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EUROPEAN COMMITTEE FOR STANDARDIZATION
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Management Centre: rue de Stassart, 36 B-1050 Brussels

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Foreword

This document (CEN/TR 15697:2008) has been prepared by Technical Committee CEN/TC 51 "Cement and building limes", the secretariat of which is held by NBN.

This CEN/TR is a state of the art review of the international research literature dealing with testing/assessing the sulfate resistance performance of cements and related binders. It outlines the difficulties faced by CEN/TC 51 in applying a prescriptive approach to the specification of sulfate resistant cements and identifies the different mechanisms and forms of deterioration that occur during sulfate attack. This report compares the advantages and disadvantages of different test specimen types (paste, mortar or concrete), different exposure conditions and different techniques used to assess specimen deterioration. The importance of test method reproducibility is reviewed with reference to the experimental work carried out by CEN/TC 51 during the 1990s. The report lists the key parameters that must be controlled in any robust standardised method and makes suggestions for the main features of a pan-European performance test.

Introduction

Under the terms of EU Mandate 114, committee CEN/TC 51, cement, building limes and other hydraulic binders, is required to develop standards for 'common cements' and also for cements with special properties such as low heat cements, calcium aluminate cements and sulfate resisting cements.

EN 197-1: Composition, specifications and conformity criteria for common cements was adopted in 2000 and was the first harmonised European Standard to be adopted for a construction product.

Since 2000, European Standards for masonry, low heat, and low early strength blastfurnace cements, very low heat special cements and calcium aluminate cements have been published. The development of a prescriptive EN for sulfate resisting cements has been complicated by national differences in the types of cement that are recognised to have sulfate resisting properties. Note, however, that all nationally standardised sulfate resisting cements meet the requirements of EN 197-1:2000 and that the absence of a specific standard for sulfate resisting cement has not constituted a barrier to trade.

In order to overcome these national difficulties, and also to permit new types of cement to be recognised in the future, work was directed towards the development of a performance test for sulfate resistance. Work commenced in 1991 and following a preliminary assessment of the French NF-P-18-837 procedure and the German, so called flat prism method, a decision was taken to concentrate on developing the French procedure. The method measures the expansion of 20 mm x 20 mm x 160 mm prisms in a sodium sulfate solution containing 16 g/l SO_4^{2-}.

During five co-operative testing exercises involving up to thirteen laboratories, the method was refined with the objective of improving reproducibility and also discrimination between sulfate resisting and non-sulfate resisting cements. In 1998 it was concluded that further development would require a more fundamental approach and efforts were directed towards obtaining EU funding for 'pre-normative' research. These applications were not successful.

In early 2004 a meeting was arranged with representatives of the NANOCEM programme to explore a more fundamental approach to the problem of sulfate resistance and sulfate resistance testing. The aspects of particular interest to CEN/TC 51 were:

a) understanding sulfate attack mechanisms in relation to the type of cement and the concentration/temperature conditions;

b) establishing a relationship between laboratory tests and field performance;

c) methods to accelerate the test;

d) using parameters other than deformation measurement to monitor the progress of the sulfate attack;

e) understanding the role of thaumasite in sulfate attack.

The NANOCEM group has formulated a research programme that addresses the above aspects and work on this programme commenced in 2006 within the framework of a larger programme funded by the Marie Curie Training Network. In parallel with this programme, CEN/TC 51 asked committee WG 12 (Additional Performance Criteria) to prepare a CEN Technical Report outlining the current state of the art concerning sulfate resistance testing.

A literature search identified over 250 relevant papers and reports published during the period 1970 to 2006. To assess the different sulfate resistance techniques employed and their possible influence on the performance of different cement/binder types, the testing details from 129 papers were entered into an Access Database. The papers selected for entry into the database were those which contained original research data and detailed information concerning test conditions.

This report draws on the information contained in these 129 papers plus a further 50 papers and reports not selected for entry into the database. In the interests of brevity the current report only includes references to selected references that are either key papers or contain specific information. It is intended that a statistical analysis of the database and a full listing of the papers studied will be made available as a supplementary document of CEN/TC 51 / WG 12.

1 Sulfate resistant cements

Portland cement concrete can undergo attack by sulfate bearing solutions such as natural groundwater or those contaminated by industrial activity. Attack can result in expansion, strength loss, surface spalling and ultimately disintegration.

The resistance that a cement matrix provides to sulfate attack depends on a number of factors which include:

- nature of the reaction products formed with the sulfate solution and in particular, whether their formation results in disruptive expansion;
- impermeability of the matrix (including the important paste-aggregate interfacial zone) which provides a barrier against penetration by sulfate ions;
- concentration of sulfate ions (in this report expressed as g/l SO_4^{2-});
- mobility of the sulfate containing groundwater;
- nature of the accompanying cation e.g. Na^+, Mg^{2+}, Ca^{2+} etc;
- pH of the sulfate bearing ground water/solution;
- presence of other dissolved salts such as chlorides;
- temperature of the exposure;
- degree of pre-curing before exposure, although in the field this is only likely to affect the performance of the concrete surface;
- presence of finely divided limestone (calcium carbonate) in the aggregate, or carbonate ions dissolved in the groundwater, which may promote the formation of thaumasite under low temperature conditions.

Almost all developed countries have product specification standards for sulfate resisting cement(s). With a few exceptions these are prescriptive standards that specify cement composition. The permitted compositions are based upon long-standing laboratory test results and also satisfactory performance in the field. National differences reflect different exposure conditions and also differences in the nature of the available cement constituents.

Poor performance under sulfate exposure conditions is normally associated either directly or indirectly, with the formation of ettringite. In the hydrated matrix of a CEM I cement, the source of reactive alumina is normally the monosulfate phase according to the reaction:

$$\underset{\text{(monosulfate)}}{C_4Al_2(OH)_{12}.SO_4.6H_2O} + 2Ca^{2+} + 2SO_4^{2-} + 20H_2O \Rightarrow \underset{\text{(ettringite)}}{C_6Al_2(OH)_{12}(SO_4)_3.26H_2O}$$

Any unreacted C_3A is also a potential source of ettringite.

Monosulfate will normally also be present in composite cements containing blastfurnace slag, fly ash or natural pozzolana but in the hydrated matrix of these cements, alumina is also present in phases such as hydrotalcite or hydrogarnet or substituted in C-S-H in which latter forms. It does not appear to be available to form an expansive reaction product [1].

Strength loss and disintegration are also associated with decalcification of C-S-H, which is an important mechanism during attack by $MgSO_4$ solutions but which also occurs to a lesser extent in Na_2SO_4 solutions [2].

Current sulfate resisting cements standardised in CEN member countries can be divided into two categories:

1) Portland (CEM I) cements with a maximum permitted C_3A content.

2) Portland composite cements containing appropriate levels of glassy blastfurnace slag, fly ash or natural pozzolana.

Low C_3A sulfate resistant cements provide a chemical resistance to sulfate attack. The products that reaction with sulfates is not expansive and consequently the matrix is not disrupted facilitating further attack. The unreactive nature of the hydration products of low C_3A cements is attributed to a low level of monosulfate and/or the formation of an iron-rich form which is slow reacting and produces a 'non-expansive' form of ettringite [3].

Portland composite cements (i.e. CEM II, III, IV and V types) provide resistance to sulfate attack which is predominantly micro-structural in nature [4 to 8]. This is derived from the significantly lower permeability of the hydrated matrix. Additional positive factors are:

- reduced level of free calcium hydroxide in the matrix which reduces calcium availability for ettringite formation and also the formation of gypsum when the matrix is exposed to concentrated sulfate solutions;

- formation of hydrates containing alumina which are non-reactive to sulfate solutions.

The reduced availability of calcium may also result in the formation of ettringite with a morphology and distribution throughout the hydrated matrix which is not expansive [9].

One factor that is often overlooked is that resistance to external sulfates is normally positively influenced by the level of SO_3 in the binder; the higher the level in a range between ~ 1 % to ~ 4 %, the greater the resistance. This applies to concrete produced from CEM I cements [10] and also particularly to slag and fly ash containing concretes. Where the ash or slag is added to the mixer [2], [11, 12] the SO_3 level is lowered by dilution and the hydrated matrix is more vulnerable to attack by penetrating sulfates in comparison with a binder with an optimised SO_3 level. The improved resistance can be attributed to the increased level of sulfated phases, such as ettringite, formed during initial hydration, which are stable in the presence of an elevated sulfate level.

2 Sulfate resistance test procedures

2.1 General review

The sulfate resistance properties of cement can be assessed by preparing realistic concretes specimens and placing them in conditions which are representative of field conditions. Unfortunately, unless the concretes are of low quality (high w/c, poorly compacted) several years exposure will be required to provide any meaningful discrimination between sulfate resistant and non-sulfate resistant cements [13, 14]. Consequently, there is a need for accelerated test procedures that provide discrimination within a timescale of weeks or months.

The first laboratory test procedure to determine the sulfate resistance properties of a cement was the Le Chatelier - Anstett procedure, [15] in which cement paste is hydrated, crushed and dried and then interground with 50 % (by mass) of gypsum. The expansion of a moist cylinder, formed from the interground mixture is determined at 1 day, 28 days and 90 days. The method is severe and cements with a low potential to form expansive products such as calcium aluminate cement and supersulfated cement perform well, while low C_3A sulfate resisting Portland cements perform poorly.

The first test procedure to attain the status of a national standard was the ASTM C 452 procedure, which was adopted in 1964. In this test, cement is blended with finely divided gypsum to bring the SO_3 level to 7,0 % and the expansion of 25 mm x 25 mm x 285 mm mortar bars (1:2,75, w/c 0,485) placed in water at 23 ^{0}C is determined at 14 days. ASTM C150, Specification for Portland cement, permits a cement to be classed as a Type V sulfate resistant cement if expansion at 14 days is less than 0,040 %. This is an optional requirement and cement can be classified as Type V if the C_3A content is less than 5 % (and the sum of $C_4AF + 2C_3A$ is less than 25 %). The method is not suitable for cements containing constituents such as blastfurnace slag and pozzolanas as firstly, the short timescale of test does not permit adequate hydration of the secondary constituent and secondly the sulfate attack is 'internal' and does not take into account the reduced permeability associated with constituents such as slag and pozzolanas.

Current thinking regarding accelerated test procedures [16, 17] is that the mechanism of deterioration in the accelerated test should be representative of those observed in service. Neither the Le Chatelier- Anstett nor the ASTM C 452 test procedures meet these criteria and they are not suitable for the assessment of cement with secondary constituents.

Accelerated tests that provide a realistic mechanism of deterioration should take into account:

- resistance to penetration by sulfate solutions (impermeabilty) provided by the cementitious matrix;
- degree of curing that can be expected before the matrix is subjected to a critical level of attack, as opposed to superficial surface damage; and
- sulfate environment, in terms of sulfate ion concentration, pH and temperature, which should not be too far removed from conditions likely to be encountered in the field.

Accelerated sulfate resistance tests may be carried out using the following types of cementitious matrix:

- paste;
- mortar;
- concrete.

The advantages and disadvantages of these three test matrices are summarised in Table 1.

Table 1 - Advantages and disadvantages of different cementitious matrices for sulfate resistance testing

Matrix	Advantages	Disadvantages
Paste	Small specimen size reduces space requirements. Can be applied worldwide without need to obtain suitable aggregate. Test results are independent of aggregate type Samples can be examined using techniques such as chemical analysis, XRD, SEM etc without dilution by aggregate.	Unless low w/c is used, or other measures adopted, such as agitation during setting, bleeding will result in heterogeneity. A low w/c (< 0,35) is likely to result in an extended testing time. Does not include the important aspect of permeability of the paste aggregate interface. Water requirement of paste does not relate very well to water demand in mortar or concrete.
Mortar	A well characterised single source of test sand can be used by all laboratories within a large geographic area. Specimen size can be small thus accelerating the test and reducing the size of storage tanks required etc. Most laboratories will have test equipment suitable for the strength testing of mortar specimens. Mortar proportions can be adjusted and w/c increased to accelerate the test without unacceptable bleeding occurring. Mortar may be gauged to constant workability/flow.	May be criticised for ‘not being concrete’.
Concrete	Results may be more directly applicable to field concrete. Concrete may be gauged to constant workability to take into account the water demand characteristics of cement or addition.	Large specimens are required in order to accommodate coarse aggregate. Space requirements for specimens storage are high. Duration of test will be long as a result of large specimen cross section. Impractical (and uneconomic) to use a standardised aggregate thus reducing reproducibility of test procedure. Large capacity test machines required for strength testing.

From Table 1, it can be seen that mortar testing offers practical advantages when seeking to develop a standardised test procedure for sulfate resistance testing.

Specimens are normally fully immersed in the sulfate solution, but as discussed in section 2.3, procedures have been developed that require specimens to be partially immersed in the solution. Supporters of this approach believe that partial immersion better simulates the mechanism of deterioration which occurs in the field. Section 2.4 briefly reviews procedures in which the specimens have been subjected to alternate wetting and drying in order to accelerate the penetration of sulfate solutions into the specimen.

Several researchers [18 to 20] have found that initial carbonation of specimens markedly improves sulfate resistance. It is likely that the inadvertent and variable carbonation of specimens is responsible, at least in part, for the poor reproducibility of existing sulfate resisting test methods.

The following performance indicators are most commonly used to assess the sulfate resistance properties of a test specimen:

- change in length (linear expansion);
- change in compressive strength;
- change in flexural strength;
- change in mass;
- change in appearance.

Less commonly, resistance has been monitored using non destructive techniques such as elastic dynamic modulus and ultrasonic pulse velocity [21 to 24].

The advantages and disadvantages of the different performance indicators are discussed in section 3.

2.2 Review of test procedures in which specimens are fully immersed in sulfate solution

2.2.1 Mortar tests

Table 2 summarises the main characteristics of mortar test procedures which have either been standardised (ASTM C1012, GOST 4798), have progressed to the status of a draft EN that describes the methods used during the round robin activities of WG 12/TG1, or have been widely used, mainly in Germany (Wittekindt, SVA and Koch Steinegger) [25].

The test procedures share the following characteristics:

- specimens have a high surface to volume ratio;
- with the exception of the GOST test procedure the use of highly concentrated Na_2SO_4 solutions (16 g/l SO_4^{2} to 34 g/l SO_4^{2-};
- replacement of the Na_2SO_4 solution at monthly intervals (apart from the ASTM C 1012 timings which vary according to age);
- use of a nationally (or European) standardised test sand;
- with the exception of the ASTM C 1012 procedure, assessment of sulfate resistance at an early age e.g. 56 days in the Wittekindt test;
- rather poor reproducibility.

The ASTM C1012 test procedure is the only internationally recognised and standardised test procedure. It has undergone a number of modifications since it was introduced in 1984. The early version used a mixed $MgSO_4$ (4,3 %) and $NaSO_4$ (2,5 %) solution. In 1987 this was replaced with a 5 % Na_2SO_4 solution as it was considered that the mixed solution gave confusing results. The procedure also differs in that the test bars are cured for 24 h at a temperature of 35 °C and subsequently in limewater at 23 °C. The bars are placed in the

5 % Na_2SO_4 solution when a compressive strength of (20 ± 1) MPa is achieved by 50 mm mortar cubes cured under the same conditions.

It should be noted that Poland incorporated the draft EN test procedure that describes the methods used during the round robin activities of WG 12/TG1 into their specification for sulfate resisting cement PN-B-19707:2003. Cements are classed as sulfate resisting if expansion is less than 0,5 % at 1 year.

Many researchers have chosen to use mortar test procedures, which do not follow those outlined in Table 2 but utilise ‘standard sized' specimens e.g. the 40 mm x 40 mm x 160 mm prisms used in the EN 196-1 strength test [24 to 28]. These prisms lend themselves to the determination of both flexural and compressive strength and also to expansion if studs are cast into the ends or affixed with epoxy. The larger cross section of the bars does delay deterioration compared to the smaller 20 mm x 20 mm or 10 mm x 40 mm section bars, particularly if the w/c is relatively low.

In all the test procedures outlined in Table 1, the pH of the test solution will vary in a cyclical manner [5, 29]. Several researchers [30, 31, 8] have shown that if the pH of the solution is controlled by means of the addition of dilute sulfuric acid then sulfate attack can be induced within a reasonable timescale without the need to resort to high sulfate concentrations. The increase in pH occurs primarily as a result of the counter-diffusion of OH^- ions from the specimens necessary to maintain electroneutrality as SO_4^{2-} ions diffuse inwards and react with the matrix.

As well as accelerating the test it can be argued that controlling the pH also simulates more closely field conditions where concrete is exposed to a mobile sulfate containing environment. However, it may not model stagnant situations.

Table 2 - Comparison of sulfate resistance tests based on mortar

	ASTM C1012: 2004	round robin	round robin	Wittekindt (VDZ modified)	SVA	GOST 4798	Koch & Steinegger
Test parameter	Expansion	Expansion	Expansion	Expansion	Expansion	Relativeflexural strength	Relative flexural strength
Specimen size (mm)	25 x 25 x 285	20 x 20 x 160	20 x 20 x 160	10 x 40 x1 60	10 x 40 x 160	10 x 10 x 30	10 x 10 x 60
Surface : volume ratio (mm^2/mm^3)	0,17	0,21	0,21	0,26	0,26	0,47	0,43
Proportions **Sand : cement : water**	2,75 :1 : 0,485 Blends with slag or pozzolana gauged to same flow as control PC	3 : 1 : 0,50	3 : 1 : 0,50	3 : 1 : 0,60	3 : 1 : 0,50	1 : 3,5 : 0,40	1 : 3 : 0,6
Sand type/source	ASTM C 109 test sand (Ottawa silica sand)	EN 196-1 Standard sand	EN 196-1 Standard sand	German test sand I and II	EN 196-1 Standard sand	Silica sand 0,4 - 0,5 mm	German test sand I and II (fine and coarse)
Studs	Stainless steel	Stainless steel or brass (projecting or recessed)	Stainless steel or brass (projecting or recessed)	Stainless steel	Stainless steel		
Compaction	Tamping	Jolting (10 jolts)	Jolting (60 jolts)	Vibration	Vibration		Jolting (20 jolts)
Curing	1 day in mould at 35 °C then in water at 23 °C until 50 mm cube strength of 20 MPa	1 day in mould 27 days in water at 20 °C	1 day in mould, 27 days in $Ca(OH)_2$ solution at 20 °C	1 day in mould 13 days in water at 20 °C	2 days in mould 12 days in $Ca(OH)_2$ solution at 20 °C	2 days in humid air, 13 days in water.	1 day in humid air 20 days in water at 20 °C

(continued)

Table 2 *(concluded)*

	ASTM C1012: 2004	round robin	round robin	Wittekindt (VDZ modified)	SVA	GOST 4798	Koch & Steinegger
Solution Type	Na_2SO_4	Na_2SO_4	Na_2SO_4	Na_2SO_4	Na_2SO_4	$(NH_4)_2SO_4$	Na_2SO_4
Solution concentration (g/l SO_4^{2-})	33,8	16 ± 0,5	16 ± 0,5	29,8	29,8	1 and 2	29,8
Solution temperature (°C)	23	20	20	20 (and recently 5)	20		20
Specimen surface : solution volume ratio (cm^2/l)	461 to 358	408	408				
Frequency of solution replacement	1, 2, 3, 4, 8 ,13, 15 weeks, 4, 6, 9, 12 months	Every 28 days	Every 28 days	Every 28 days	Every 28 days	Every 30 days	
pH controlled	No	No	No	No	No		
Test limit	High sulfate resistance < 0,10 % expansion at 12 months	Not established	Not established	≤ 0,5 % expansion at 56 days	≤ 0,5 % expansion at 91 days	Flexural strength > 80 % of control at 1, 2, 4, 6 months	Flexural strength >70 % of control at 77 days
Coefficient of repeatability (%)	15 to 22	> 10	10	Satisfactory	Satisfactory		
Coefficient of reproducibility (%)	25 to 40	> 50	20 to 50	poor	poor		

2.2.2 Cement paste tests

Cement paste specimens have most frequently been selected by researchers who wish to examine the specimens using techniques such as SEM and XRD [32 to 35].

A paste test procedure using 12,7 mm cubes and a pH controlled sulfate solution was first proposed by Mehta and Gjorv in 1974 [36]. A relatively high w/c of 0,5 is used and bleeding is minimised by mixing the paste using a high speed agitator. To promote the hydration of additions such as slag and fly ash, the pastes are cured at 50 °C for 7 days before immersion in a Na_2SO_4 solution with 27,1 g/l SO_4^{2-}. In the initial work the pH was controlled at 6,2 using 0,1N H_2SO_4 but in more recent work [37] the procedure was modified to make it less aggressive. The paste cubes are cured at 40 °C for 14 days, the solution pH is neutral pH (7 ± 0,5) and the sulfate concentration is reduced to 13,6 g/l. The pH adjustment may be achieved using automatic titration equipment linked to a pH meter.

Sulfate resistance is determined by comparing the mean compressive strength of 10 paste cubes after 28 days immersion in the sulfate solution with that of 10 cubes broken at the end of the initial curing period. A strength loss of 25 % is rated as satisfactory.

Chemical analysis and X-ray diffraction of cement pastes tested using the Mehta procedure with a SO_4^{2-} concentration of 27,1 g/l, showed considerable gypsum formation but only traces of ettringite [38]. The authors conclude that the test is based on the reaction between free lime and sodium sulfate. However, the evidence available in the literature [9] indicates that ettringite causes the initial disruption and that this facilitates further reactions, which can in some cases be more damaging. Ettringite is stable at a pH of 11,5, unstable at pH 10 and highly unstable at a pH of 6 [29]. Thus, although ettringite is almost certainly formed at the reaction front in the interior of the specimen, where the pH will be in excess of ~12, as the reaction front progresses and the pH is reduced in this region (as a result of the controlled pH of the external solution) the ettringite will decompose and the dominant reaction product will be gypsum. All published data from tests using a controlled pH sulfate solution show the expected relationship between CEM I C_3A content and sulfate resistance.

An assessment of the method alongside the Wittekindt and Koch Steinneger method [39] found reasonable agreement between the three methods. The test that produced the most consistent results was an extended version of the Wittekindt method.

As indicated in Table 1, paste test procedures have a number of disadvantages which include the absence of aggregate paste interfaces which provide one of the main pathways by which aggressive solutions enter concrete or mortar.

2.2.3 Concrete tests

As summarised in Table 1, concrete based sulfate resistance test procedures suffer from a number of practical disadvantages.

The test procedure which comes closest to the status of a standardised method is that developed by the US Bureau of Reclamation (USBR 4908). The procedure utilises 150 mm x 300 mm concrete cylinders and changes in length and mass are monitored under three sulfate exposure conditions:

Method A: immersion in a 14 g/l Na_2SO_4 solution;

Method B: immersion in a 68 g/l Na_2SO_4 solution;

Method C: alternate immersion in a 14 g/l Na_2SO_4 solution for 16 h and forced air drying for 8 h at 54 °C.

Even the more aggressive test method B and test method C require at least 1 year to 2 years before significant results can be obtained [6].

At the Building Research Establishment in the UK the sulfate resistance properties of cement and cement-addition combinations has been assessed utilising 100 mm concrete cubes. Concrete deterioration is assessed with reference to loss in mass and change in appearance. Although concretes are classified according to key changes in appearance, the assessment is subjective and does not lend itself to standardisation.

2.3 Review of test procedures in which specimens are partially immersed in sulfate solution

2.3.1 Mortar tests

A method was developed in France [40] in which 20 mm x 20 mm x 100 mm mortar prisms are half immersed in a $MgSO_4$ solution containing 19,5 g/l SO_4^{2-}. The mortars have a sand : cement ratio of 3 : 1 and are gauged to constant flow with a mean w/c of ~ 0,50. They are cured for 28 days at 20 °C in water before exposure to the sulfate solution. The solution level is maintained constant and is replaced at monthly intervals. After 2, 5 and 10 months of exposure the compressive strengths of the exposed and immersed ends are determined and compared with the strengths of reference specimens half immersed in water.

The testing of a wide range of production (i.e. factory-made) cements [41] showed that whilst cements with a high blastfurnace slag content performed well in immersed conditions the exposed ends suffered a marked strength loss.

2.3.2 Paste tests

Cement paste cubes, sealed on four faces have been partially immersed in pH controlled Na_2SO_4 solution in order to monitor sulfate penetration and changes in microstructure [42].

2.3.3 Concrete tests

Published data for partial immersion relates mainly to specimens placed in the field under controlled conditions. A 16 year investigation was undertaken in Sacramento, California, in which 152 mm x 152 mm x 762 mm concrete beams were placed horizontally to a depth of 75 mm in a sodium sulfate rich soil [14].

The SO_4^{2-} concentration was 65 000 ppm by mass of the soil. The specimens underwent approximately 10 wetting and drying cycles a year. As in the French laboratory studies, (section 2.3.1) concretes containing slag (and also silica fume) performed less well than equivalent Portland or fly ash containing concretes. The major mechanism of deterioration appeared to be physical rather than chemical and was attributed to cyclic crystallisation of sodium sulfate salts.

Similar conclusions concerning the mechanism of degradation were made following similar field exposure tests in Argentina [43].

A novel approach for testing for sulfate resistance utilises hydrostatic pressure to accelerate the penetration of a concentrated sulfate solution [44]. Concrete cylinders (150 mm x 300 mm) were cast with a tapered inset, which resulted in a central void of 35 mm in diameter narrowing to 30 mm at its end point, which was 60 mm above the base of the cylinder. The central void was almost filled with distilled water and the cylinder immersed to the same level in a concentrated sodium sulfate solution (54 g/l SO_4^{2-}). Deterioration was monitored by means of weight loss and CEM I concrete (250 kg/m^3, w/c ~ 0,65) showed significant deterioration within a few weeks. Partial replacement of cement by fly ash markedly improved sulfate resistance.

2.4 Review of test procedures in which specimens are subjected to alternate wetting and drying

2.4.1 Mortar test

The literature search did not identify any mortar test procedures which involved cycles of wetting and drying.

2.4.2 Paste tests

Cement paste cubes (25 mm, w/c 0,30) were cured for 28 days and then subjected to immersion in $MgSO_4$ solution (80 g/l SO_4^{2-}) and drying at 60 °C at intervals of 10 days [45]. Compressive strengths were determined at ages of 90 days, 120 days and 180 days. In contrast to most studies in mortar or concrete it was found that the partial replacement of cement by silica fume did not improve performance in the tests.

2.4.3 Concrete tests

Wetting and drying is incorporated in method C of the USBR procedure (section 2.2.3).

The Swiss standard for concrete SN 505 262/1 has included an accelerated sulfate resistance test since 2003. Concrete cores are dried for two days at 50 °C and then immersed in Na_2SO_4 solution containing 34 g/l SO_4^{2-} for five days at 20 °C. The change in length after four cycles is compared with that of parallel cores subjected to the same drying cycles but immersed in water.

Ambient temperature air drying (eight hours in air, 16 h in sulfate solution) [46] has been applied with the objective of accelerating the test.

Concrete cubes have been also been subjected to wetting and drying cycles using procedures developed for natural stone [47]. The 50 mm cubes were subjected to 120 cycles of drying at 105 °C and immersion in a Na_2SO_4 solution containing 42 g/l SO_4^{2-}. The change in mass and compressive strength was determined. The performance of the concrete was determined by physical rather than chemical characteristics. The best performance was given by low w/c superplasticised concretes.

3 Review of most appropriate methods to assess specimen deterioration in laboratory tests

As outlined in section 2.1 the deterioration of specimens subjected to sulfate attack in the laboratory is normally assessed using one of the following performance indicators:

- change in length (linear expansion);
- change in compressive strength;
- change in flexural strength;
- change in mass;
- change in appearance;
- change in elastic dynamic modulus.

In several investigations, more than one indicator has been used thus enabling a comparison to be made of their sensitivity and efficacy. Table 3 compares the advantages and disadvantages of the various performance indicators.

Table 3 - Comparison of performance indicators for sulfate resistance

Performance indicator	Advantages	Disadvantages
Linear expansion	The same set of specimens typically six in total, (three in test solution and three in water) can be measured many times. Test equipment is simple and low cost.	May not adequately detect specimen deterioration due to softening of the matrix rather than expansion (see text in this section) Measurements are subject to operator error. Requires frequent calibration of the apparatus for measurement of length changes (see ASTMC490-00a).
Compressive strength	Measures a fundamental property of specimen and damage caused by expansion as well as softening of matrix. Most cement and concrete laboratories well skilled in compressive strength testing.	Destructive technique requiring a large number of specimens if tests are to be performed at a number of ages. Specimens will normally show an increase in compressive strength during the early stages of sulfate attack attributable to the formation of reaction products.
Flexural strength	Measures a fundamental property of specimen and damage caused by expansion as well as softening of matrix. Potentially more sensitive to specimen deterioration than compressive strength.	Destructive technique requiring a large number of specimens if tests are to be performed at a number of ages. Specimens will normally show an increase in flexural strength during the early stages of sulfate attack attributable to the formation of reaction products. Flexural strength testing normally shows a higher coefficient of variation than compressive strength testing.
Change in mass	The same set of specimens can be measured many times. Test equipment is simple and low cost. Detects deterioration which may not be expansive in nature.	Loss of mass is strongly influenced by operator handing and, in particular, the degree of brushing applied to remove loose material.
Change in appearance	The same set of specimens can be assessed many times. Test equipment is simple and low cost (photographic evidence highly desirable).	Assessment is subjective and not appropriate for standardisation. Influenced by operator handling.
Dynamic modulus	The same set of specimens can be assessed many times. Measures a fundamental mechanical property of specimen relative to structural performance.	Published information indicates technique is not very sensitive to the early detection of sulfate induced deterioration. Equipment is relatively complex and technique is more difficult to standardise than other available techniques.

Linear expansion is the most widely used performance indicator in published papers and reports. However, its major weakness is that it measures deterioration associated with the formation of ettringite from monosulfate. Investigations in which both expansion and strength have been determined have indicated that slag and fly ash containing cements can undergo deterioration while showing relatively low levels of expansion. For example examination of data reveals a compressive strength loss of 50 % for fly ash and slag blends (EN 196-1 prisms in $MgSO_4$ solution with 24 g/l SO_4^{2-} at 10 ^{0}C) with expansions of less than 0,05 %

[28]. Similar results were obtained with ASTM C1012 prisms and ASTM C109 mortar cubes, in Na_2SO_4 solution (33,8 g/l SO_4^{2-}) [48]. A 50 % slag blend performed better than low C_3A Portland cement when assessed by expansion but at a comparable level when assessed by compressive strength. Thus, whilst expansion may be an appropriate test procedure for CEM I cement or cements with a relatively high clinker content, it may be less reliable when applied to cement which when hydrated contain low levels of monosulfate.

The determination of dynamic modulus appears to be less sensitive to specimen deterioration than either change in length, compressive strength or visual appearance [49, 50].

4 Review of most appropriate methods to accelerate the test procedure

As discussed in section 2.1, if good quality concretes are placed in sulfate solutions with SO_4^{2-} concentrations similar to those likely to be encountered in the field (< 6 g/l), several years of exposure are required before meaningful discrimination can be made between sulfate resisting and non-sulfate resisting cements.

In order to accelerate the identification of cements that do not have an adequate level of resistance to sulfates the following conditions has been used to accelerate the test:

- highly concentrated sulfate solution;
- use of $MgSO_4$ rather than Na_2SO_4 or a combination of both sulfates;
- control of solution pH;
- high specimen w/c;
- small specimen size;
- elevated temperature;
- low temperature;
- short pre-curing period.

Table 4 compares the advantages and disadvantages of these various test conditions.

Table 4 - Comparison of test conditions for sulfate resistance

Test condition	Advantages	Disadvantages
Highly concentrated sulfate solution	Effective in accelerating deterioration.	Mechanism of deterioration may be different from that in the field. For example, high sulfate concentrations result in extensive formation of gypsum in the hydrated matrix. Gypsum formation is not normally found in the field. There is a risk, particularly with test aggregates which are not well characterised, of expansion occurring as a result of alkali silica reaction induced by a highly alkaline Na_2SO_4 solution.
$MgSO_4$ or mixed solution with Na_2SO_4	Sulfate solutions encountered in the field normally have several cations present, often including Mg^{2+}. $MgSO_4$ is more aggressive than Na_2SO_4, particularly towards cements containing slag and fly ash.	Layer of brucite may provide variable protection to specimens. Less easy to make comparison with the bulk of published test data which relates to Na_2SO_4 solution.
Control of solution pH	Enables sulfate attack to be accelerated without the use of unrealistically high sulfate concentrations. Should enable a single tank to be used for multiple samples. Near neutral conditions relate better to most field conditions. Continuous immersion of specimens reduces likelihood of surface carbonation.	Equipment cost, as best carried out with automated titration equipment. SO_4^{2-} concentration may need regular adjustment.
High specimen w/c	Accelerates deterioration without the use of unrealistically high sulfate concentrations. Easy to implement by adjustment of aggregate to cement ratio.	May be criticised for departing from range of w/c recommended in concrete standards (such as EN 206-1) for sulfate resisting concrete.
Small specimen size	Accelerates deterioration without the use of unrealistically high sulfate concentrations. Easy to implement. Reduces space requirements for specimen storage.	Very small specimens (e.g. 12,5 mm cubes) are subject to higher variability than larger specimens.
Elevated temperature	Accelerates rate of deterioration.	Different binder types differ in response to elevated test conditions. Conditions not relevant to most field conditions.
Low temperature	More severe test for most binders as continuing hydration is inhibited. Permits the potential for thaumasite form of sulfate attack (TSA) to be assessed. Relates better to European field conditions than the standard laboratory conditions of 20 °C.	Different binder types differ in response to low temperature sulfate exposure conditions.
Short pre-curing period	Accelerates rate of deterioration Minimises risk of protective carbonated surface forming prior to exposure to sulfate solution	Exaggerates the risk of significant deterioration of structures in the field particularly with cements containing slowly reacting constituents such as fly ash or pozzolanas

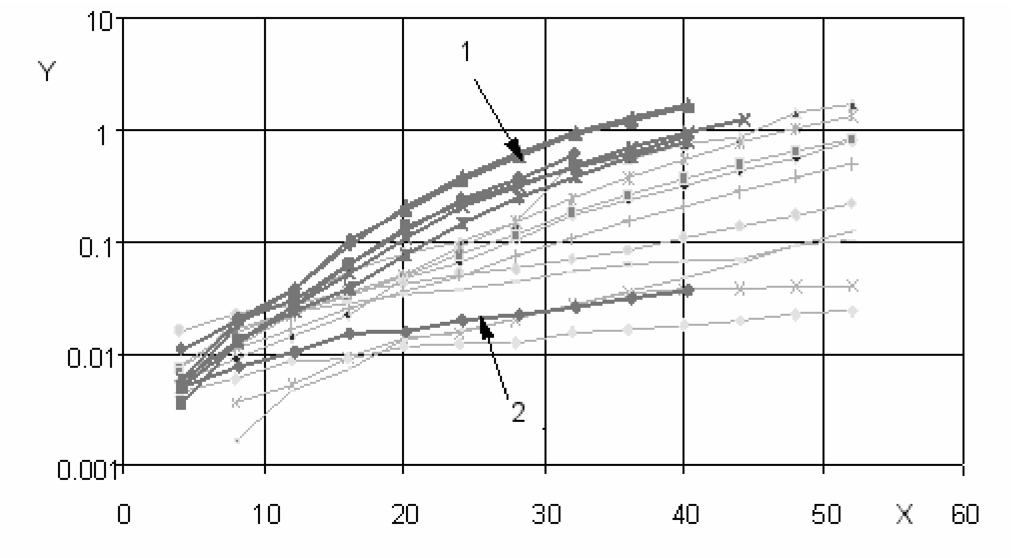
Although highly concentrated sulfate solutions have predominated in the test procedures applied in the past there is increasing recognition [51, 16] that the results do not relate to field experience and that the mechanism of deterioration should not deviate from that occurring in practice.

5 The importance of test method reproducibility

With the exception of the papers that reported on the collaborative work leading to the development of ASTM C 1012 [52] and the work within CEN/TC51/WG12/TG1 to develop an EN test procedure [53], the publications reviewed have not provided information concerning test method reproducibility. This is because all of the investigations have involved single laboratories.

It is essential that a standardised test method has a satisfactory level of reproducibility.

Figure 1 illustrates the range of expansion values obtained when ten cement testing laboratories applied the methods used during the round robin activities of WG 12/TG1 to a moderate C_3A cement.



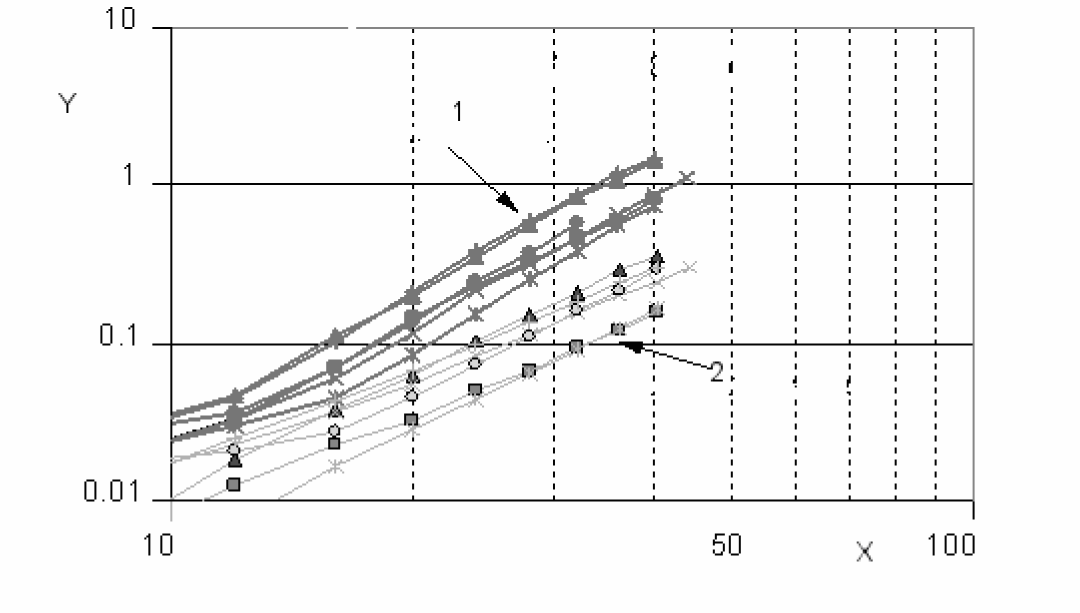
Key

X Time in solution (weeks)
y Expansions (%)
1 4th in bold
2 Laboratory No.1

Figure 1 – CE/TC51/WG12/TG6 4th Round Robin compared to 2nd cement No. 1

The modifications to the method introduced in the fourth round robin improved reproducibility but one laboratory remained an outlier. An investigation revealed that the low expansions determined in laboratory 1 were probably associated with specimen storage and measurement rather than specimen preparation.

Figure 2 illustrates the discrimination achieved between a moderate C_3A CEM I cement and a low C_3A CEM I SR in the fourth round robin.



Key

X Time (weeks)
Y Expansion %
1 Cement 1 (C3A 8,5 %)
2 Cement 2 (C3A 1,9 %)

Figure 2 – CEN/TC51/WG12/TG1 Fourth testing programme Mix A, w/c 0,50, 60 jolts

While within the laboratory repeatability was satisfactory at ~ 10 %, the between laboratory reproducibility was poor and in the range 20 % to 50 %.

If a test procedure is to be used for the acceptance or rejection of a cement or related binder, it is suggested that the coefficient of reproducibility should be 15 % or better. Note that the coefficient of reproducibility of the EN 196-1 compressive strength test is 6 % at 28 days.

6 Suggested features of a standardised sulfate resistance test method for cements

From examining the literature required for preparing this report and the experience gained during the CEN/TC51/WG12/TG1 test programme, it is suggested that a standardised test method for the sulfate resistance testing of cement might have the following features listed in Table 5.

Table 5 – Main features of a possible EN test method for sulfate resistance

Feature of test	Justification
40 mm x 40 mm x 160 mm prisms prepared using EN 196-1 Standard sand	High level of competence in preparing and testing EN 196-1 prisms. Widespread availability of equipment. Availability of standard test sand.
Prisms well cured before exposure to sulfate solution. Minimum curing period of four weeks at 20 °C in limewater suggested. A longer curing period (e.g. three months) may be justified if surface damage to field concrete is considered relatively unimportant. Moderate temperature accelerated curing (e.g. 28 days at 35 °C) is a possibility.	Inadequate curing of cements containing constituents such as fly ash or pozzolanas will yield pessimistic results, which only relate to the performance of the outer skin of concrete Realistic assessment of long term performance of structure requires adequate curing Limewater rather than water reduces leaching $Ca(OH)_2$
Prisms fully immersed in test solution.	While the relevance of partial immersion to many field situations is recognised, results obtained are likely to be ambiguous. Test is designed to test resistance of binder to sulfate attack alone; not sulfate attack and resistance to crystallisation pressures.
Na_2SO_4 solution test solution prepared from deionised water.	Use of Na_2SO_4 enables comparisons to be made with the majority of prior published work Single cation simplifies interpretation of results Deionised water eliminates presence of other ions, which might affect reproducibility
Maximum SO_4^{2-} concentration of 10 g/l and performance at 3 g/l to be investigated.	Evidence that mechanism of failure in concentrated solutions differs from that in the field Poor correlation between accelerated tests with high concentration sulfate solutions and field experience
pH and SO_4^{2-} to be controlled by automatic titration [a pH of (8 ± 0,5) is suggested] and solution to be agitated/recycled.	Relates better to the majority of field situations than the pH environment of ~12 created in tests where pH is allowed to rise before solution replaced Has the potential to allow a single tank to be used for a large number of specimens and for specimens to be added at different times over a lengthy timescale Evidence that controlling pH in range 7 to 8 accelerates attack without need for high SO_4^{2-} concentrations
Temperature of test solution selected according to the relevant local conditions.	The normal laboratory temperature of 20 °C does not relate to European field conditions which show a considerable range between Mediterranean and Nordic countries In many situations a solution temperature of 10 °C may be most appropriate but performance at temperatures as low as 5 °C should be evaluated The lower temperatures also provides the potential for the thaumasite form of sulfate attack (TSA) to be assessed, if appropriate source of carbonate made available

Table 5 *(concluded)*

Mortar w/c 0,60 with sand : cement ratio adjusted to 3,375.	Higher w/c ratio than EN 196-1 to achieve discrimination in a reasonable timescale. Fixed w/c in order to improve reproducibility (compared to mortars gauged to constant flow). Adjustment of sand : cement ratio based on experience in CEN/TC51/WG12/TG1 test programme, although this was limited to CEM I cements.
Procedures to be introduced to minimise the possibility of specimen carbonation at any stage of testing. Consider specimen immersion in dilute sulfuric acid (H_2SO_4) for a number of hours prior to immersion in test solution in order to remove any protective carbonated surface formed during specimen curing.	A lot of evidence to show that early age carbonation of specimen surface markedly inhibits sulfate attack. Belief that variable level of carbonation makes a marked contribution to the poor level of reproducibility of sulfate resistance tests.
Pass/fail criteria to be based on the relative strength of specimens stored in the sulfate solution and in limewater. If 40 mm x 40 mm x160 mm prisms are selected then it will be possible to determine both flexural and compressive strengths on the same specimens.	Evidence that some binder types can show significant deterioration with minimal expansion. Fundamental property of binder. More appropriate than expansion for unknown types of binder which might be tested in the future. Flexural strength may give an earlier indication of deterioration than compressive strength. Compressive strength testing offers potential for higher level of precision.
Specimens to be broken at 1 year and 2 years.	Timescale based on examination of literature and expectation that sulfate resistant binders will maintain 80% of strength at age of 2 years.

The suggestions above for a standardised test method assume that the test will be applied for initial type testing of a new/unproven cement or mixer-combination. Experience has shown that highly accelerated tests which could possibly be applied on a regular basis to assess product conformity yield unreliable results.

An experimental programme will be required to enable the selection of the most appropriate test parameters for:

- mortar composition (w/c ratio, sand/cement ratio);
- pre-curing period;
- temperature of exposure;
- concentration of solution;
- critical test ages for application of pass/fail criteria.

In addition, the effectiveness of specimen pre-treatment, to remove any surface carbonation, in improving test method sensitivity and reproducibility needs to be evaluated experimentally.

It is believed that strength testing is more generally applicable to novel types of binder which may be assessed in the future and which may not react expansively with sulfate solutions. Test laboratories have considerable experience with the EN 196-1 strength testing procedure and it is believed that strength testing offers the potential of an acceptable level of reproducibility.

Note that the results from a test method such as that outlined above cannot be applied directly to the performance of concrete in the field. The test is designed to assess the sulfate resistance characteristics of a cement or related binder when the penetration of the sulfate solution is facilitated by a w/c higher than would be permitted by concrete specifications.

Bibliography

[1] R. S. Gollop & H. F. W. Taylor, Microstructural and microanalytical studies of sulfate attack IV. Reactions of a slag cement paste with sodium and magnesium sulfate solutions, Cement and Concrete research, Vol 26, pp 1013-1028, 1996.

[2] R.S. Gollop and H.F.W. Taylor, Microstructural and microanalytical studies of sulfate attack V. Comparison of different slag blends, Cement and Concrete Research, Vol 26, pp.1029-1044, 1996.

[3] Odler, A discussion of the paper 'Mechanism of sulfate attack on Portland cement and concrete – another look' by P. K. Mehta, Cement and Concrete Research, Vol 14, pp 147-148, 1984.

[4] P. K. Mehta, Sulfate resistance of blended cements, Proceedings of Concrete 88 Workshop on the use of fly ash, slag, silica fume and other siliceous materials in concrete, Ed W.G. Ryan, pp 337-351, Sydney 1988.

[5] Soroushian and A. Alhozaimy, Correlation between fly ash effects on permeability and sulfate resistance of concrete, 9th International Congress Chemistry of Cement, New Delhi, Vol V, pp.196-202, 1992.

[6] J. B. Stephens and R. L. Carrasquillo, Evaluating performance-based test and specifications for sulfate resistance in concrete, Texas Dept of Transportation, Report No 0-1706-3, 182 pages, 2000.

[7] D.C. Hughes, Sulfate resistance of OPC/fly ash and SRPC pastes: pore structure and permeability, Cement and Concrete Research, Vol.15, pp.1003-1012, 1985.

[8] J. L. Jang, Role of permeability in sulfate attack, Cement and Concrete Research, Vol 27, pp1179-1189, 1997.

[9] P. W. Brown and H. F. W. Taylor, The role of ettringite in external sulfate attack, Materials Science of Concrete, Special Volume, Sulfate attack mechanisms, Pub American Ceramic Society, pp 73-97, 1999.

[10] G. Frigione and F. Zenone, Influence of gypsum content in Portland cements on sulfate resistance, 9th International Congress on Chemistry of Cement, New Delhi, Vol IV, pp 60-66, 1992.

[11] J. J. Kollek and J. S. Lumley, Comparative sulfate resistance of SRPC and Portland slag cements, Durability of Building Materials and Components, Proceedings of 5th International Conference, Brighton UK, Pub E.& F. N. Spon, Paper 43, 1990.

[12] J. R. Prusinski and R. L Carrasquillo, Using medium-to high volume fly ash blended cements to improve the sulfate resistance of high-lime fly ash concrete, Natural Pozzolans in Concrete, Proceedings Fifth International Conference, Milwaukee, ACI SP-153, pp 43 – 65, 1995.

[13] R. D. Hooton and J. J. Emery, Sulfate resistance of a Canadian slag cement, ACI Materials Journal, pp 547-555, 1990.

[14] D. Stark, Performance of concrete in sulfate environments, Portland Cement Association, Report RD 129, 28 pages, 2002.

[15] K Mather, Tests and evaluation of Portland blended cements for sulfate resistance, ASTM STP 663, pp 74-86, 1978.

[16] J. R. Clifton, G. Frohnsdorff and C. Ferraris, Standards for evaluating the susceptibility of cement-based materials to external sulfate stack, Materials Science of Concrete, Special Volume, Sulfate attack mechanisms, Pub American Ceramic Society, pp 337-355, 1999.

[17] ASTM E 632, Standard practice for developing accelerated tests to aid the prediction of the service life of building components and materials.

[18] R. Sersale et al, Sulfate attack of carbonated and uncarbonated Portland and blended cement mortars, 10th International Congress on Chemistry of Cement, Goteborg, Vol 4, 1997.

[19] J. P. H. Frearson and D.D. Higgins, Effect of test procedures on the assessment of the sulfate resistance of slag cements, 5th International Conference on fly ash, silica fume, slag and natural pozzolans in concrete, ACI SP 153-51, pp 975-993, 1995.

[20] D. D. Higgins and N. J. Crammond, Resistance of concrete containing GGBS to the thaumasite form of sulfate attack, Cement and Concrete Composites, 25, pp 921-929, 2003.

[21] A. Borsoi et al, Sulfate attack on blended Portland cements, 5th international conference on durability of concrete, Barcelona, ACI SP – 192, Vol 1, pp 417-432, 2000.

[22] M. Santhanam et al, Differentiating seawater and groundwater sulfate attack in Portland cement mortars, 6th CANMET-ACI International Conference on Durability of Concrete, Thessaloniki, ACI SP 212, pp 21-35, 2003.

[23] A. Fraay et al, Sulfate resistance of mortars with pulverised fuel ash, Concrete Durability, ACI SP 100-104, pp2041-2058, 1987.

[24] K. Torii and M. Kawamura, Effect of fly ash and silica fume on the resistance of mortar to sulfuric acid and sulfate attack, Cement and Concrete Research, Vol 24, pp361-370, 1994.

[25] F.W. Locher, The sulfate resistance of cement and its testing, Zement Kalk Gyps, 51, pp 388-398, 1988.

[26] F. Turker et al, Effect of magnesium sulfate concentration on the sulfate resistance of mortars with and without silica fume, Cement and Concrete Research, Vol 27, pp 205-214, 1997.

[27] H. Binic and O. Aksogan, Sulfate resistance of plain and blended cement, Cement and Concrete Composites, 28, pp 39-46, 2006.

[28] C. D. Lawrence, Comparative sulfate resistance of mortars made with different binder types, British Cement Association, report C/14, 71 pages, 1993.

[29] P. W. Brown, An evaluation of sulfate resistance of cement in a controlled environment, Cement and Concrete Research, Vol 11, pp 719-727, 1981.

[30] H. T. Cao et al, The effect of cement composition and pH of environment on sulfate resistance of Portland cements and blended cements, Cement and Concrete Composites, 19, pp 161-171, 1997.

[31] C. F. Ferraris, Mechanism of degradation of Portland cement-based system by sulfate attack, Mechanisms of chemical degradation of cement-based systems, Ed K. L. Scrivener and J. F. Young, E &. F N Spon, paper 22, 1997.

[32] Rasheeduzzafar et al, Influence of cement composition on the corrosion of reinforcement and sulfate resistance of concrete, ACI Materials Journal, V 87, pp 114-122, 1990.

[33] D. Bonen and M. D. Cohen, magnesium sulfate attack on Portland cement paste, 1. Microstructural analysis, Cement and Concrete Research, Vol 22, pp 169-180, 1992.

[34] M Collepardi et al, Resistance to sulfate attack of mixtures with limestone-Portland blended cements, International Conference on Durability of Concrete, Thessaloniki, ACI SP 212, pp 941-949, 2003.

[35] J. G. Cabrera and C. Plowman, The mechanism and rate of attack of sodium sulfate solution on cement and cement/pfa pastes, Advances in Cement Research, No 3, pp 171-179, 1988.

[36] P. K. Mehta and O. E. Gjorv, A new method for testing sulfate resistance of cements, Journal of Testing and Evaluation, Vol 2, pp 510-515, 1974.

[37] P. K. Mehta, Effect of fly ash composition on sulfate resistance of cement, ACI Journal, pp 994-1000, Nov-Dec 1986.

[38] F. Massazza and M. Testolin, Chemical resistance of pozzolanic cements and silicate structures of hardened pastes, Il Cemento, 4, pp 519 – 528, 1988.

[39] G. J. Osborne, Determination of the sulfate resistance of blastfurnace slag cements using small-scale accelerated methods of test, Advances in Cement Research, 2, pp 21-27, 1989.

[40] J. Eustache and R. Magnan, Method for determining resistance of mortars to sulfate attack, Journal of American Ceramic Society, Vol 55, pp 237-239, 1971.

[41] R. Guyot et al, Comparison of the resistance to sulfate solutions and to sea water of different Portland cements with or without secondary constituents, fly ash, silica fume, slag and other mineral by-products in concrete, ACI SP-79,Vol. I, pp 453-469, 1983.

[42] J. G. Wang, Sulfate attack on hardened cement paste, Cement and Concrete Research, Vol 24, pp 735-742, 1994.

[43] E. F. Irassar et al, Sulfate attack on concrete with mineral admixtures, Cement and Concrete Research, Vol 26, pp 113-123, 1996.

[44] S. Alegre et al, New prediction methods of durability application to concrete with and without fly ash, Concrete Durability, ACI SP 100 - 39, pp 681-704, 1987.

[45] E. E. Hekal et al, Magnesium sulfate attack on hardened blended cement pastes under different circumstances, Cement and Concrete Research, Vol 32, pp 1421-1427, 2002.

[46] C. Hartmann and E. Mangotich, A method for predicting sulfate durability of concrete, Concrete Durability, ACI SP 100-110, pp2135-2151, 1987.

[47] I. R. de Almeida, Resistance of high strength concrete to sulfate attack: soaking and drying test, Durability of concrete, 2nd International Conference, Montreal, ACI SP-126, Vol II, pp 1073-1092, 1991.

[48] H. Y. Moon et al, Experimental study on the sulfate resistance of concrete blended ground granulated blast-furnace slag for recycling, Geosystem Engineering, 5 (3), pp 67-73, 2002.

[49] N. P. Mailvaganam et al, Sulfate resistance of mortars containing superplasticiser with fly ash or blastfurnace slag, Developments in the use of superplasticisers, ACI SP-68, pp 535-556, 1981.

[50] K. Torii et al, Sulfate resistance of high fly ash content concrete, Cement and Concrete Research, Vol 25, pp 759-769, 1995.

[51] VDZ activity report 2003-2005, III Performance of cement, 2005.

[52] T. Patzias, Evaluation of sulfate resistance of hydraulic cement mortars by the ASTM C1012 test method, Concrete Durability, ACI SP 100, pp2103-2120, 1987.

[53] G. K. Moir, Development of a sulfate resistance performance test for cements, Proceedings of Duranet Conference, Berlin, June 1999.

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