Effect of complex admixtures on cement properties and the development of a test procedure for the evaluation of high-strength cements

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Recent research has shown that a state-of-the-art process for high-performance (HP) cement adds a new dimension to 'classical' cement technology. The key idea of the process involves the mechano-chemical activation of cement at the grinding stage. A silica-based complex admixture is used for the activation of the cement imparting high strength and improved durability to mortars and concrete. This paper describes the effect of different complex admixtures on the strength of HP cement. According to the available results, mortars based on HP cement possess a compressive strength of up to 148 MPa. A comparison of alternative evaluation methods helps to recommend the test procedure for HP and high-strength cement.

Notation

The following notation were applied to distinguish the samples

NPC-#	reference Portland cement
LWDB-#	reference low water demand binder
HPC-A-#	reference high performance cement type A
HPC-BS-#	reference high performance cement type B (50% of GBFS)
HPC-BL-#	reference high performance cement type B (35% of LS)
SFC-#	high performance cement produced using SF admixture
MSC-#	high performance cement produced using MS admixture
MKC-#	high performance cement produced using MK admixture

The additional letter after the main notation identifies of the test procedure as follows

I	reference Portland cement CEM-I 42·5 (without additional grinding)
W	for EN 196 mortars (constant w/c)
F	for ASTM C109 mortars (constant flow)
S	for ASTM C387 mortars (variable S/C)

Introduction

The latest developments in cement and concrete science show the following trends[1,2]

(*a*) Increasing strength: major breakthroughs in knowledge have led to the design of cement based materials with greater strength; this has resulted in the development and application of super high-strength concretes with a compressive strength of up to 135–250 MPa, high flexural strength and improved ductility.[3–12]

(*b*) Application of chemical admixtures: added to the concrete mixture, relatively small amounts of

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chemical admixtures can alter the behaviour of fresh or hardened concrete. Chemical admixtures can improve almost any problematic property of concrete: they are one of the essential components of modern concrete that help control strength and durability.[13,14]

(*c*) Utilization of industrial by-products and waste (IBPW): the use of IBPW as mineral additives has become an important part in cement and concrete technology. According to wide- ranging investigations,[1,2,13–16] the performance of conventional concrete can be significantly improved by adding IBPW in specific quantities. Well-known mineral additives include granulated blast furnace slag, fly ash and silica fume. These mineral additives not only yield a concrete that has better properties and is more cost effective, but they also reduce the environmental impact of construction activities.[16]

Following these developments, the concept of high performance concrete (HPC) has been put forward and successfully applied worldwide. It is generally accepted that concrete with improved properties (workability, strength, permeability, durability, etc.) above the conventional benchmarks can be specified as HPC. According to Forster,[17] HPC is 'a concrete made with appropriate materials combined according to a selected mix design and properly mixed, transported, placed, consolidated, and cured so that the resulting concrete will give excellent performance in the structure in which it will be exposed, and with the loads to which it will be subjected for its design life'.

To realize the HPC concept, a range of chemical admixtures and mineral additives are required, and consequently, a modern concrete batching plant must use appropriate equipment for precise control, dispatching, dosing and batch processing.[7–14] This modernisation of production facilities is an unavoidable cost for improved performance since variations in dosing or mixing the components of the concrete may affect the performance of the final product. The compatibility of the admixtures is yet another problem for the production and application of HPC.[13,14]

However, an alternative method for controlling concrete properties and the design of HPC is the use of special cements possessing higher strength and better resistance to the deterioration caused by various physical, chemical and mechanical factors.[18–26] These superior cements are often based on special clinkers or blended cements such as shrinkage compensating or expansive cements, high early strength cements, regulated-set and jet-cements.[3] The group of these cements also includes newly developed Ultimax cement, blended Pyrament and SF cements.[20–24]

The application of chemical admixtures offers an advanced approach to improving cement performance; modifing the cement grinding process.[19] Using this method, a number of chemical admixtures and cement products have been developed. Well-known examples include air-entraining, hydrophobic and plasticised cements, and also the family of cements that are manufactured using grinding aids. It has been suggested that the action of these modifiers is based on mechano-chemical activation.[19]

The theory of mechano-chemical activation has been successfully applied to processing nanopowders, pigments, fillers, binders, ceramic and ferromagnetic materials. Using this method, an improvement of cement strength can also be achieved.[19,20,23,26] Low water demand binder (LWDB) is produced by intergrinding cement and a dry modifier at a high energy.[25] In spite of a similar technique already suggested by Royak and Royak, and Skvara *et al.*,[19,20] only the application of a specially selected admixture-modifier at a relatively high dosage (up to 4%) resulted in the manufacture of a cement with both reduced water demand and high strength.

The group of mineral and organic complex admixtures for application in cement technology has been proposed.[18] Basically, these admixtures contain a reactive silica-based sorbent as a mineral constituent and a surfactant as its organic constituent plus some minor corrective admixtures (Fig. 1, further details are provided in Reference[18]). Supersilica, a reactive silica-based complex admixture was developed using this principle.[16,18] Although all the effects of supersilica have not been completely investigated, it is hypothesised that, when added during the cement grinding

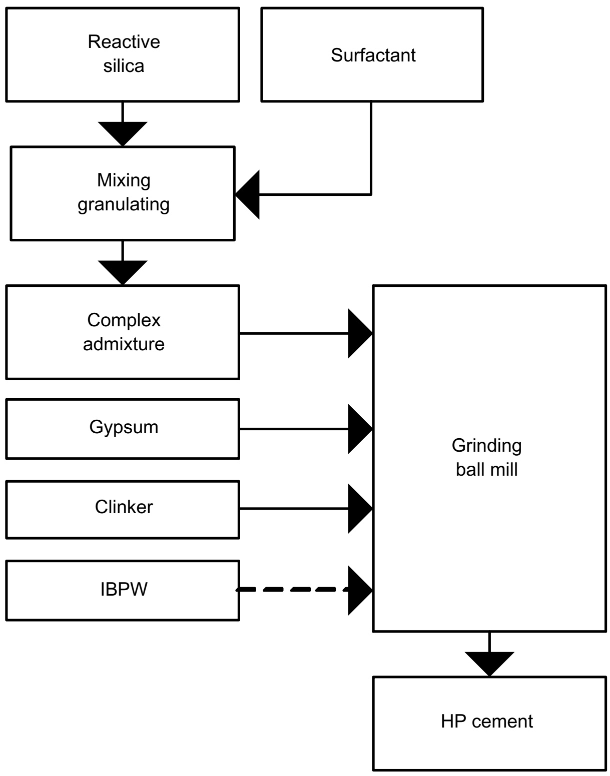

Fig. 1. Flow chart of HP cement technology

process, supersilica modifies the surface of cement particles and also promotes the formation of highly reactive amorphous structures and pre-hydrates.[18] Further, the reactive silica component also acts as a micro-filler and participates in a pozzolanic reaction. The mechano-chemical activation of cement with supersilica at the grinding stage results in a high performance (HP) cement.

HP cement can be defined as a product manufactured by the mechano-chemical activation of certain proportions of clinker, gypsum, complex admixture and, optionally, a mineral additive of industrial (IBPW) or natural origin (Fig. 1), which imparts high strength and extreme durability to the concrete or mortar made from such cement.[18,26] Such high strength can be used for the engineering of a cement with high volume mineral additives (HVMA). As a result, relatively large amounts (up to 70%) of portland cement clinker can be replaced with inexpensive locally available mineral additives. Natural pozzolanic materials, sand, limestone, granulated blast furnace slag, fly ash, glass cullet and ceramic waste, all can be used as mineral additives in HVMA cements.[16]

There are two types of HP cement: type A basic HP cement; and type B blended HP cement. Basic type A cement involves intergrinding the clinker, gypsum with a complex admixture. Blended type B cement covers a wide range of HP cements with mineral additives. The content of a mineral additive in blended HP cement varies with the specified level of the properties and with the type of additive used. Preliminary research[16] demonstrated that type B blended HP cements with a mineral additive content within the standard limitations (25–50% as specified in EN 197-1,[27]) can be produced. Furthermore, the range of mineral additives can be extended for HVMA cement. New types of mineral additives (quartz sand, glass cullet and ceramic waste) can be used as components of the blended HP or HVMA cements.[16,18]

The application of granulated blast furnace slag (GBFS) in blended HP cement in addition to high ultimate strength (more than 80 MPa), results in a product possessing high resistance to chemical attack and to elevated temperatures (up to 700°C).[26]

Research significance

The effect of silica fume on the behaviour of cement-based materials has been extensively reported in the literature.[1–14] A number of published papers on HP cement deals with the application of a silica fume based complex admixture.[16,26] At the same time the use of other types of reactive silica products as a component of the complex admixture has been proposed.[18] Consequently, the evaluation of the effect of different reactive silica materials on the cement strength is important for the manufacture and practical application of HP cement based materials.

Experimental programme

Materials used

Three different reactive silica components were used in the complex admixture: silica fume (SF), natural microsilica (MS) and metakaolin (MK). The reference cements were: Portland cement CEM-I 42·5[27] (NPC-I, type I according to ASTM C150[28]) and low water demand binder (LWDB). The samples of optimised high-performance cement type A (HPC-A) and type B (HPC-BS containing 50% of GBFS and HPC-BL containing 35% of limestone, LS) were also tested for comparison.[26] The chemical composition of these materials is presented in Table 1. It is noticed that due to a high volume of GBFS, HPC-BS had a relatively high MgO content, but no subsequent expansion of cement was observed during the hardening. The particle size distribution of some selected cement samples is provided in Fig. 2. Standard Rilem Chembereau sand[29] was used in the preparation of the mortars.

Research programme

The experimental programme had three main components

(*a*) The investigation of cement compressive strength at constant w/c according to EN 196[30]

Table 1. Chemical analysis of cementitious materials

Composition	SF	MS	MK	NPC-I	BFS	LS	LWDB	HPC-A	HPC-BS	HPC-BL
SiO_2	90·00	84·60	86·80	19·42	37·43	-	19·77	28·47	34·28	20·87
Al_2O_3	0·40	5·14	5·17	5·27	10·91	-	5·15	4·18	7·83	4·74
Fe_2O_3	0·36	0·85	1·00	2·80	0·64	-	4·01	3·36	2·46	1·65
CaO	1·63	0·50	4·10	62·07	35·88	52·05	56·00	55·01	43·43	49·83
MgO	1·02	1·05	1·03	2·04	8·07	3·04	1·91	1·89	5·69	2·73
Na_2O	0·50	-	0·24	0·70	0·35	-	0·25	1·02	1·01	-
K_2O	2·28	-	0·46	1·00	1·17	-	0·42	0·38	0·19	-
SO_3	0·44	0·99	-	2·30	2·06	-	3·82	2·61	2·35	1·89
LOI	3·03	6·15	1·20	3·50	-	42·90	2·7	2·83	1·50	15·89

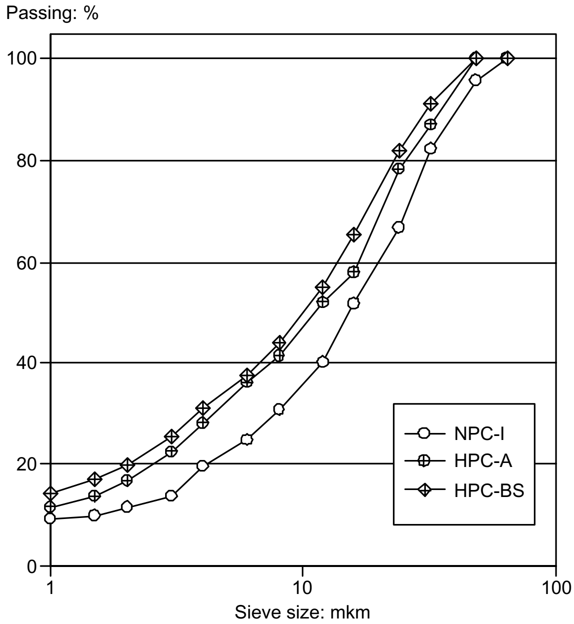

Fig. 2. Particle size distribution of HP cement

(*b*) The investigation of cement compressive strength at constant flow according to ASTM C109[31,32]

(*c*) The investigation of compressive and flexural strength of mortars at different sand to cement ratio (s/c) according to ASTM C387.[31–34]

Mixture proportioning

The strength properties of eight different cements were investigated. These included HP cements produced using different complex admixtures (SFC, MSC and MKC) and reference cements. Ten percent (by weight) of complex admixture was used to produce these cements. Quartz sand (S) was used at a small amount (5%) in the compositions of the investigated cements in order to facilitate the grinding in the ball mill. The composition of the investigated cements is given in Table 2.

Three sets of mortars were prepared: W according to EN 196; F according to ASTM C109; and S according to ASTM C387. For W mortars a constant w/c of 0·5 and a s/c ratio of 3·0 were used. F and S mortars were produced at w/c adjusted to constant flow (as described in ASTM C109). A s/c ratio of 2·75 was applied for F mortars. HPC-BS cement was used for the preparation of S group of mortars, which were produced at the s/c of 1, 3, 5 and 7.

Preparation of specimens

Samples of high performance cement were obtained by grinding mixtures specified in Table 2 in a laboratory ball mill. The sample batch weight was 5 kg and grinding media weight was 65 kg. Grinding time was 30 min. In order to eliminate the effect of the additional grinding on the properties of investigated cements, the reference Portland cement NPC-I was also ground for an equivalent time. The resulting fineness data of the investigated cements are summarized in Table 2.

Mortars based on these obtained cements were prepared following EN 196.[30] For F and S mortars the flow table was applied to obtain the standard flow of 105–115 mm. The mortars for the strength test were cast into three-gang (40 × 40 × 160 mm) prism molds, and compacted in accordance with EN 196.[30]

Curing of specimens

After the compaction procedure, the molds were placed in a humidity cabinet for 24 h (keeping a relative humidity of 95% and a temperature of 20°C). Following this period, the specimens were removed from the molds and kept in water until the testing age.

Tests performed

Compressive strength tests were conducted using the portions of prisms broken in flexure.[28] The compressive strength results indicated are the average of the six values and flexural strength results are the average of the three values. Different age conditions reflecting the requirements of References[25–27] were applied: W-mortars were tested at the age of 2, 7 and 28 days; and F-mortars tests were carried out at the age of 1, 3, 7 and 28 days. The compressive and flexural

Table 2. Composition and fineness of investigated cements

Cement type	Cement composition								Fineness		
	NPC-I	LWDB	LS	BFS	S	Complex admixture			% retaining		Blaine m^2/kg
						SF	MS	MK	45 μm	90 μm	
NPC	100	-	-	-	-	-	-	-	8·2	1·3	516
SFC	85	-	-	-	5	10	-	-	17·3	3·0	477
MSC	85	-	-	-	5	-	10	-	11·8	2·6	505
MKC	85	-	-	-	5	-	-	10	12·6	3·0	422
LWDB	-	100	-	-	-	-	-	-	4·3	0·5	409
HPC-A	85	-	-	-	5	10	-	-	5·4	0·7	570
HPC-BS	40	-	-	50	-	10	-	-	5·2	0·6	580
HPC-BL	50	-	35	-	5	-	-	10	10·5	2·4	416

strength tests of S-mortars were performed at the age of 28 days.

Test results

Compressive strength of mortars with constant w/c

The test results of W- mortars (following EN 196) are presented in Table 3 and Fig. 3. According to the obtained results the best 28-day compressive strength value of 62·1 MPa was obtained from the cement produced with metakaolin (MKC). This value is close to the strength demonstrated by an optimised sample of HPC-A (67·0 MPa). The silica fume (SFC) and natural microsilica (MSC) based cements reached 51·4 and 51·9 MPa, respectively, which is close to the 28 day compressive strength of HPC-BS (52·1 MPa). The reference sample of NPC demonstrated a compressive strength of 43·8 MPa (which meets the requirements for 42·5 grade cement), while the strength of LWDB sample was only 26·6 MPa (outside the limits specified by EN 196 for a cement of the lowest grade of 32·5). The strength of investigated HP cements at the 2-day age is almost the same for all investigated cements and lies in the range of 25·5–28·6 MPa, with the best value for MSC.

At the 7 day point MK-based cement demonstrated an even better strength value than optimised HPC-A, gaining 49·2 against 45·6 MPa. The intermediate level of 38·4 MPa was shown by MSC. In this group SFC was the least satisfactory, developing slightly lower strength compared with NPC: 32·6 and 32·8 MPa, respectively. The lowest 7 day strengths were obtained from HPC-BL, LWDB and HPC-BS with 25·4, 22·6 and 21·9 MPa, respectively.

Compressive strength of mortars with constant flow

The test results of F-mortars (ASTM C109) are presented in Table 4 and Fig. 3. Based on the flow test, a w/c ratio was selected for the mortars. It was found that all cements (except reference NPC) required a w/c in the range of 0·28–0·30 in order to obtain the standard flow of 105–115 mm. Due to its special design, the complex admixture overcomes the relatively high water absorption of reactive silica component; yet, conversely, it also provides a strong water reducing effect, resulting in a cement with water demand comparable to or less than that of LWDB.

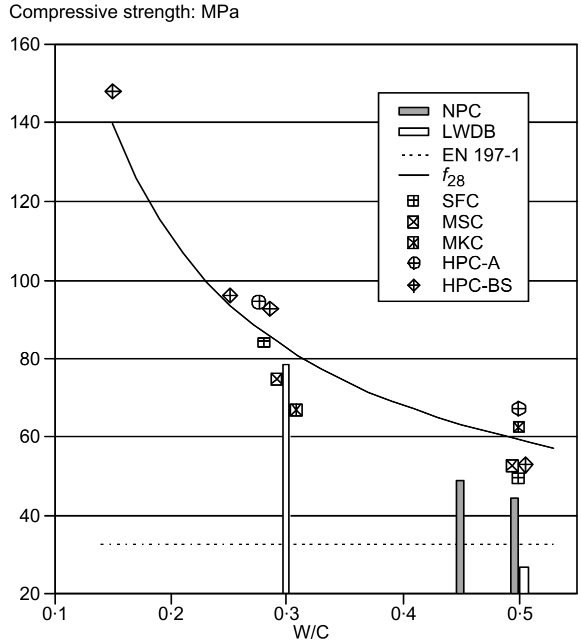

Fig. 3. W/C law for high performance cement

The results demonstrated that cement produced with a SF-based complex admixture (SFC) possessed the highest compressive strength of 84·1 MPa at the age of 28 days. However, this value is lower than the strength of optimized samples of HPC-A and HPC-BS (94·4 and 92·7 MPa, respectively). The microsilica cement (MSC) demonstrated a strength value of 74·5 MPa, slightly lower than LWDB strength of 78·7 MPa. The lowest 28 day strength (66·8 MPa) was obtained for metakaolin cement (MKC), which is comparable to the strength of reference HPC-BL (66·7 MPa). The strength of NPC reference sample was only 48·9 MPa.

The 1 day strength of all investigated HP cements was found to be very high: between 40·6 and 33·0 MPa, with the best value for SFC. Similar strength was

Table 3. Compressive strength of W- mortars (EN 196)

Mix Type	S/C	W/C	Compressive strength: MPa at age, days		
			2	7	28
NPC-W	3·00	0·50	21·6	32·8	43·8
SFC-W	3·00	0·50	25·5	32·6	51·4
MSC-W	3·00	0·50	28·6	38·4	51·9
MKC-W	3·00	0·50	25·5	49·2	62·1
LWDB-W	3·00	0·50	14·3	22·6	26·6
HPC-A-W	3·00	0·50	31·5	45·6	67·0
HPC-BS-W	3·00	0·50	7·5	21·9	52·1
HPC-BL-W	3·00	0·50	13·3	25·4	33·0

Table 4. Compressive strength of F- mortars (ASTM C109)

Mix type	S/C	W/C	Compressive strength: MPa at age, days			
			1	3	7	28
NPC-F	2·75	0·45	16·9	36·6	39·8	48·9
SFC-F	2·75	0·28	40·6	43·5	53·3	84·1
MSC-F	2·75	0·30	36·5	51·1	55·7	74·5
MKC-F	2·75	0·30	33·0	38·4	45·5	66·8
LWDB-F	2·75	0·30	35·1	59·2	69·0	78·7
HPC-A-F	2·75	0·28	44·3	62·2	74·1	94·4
HPC-BS-F	2·75	0·28	35·2	54·2	65·6	92·7
HPC-BL-F	2·75	0·28	21·6	40·7	54·8	66·7

also developed by HPC-BS. The strength of optimized HPC-A exceeded these values and reached 44·3 MPa. The strength of LWDB was found to be close to the strength of MSC and MKC. All these strength values are much higher than 16·9 MPa demonstrated by NPC.

The behaviour of investigated cements at 3 and 7 days age was similar: microsilica-based HP cement (MSC) demonstrated the best strength 51·1 and 55·7 MPa, respectively. The NPC strength was lower than that of all tested cements.

Strength of mortars at different s/c

The test results of S-mortars (ASTM C387) are presented in Table 5 and Fig. 3. The 28 day compressive strength of HP cement mortars lays in the range of 39–148 MPa depending on s/c. According to the obtained results, the strength of HPC-BS mortars at s/c = 7 is only slightly less than the strength of NPC mortars at s/c = 3.

Discussion

Strength development

The test results confirmed a similar effect of different reactive silica products on the strength properties of HP cement at a reduced w/c. The three different stages of cement strength development can be specified as follows

(*a*) 1st stage: very rapid strength growth during first day of hardening
(*b*) 2nd stage: latent period between 1 and 3 days characterised by a delay in hardening, especially noticeable for SFC and MKC
(*c*) 3rd stage: more intensive, strength development of SFC and MKC, comparing to HPC-A and MSC, at the age of 3–28 days.

The strength development of investigated cements at a constant w/c of 0·50 has a clearly defined critical point at 2 days age (Table 3). According to the test results, MKC maintained a very high strength up to 7 days age, giving remarkable strength values. The continuous strength growth up to 28 days age was observed for HPC-BS. LWDB demonstrated very little increase in strength after 7 days age.

HP cement versus LWDB

The research has demonstrated the advantage of HP cement over LWDB for early and 28 day strength in the range of the investigated w/c. This advantage is even multiplied at high w/c, when HP cement showed 2–2·5 times higher strength (Fig. 3). It can be suggested that the criterion similar to the performance of plasticisers or superplasticisers in concrete,[13] which allows only very small strength loss of modified concrete (being tested at the same w/c), must be applied in the case of new cements with low water demand. This procedure could provide a better understanding of the behavior of these cements and also, prevent an unexpected strength loss of the concrete at high w/c.

W/c effect

The function of the HP cement compressive strength versus w/c is presented in Fig. 3. The 28 day compressive strength value for HP cement mortars can be estimated by the following equation

$$f_{28} = \frac{17{\cdot}283}{W/C} + 24{\cdot}725 \qquad (1)$$

According to the empirical rule stated in,[1,2] the pozzo-

Table 5. 28-day Strength of HP Cement Based Mortars

Mix Type	Flexural strength: MPa at S/C				Compressive strength: MPa at S/C			
	1	3	5	7	1	3	5	7
NPC-S	12·2	8·9	5·3	3·7	70·2	43·8	22·3	12·9
HPC-BS-S	24·5	12·8	8·3	5·9	148·1	96·4	60·8	39·8

lanic effect of highly reactive silica dioxide materials (such as silica fume) is approximately equal to the strength of the cement used. In this case, the difference in strength between NPC and HP cement (or SF cement) at the same w/c can be explained by the contribution of mechano-chemical activation, which was also observed in Reference.[23] The optimal composition of HP cement allows a significant reduction of the w/c ratio keeping a high workability level with consequent increase in strength due to super-dense packing.

Sand to cement ratio

According to Table 5, the s/c of mortars has a significant effect on the compressive and flexural strength of mortars. It can be noticed that in tests involving high strength cement the s/c is one of the most critical factors limiting w/c and workability.[1,2,26] When the s/c is reduced to 1, high strength (up to 148 MPa) mortars based on HP cement with required fluidity level can be obtained at very low w/c of 0·15–0·18.

The application of the standard s/c (2·75–3·00) imparts a certain volume of voids (determined by the voids content of the sand) to be filled with the cement paste. This volume is critically high when cements with low water demand are used (since a certain volume of cement paste must be used to occupy the voids between sand particles to maintain the required fluidity level). This dictates the addition of excessive water (because cement content is fixed), which increases the w/c, otherwise it causes air entraining or air entrapping (if volume of cement paste is not sufficient). The optimising of the composition of high strength mortars cannot be achieved in such conditions. A solution was specified in LWDB pre-standard by replacing 50% of sand with ground sand. An alternative solution was found to decrease s/c to its minimum level in high strength concrete.[1,2] Actually, ASTM has already set a reduced s/c for special cements. According to References,[1,2] the s/c equal to 1·0 is suitable for the optimisation of mortars with a superplasticiser and mineral additives. Because of extremely dense compaction and a very low water demand the s/c of 1·0 corresponds to its minimal boundary in high strength concrete, optimised for maximal strength. The same approach can also be suggested for testing HP cements or LWDB-based mortars.

The comparison of EN 196 and ASTM C109 test procedures

The comparison of the test results demonstrated that the EN 196 procedure cannot be adopted for measuring the performance of newly developed high strength cements. Designed to compare portland cements of different manufacturers by its adjustment to a 'common divider', the compressive strength obtained at constant w/c is not sufficient to predict the performance of the different types of cement in concrete (for example, pozzolanic cements with high value of normal consistency). In the practical application of these types of cement the comparison of its performance in concrete may be required when the EN 196 test procedure is used. This test should consider not only the strength of concrete, but also cement effect on workability of concrete mixtures.

Since the constant flow procedure of ASTM C109 returns a compressive strength as an integrated value involving workability parameter, it can be applied as a performance criterion to a wide range of cements. As a result, the ASTM strength can be directly used for the prediction of concrete strength based on different types cement. Therefore, the ASTM C109 procedure is suggested as the most appropriate strength test for high strength cements such as HP cement or LWDB.

The demand for high strength cement standard

Obviously, the development of existing standards and the creation of new standards are needed to test improved materials. In spite of the evident advantages of high performance and high strength cements, the lack of corresponding Standards constrains their application.[11,21–26] Summarising the available experience with HP cement, the following strength test procedure is suggested

(*a*) The compressive strength test of mortar at constant w/c of 0·5 and s/c of 3·00 (similar to EN 196) must return a strength value not less than that of 'conventional' Portland cement used for high strength cement manufacturing. If portland cement strength is not specified, the minimum value of 50 MPa must be applied.

(*b*) The compressive strength test of mortar at constant flow of 105–115 mm and s/c of 2·75 (ASTM C109) must return a strength value 50% higher than that of 'conventional' portland cement or at least 70 MPa.

(*c*) The compressive strength test of mortar at constant flow of 105–115 mm and s/c of 1·00, (ASTM C387), must return a strength value 50% higher comparing to 'conventional' portland cement or at least 100 MPa.

The comparison of these results provides a better insight into the behaviour of high strength cement in concrete, which is important for both the proportioning of the concrete mixture and for the prediction of its performance.

Conclusion

(*a*) Reactive silica additives like silica fume, natural microsilica and metakaolin increase the early and ultimate strength of cement and concrete. These materials can be used as components of a complex admixture for consequent application in high performance cement. The test results demonstrated similar behaviors of different reactive silica additives in high performance cement.

(*b*) Using the HP cement approach, cements with 28 day strength (according to ASTM C109) in the range of 66·8–94·4 MPa were obtained.

(*c*) The w/c ratio was recognized to be the most critical parameter in measuring the performance of high strength cement. The difference in strength according to different standards (EN 196 versus ASTM C109) was found to be in the range of 10–60% for different types of HP cement, and about 200% for LWDB. Based on completed research, the ASTM C109 is proposed as the most convenient procedure for testing high strength cements.

(*d*) It is also suggested that the strength of high strength cements should not be less than that of normal Portland cement at w/c specified by EN 196.

(*e*) The development of a new standard for high performance and high strength cements is suggested to encourage the manufacture and wide- scale application of these new cements.

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