21)
Compressive strength after	2 d	28.5	24.5	22.2	18.7	24.4
	7 d	46.6	42.5	41.1	40.8	40.1
	28 d	61.9	50.3	61.8	61.9	51.9
	91 d	71.0	60.8	73.3	73.9	65.4

** Combination of 25 wt.-% FA and 75 wt.-% CEM I 42.5 R*

Table 2: Strength development of cements and fly ash

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 outsourcing tests?

(3) Is there a limit value to differentiate between concretes with high and insufficient sulphate resistance?

At the beginning of the project the parameters influencing the sulphate resistance were systematically varied. Furthermore, their influence on various parameters characterizing the microstructure was determined, thereby using experience gained from currently used methods as well as the findings from the state-of-the-art reports of DAfStb [9, 12, 13] and CEN/TC 51 [10]. In contrast to the common test methods, however, the actual performance of the concrete was the focus of the evaluation. This means that not only the chemical but also the physical resistance of the concrete to sulphate attack should be taken into account, since both partial resistances are important for the durability of the concrete under practical conditions.

Based on the statistical evaluation of the results, a test procedure could be defined. It was used to test another approx. 25 concretes. These concretes contained binders with both high and low sulphate resistance. At the same time, several of these concretes were stored under practical conditions at two different sites for at least one year. The evaluation of all results made it possible to propose an acceptance criterion for the test procedure with which the sulphate resistance of a tested concrete can be evaluated reliably.

EXPERIMENTAL SETUP

Statistical design of experiments (DoE)

In order to consider a wide as possible test matrix, statistical methods of design of experiments and evaluation were used intensively. The influencing variables included into the investigation are listed in Table 1 [11].

A first general full-factorial experimental design with a total of 180 experiments was created covering all binders and a constant w/c_{eq} at 0.45. From this, individual combinations were selected by the DoE software in such a way that an optimal experimental design with 100 experiments was created. Methods of sequential optimization were used and terms up to the 2nd order were considered. 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 [11].

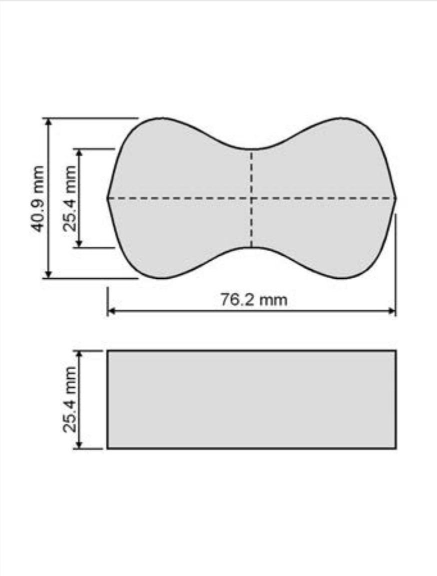

Figure 1: Concrete test specimens acc. to ASTM C307-03

Concrete making and storage

With the cements or cement-fly ash combination mentioned in Table 1, fine concretes with a maximum aggregate grain size of 8 mm were produced in accordance with DIN EN 12390-2 for the parameter study. While the 4 commercially available cements complied with DIN EN 197-1, the hard coal fly ash (FA) was in line with DIN EN 450-1. The strength development of the binders is shown in Table 2.

For testing the flexural tensile strength and the dynamic modulus of elasticity prisms with the dimensions 40 x 40 x 160 mm³ were used. For testing the tensile strength bone specimens (Figure 1) according to ASTM C307-03 were produced. All specimens were demoulded after one day and then stored in saturated $Ca(OH)_2$ solution at 20 °C for 27 days.

Execution of tests and test parameters

The test started at a concrete age of 28 days. The specimens were stored in sulphate 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. The results were related to reference values, resulting in relative values that can be directly compared. The 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 sulphate storage. Furthermore, a maturity function was used for the bending and tensile strengths. The basis was the function described in the fib Model-Code [14]. It was adapted to the influence of supplementary cementitious materials on the strength development of concrete, as proposed by Vollpracht et al. [15]. Since the adapted method was originally developed for the prediction of compressive strength, its suitability was tested in advance for bending and tensile strengths [11].

RESULTS AND DISCUSSION

Selection of suitable test criterions

The different relative test parameters were analyzed in terms of their significance for the test procedure. Damage to concrete due to sulphate attack was best characterized by the tensile strength of ASTM briquettes. 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 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.

Selection of the test parameters and definition of the test procedure

The evaluations of the parameter study provided 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 in Figure 2 for fine concretes with the 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 sulphate solution on the expected relative tensile strength. As a result, the lowest relative tensile strength can be

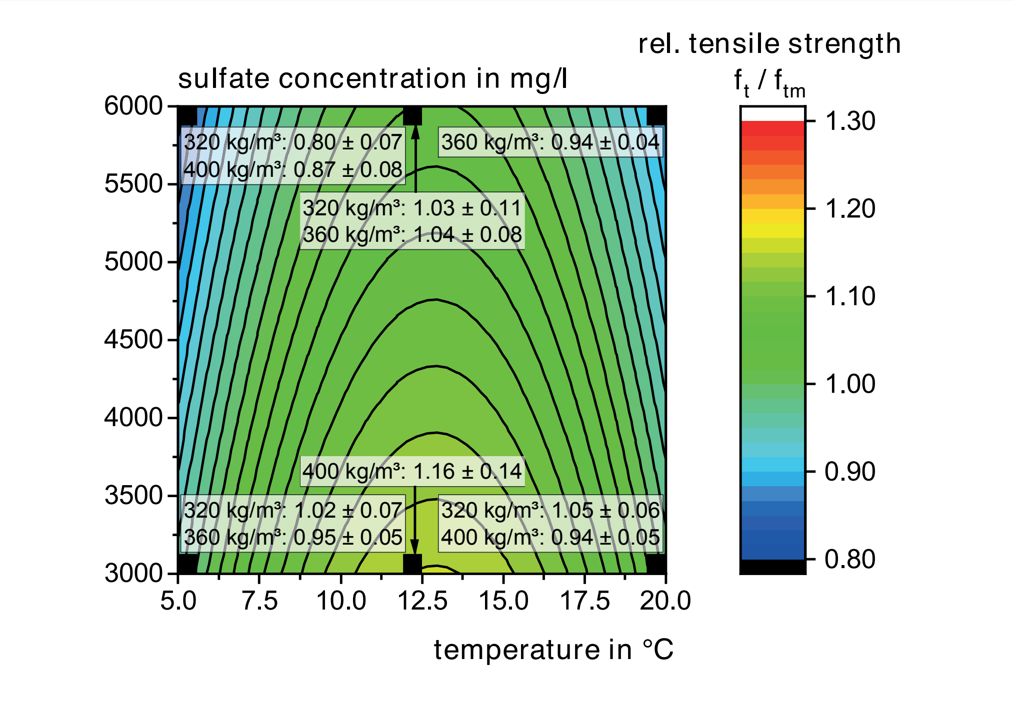

Figure 2: Contour diagram of relative tensile strength f_t/ f_{tm} of concretes with (2) CEM I 42.5 after 182 days storage in Na_2SO_4 solution, reference: fib maturation function

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	■ Briquettes 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, practical test method for evaluating the sulphate resistance of concrete

expected if the concrete is tested at 5 °C and 6000 mg/l SO_4^{2-} concentration. The figure also contains the results of 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, the relative tensile strength f_t/f_{tm} at a test age of 273 days decreased by 0.10 - 0.15 when the sulphate concentration was increased from 3000 mg/l to 6000 mg/l (see Figure 2). At the same time, no excessive gypsum formation was observed in the pore space of the specimens. Consequently, the increase of sulphate concentration to 6000 mg/l accelerates the test and does not cause artefacts, especially for less sulphate-resistant concretes. The test procedure could be described as a result of this evaluation. Its main features are summarized in Table 3.

Storage under practical conditions

A large number of fine concretes as well as normal concretes – the latter one fulfilled the minimum requirements of DIN 1045-2 for the composition of exposure class XA2 – was stored under practical conditions at two sites. Figure 3 shows the exposure site in a

Figure 3: Storage site in a German gypsum mine

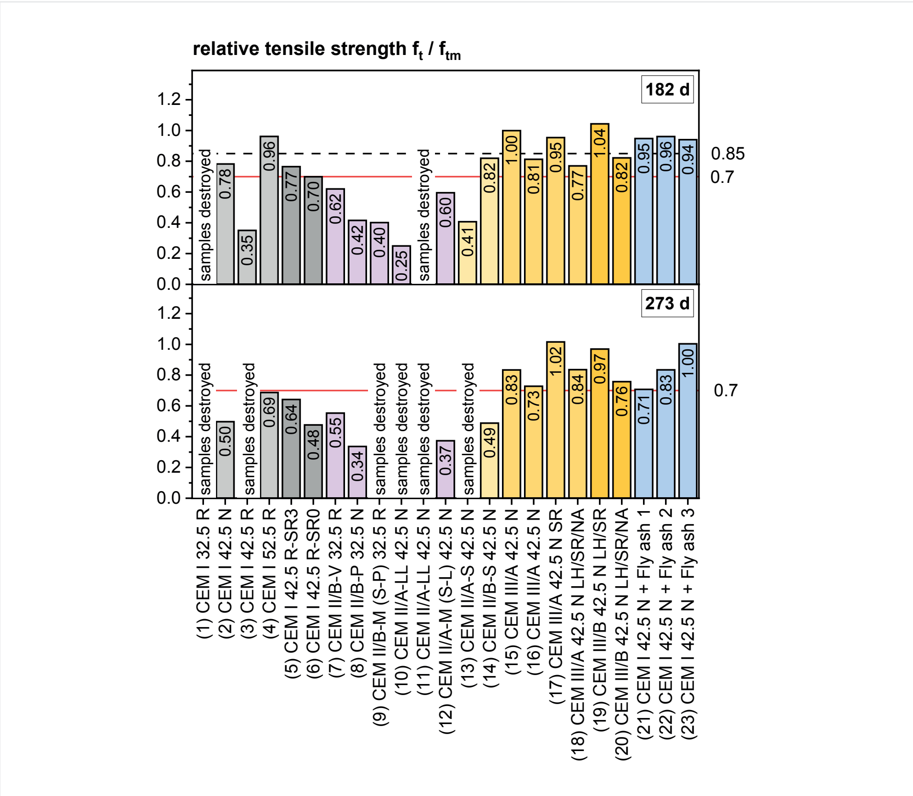

Figure 4: Relative tensile strength f_t/f_{tm} of concrete with 23 different binders after 182 and 273 days

gypsum mine. The laboratory results obtained with the new method will be verified by long-term tests with a real sulphate 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 expected, the storage time was too short to induce damage even on concretes that experience has shown to have insufficient sulphate resistance. The trials under practical conditions will be continued after the end of the project.

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 sulphate 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 4 illustrates the relative tensile strengths of the 23 concretes after 182 and 273 days of sulphate storage.

After 119 days of storage no reliable statement can be made on the sulphate resistance. The first differences between concretes with different binders did not occur before 182 days of storage. However, a definitive differentiation was not yet possible between concretes with known high or low sulphate resistance. Some concretes made with Portland cement without SR property showed residual tensile strengths comparable to those of some slag cement concretes, for which a high sulphate resistance can be expected in the light of experience. After 273 days (9 months) of sulphate storage, it was possible to make a clear distinction regarding sulphate 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 sulphate 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. A sufficient C_3A content of the clinker was proved by x-ray diffraction for all these cements. Presumably, it is sufficiently high to trigger a damaging ettringite reaction with the sulphate ions in the cement stone structure.

Under consideration of the discussed results, a concrete has a sufficient high sulphate resistance if its relative tensile strength is not lower than 0.70 (70 %) after 273 days of sulphate storage. Furthermore, two stop criteria can also be defined for the test after 182 days. Firstly, a concrete has a high sulphate resistance if its relative tensile strength f_t/f_{tm} is higher than 0.85, since the criterion is also met after 273 days. Secondly, a low sulphate resistance is already proven if the relative tensile strength f_t/f_{tm} is lower than 0.70.

CONCLUSION

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 sulphate resistance. In the development of the test method, the recommendations of the state-of-the-art report [9] were taken into account and the tensile strength was determined as the best test parameter [11].

Based on the statistical evaluation of approx. 3850 tensile tests on ASTM briquettes, 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 sulphate attack,
- considers not only the chemical, but also the physical resistance of a concrete against sulphate attack,
- leads much faster to an evaluation of the sulphate resistance compared to the common SVA method (current regulation: 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).

ACKNOWLEDGEMENT

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. <<<

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PRODUCTION AND USE OF IRON AND STEEL SLAG IN GERMANY AND EUROPE IN 2019

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GERMANY

For many years, ferrous slag (blast furnace slag and steel furnace slag), which is obtained as a by-product in the production of iron and steel, has been established on the market as a proven building material and fertilizer. Data on production and use in Germany in 2019 are given in Tables 1 (blast furnace slag) and 2 (steel furnace slag). For comparison, the data for 2018 are also shown.

In 2019, crude steel production in Germany fell below the 40-million-ton mark for the first time since the crisis year 2009 (39.7 million tons, 2018: 42.4 million tons) [1]. The production of by-products also declined accordingly - both blast furnace slag and steel slag were down in 2018. In 2019, a total of 12.3 million tons of ferrous slag was produced (2018: 13.1 million tons). Of this figure, 11.4 million tons were used in the various fields of application as building materials and fertilizers in 2019. This corresponds to a continued high usage rate of 93 %. Only 0.7 million tons of slag were disposed of. On balance, inventories at the interim storage facilities were increased by 0.3 million tons.

Production	2019	2018
Granulated BFS	**6.48**	6.96
Air-cooled BFS	**0.82**	0.82
Sub-total	**7.30**	7.78
From interim storage	**0.74**	1.04
Total	**8.04**	8.82

Utilisation	2019	2018
ABS for aggregates	**0.13**	0.29
ABS for aggregate mixtures	**0.51**	0.60
GBS for cement production	**7.15**	7.70
GBS for other purposes	**0.11**	0.11
Intra-industry consumption	**0.14**	0.12
Total	**8.04**	8.82

Table 1: Production and use of blast furnace slag in Germany 2019/2018 (in million tons)

Production	2019	2018
Slag from oxygen steel making	**3.02**	3.23
Slag from electric arc steel making	**1.47**	1.54
Others (SecMS etc.)	**0.52**	0.53
Total	**5.01**	5.29

Utilisation	2019	2018
Metallurgical use	**0.56**	0.61
Fertilizer	**0.40**	0.39
Construction material	**2.12**	2.25
Others	**0.27**	0.41
Sub-total	**3.35**	3.66
Final deposit	**0.66**	0.66
To interim storage	**1.00**	0.97
Total	**5.01**	5.29

Table 2: Production and use of steel furnace slag in Germany 2019/2018 (in million tons)

Looking at the data in detail, in the case of blast furnace slag (BFS), the granulation rate, i.e. the proportion of granulated blast furnace slag in blast furnace slag, is about 90 % of the total quantity. Consequently, the use of granulated blast furnace slag in the cement industry remains at a high level, even though the absolute value has declined considering the reduced production volume (7.2 million tons, 2018: 7.7 million tons). The production of CEM II and CEM III cements is the most important sales area for blast furnace slag, which thus also makes an important contribution to CO_2 reduction. In absolute figures, the use of air-cooled blast furnace slag as an aggregate or aggregate mixture to produce asphalt, concrete and base courses without binders decreased to 0.6 million tons (previous year: 0.9 million tons). The decrease would have been even more significant if the amount of blast furnace slag temporarily stored for later use could not have been further reduced.

In 2019, a total of 5.0 million tons of steel furnace slag (SFS) was produced in Germany. This corresponds to a year-on-year decrease of 0.3 million tons. Comparing the use in the different application areas only few changes can be seen in the use as lime and iron carriers in the metallurgical cycle (0.6 million tons, approx. as in the previous year) and in the use as lime fertilizer (0.4 million tons, almost identical to the previous year). The amount of material deposited has also remained more or less constant (see above). In contrast, there has been a slight decline in the use as a construction material (2.1 million tons, previous year: 2.3 million tons) and as simple bulk material without formal monitoring (0.3 million tons, previous year: 0.4 million tons).

EUROPE

The European slag association EUROSLAG regularly asks its members for European slag data. Preliminary figures for 2019 are given in Tables 3 (blast furnace slag) and 4 (steel furnace slag). However, these figures must be qualified as it has not yet been possible to obtain a satisfactory number of responses, perhaps due to the corona pandemic situation.

Based on the hot metal and crude steel production provided by Worldsteel [2], a rough estimation of BFS production and SFS production can be made. This leads to about 24 million tons of BFS slag and 21 million tons of SFS slag produced in the EU-28 countries during the year 2019. The reported data provided in Tables 3 and 4 present about 50 % of these figures.

It may therefore be wise to rely not on the figures, but rather on the percentages. These percentages are quite close to the percentages reported from Germany: 90 % of the blast furnace slag is granulated and subsequently 90 % of it is used for cement production. Only a very small amount is used for purposes such as fertilising and glass production.

As far as the SFS figures are concerned, 45-50 % of this slag is produced using the converter process (this percentage is probably larger in reality). 40-45 % is produced using the electric arc furnace process of which 3/4 arises from carbon steel production and 1/4 from stainless/high alloyed steel production. About 70 % of the SFS produced is used for construction purposes, for roads, dams, and hydraulic structures, for example. Some 10 % is used as fertilizer and for metallurgical purposes respectively.

Roughly 15 % of the SFS produced was deposited in landfills and about 15 % was taken to storage heaps for future use. These amounts should be reduced in future but it can generally be stated that, based on the figures received, ferrous slag products are marketed with success in Europe even if there is potential for improvement. <<<

Production	t million
Granulated BFS	10.55
Air-cooled BFS	1.20
Sub-total	11.75
From interim storage	0.72
Total	**12.47**

Use	t million
Cement production and concrete additives	11.00
Road construction	1.02
Others	0,23
Sub-total	12.25
Final deposit	0.22
Total	**12.47**

Table 3: Production and use of blast furnace slag in Europe 2019 – preliminary figures as of October, 29th, 2020

Production	t million
Slag from oxygen steel making	4.87
Slag from electric arc steel making	4.46
Others (SecMS etc.)	1.01
Total	**10.34**

Use	t million
Metallurgical use	0.82
Fertilizer	0.55
Construction material	5.20
Others	0.84
Sub-total	7.41
Final deposit	1.49
To interim storage	1.44
Total	**10.34**

Table 4: Production and use of steel furnace slag in Europe 2019 – preliminary figures as of October, 29th, 2020

Source: EUROSLAG e. V., Duisburg

LITERATURE

[1] https://www.stahlonline.de/wpcontent/uploads/2020/04/Rohstahlproduktion_in_Deutschland_April2020.png
[2] https://www.worldsteel.org/en/dam/jcr:f7982217-cfde-4fdc-8ba0-795ed807f513/World%2520Steel%2520in%2520Figures%25202020i.pdf

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Imprint // **Editor:** FEhS – Building Materials Institute // **Responsible:** Thomas Reiche, Managing Director // **Layout:** del din design

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