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Effects of mix composition and water–cement ratio on the sulfate resistance of blended cements

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Abstract

This paper presents an experimental investigation on the sulfate resistance of blended cements containing various amounts of natural pozzolan and/or Class-F fly ash. The performance of blended cements was monitored by exposing the prepared mortar specimens to a 5% Na_2SO_4 solution for 78 weeks. For comparison, an ordinary Portland cement (produced with the same clinker as blended cements) and a sulfate resistant Portland cement (produced from a different clinker) were also used. In addition to the cement chemistry, water–cement (w/c) ratio of mortars was another parameter selected that will presumably affect the performance of mortars. The experimental results of expansion measurements showed that the effect of w/c ratio was more pronounced for the low sulfate resistant cements with higher C_3A amounts, while the blended cements were less affected by an increase in the w/c ratio.

Keywords: Blended cement; Sulfate attack; Fly ash; Natural pozzolan

1. Introduction

External sulfate attack which was first identified in 1908 by the United States Bureau of Reclamations [1] is one of the durability problems associated with concrete. Since its identification it has been the subject of numerous studies and still not totally understood [2]. The extent to which concrete is affected by sulfates depends on several factors including its permeability, water to cement (w/c) ratio, type of cement, exposure conditions and the environment [3]. Assuming the same environmental conditions, two factors will tend to control the resistance of a given concrete to sulfate attack: the chemistry of the cement and the permeability of the concrete.

- To control the cement chemistry, American Standards [4] suggest a limit on the (C_3A) and ($2C_3A + C_4AF$) contents of sulfate resistant, type V, cements as 5% and 25%, respectively. On the other hand, cements with low C_3A and C_4AF compounds generally tend to have a higher C_3S/C_2S ratio, and an increase in the C_3S content of cement generates a significantly higher amount of calcium hydroxide, as the hydration of C_3S produces nearly 2.2 times more calcium hydroxide (CH) than the hydration of C_2S. CH is known to be responsible for the formation of gypsum, and gypsum is known to be the first step of the formation of ettringite, which can be considered as the principal cause of deterioration [5]. Irassar et al. found mortar bars containing a low C_3A and low C_3S cement showed 10 times less expansion than those with a low C_3A and a high C_3S content [6].
- To control the permeability of concrete, lower w/c ratio and/or pozzolans are recommended [7,8]. Effect of various pozzolans on the resistance of cements to external sulfate attack has also been studied by other researchers [9–11]. Pozzolans reduce not only the permeability but also the C_3A amount if they are a partial replacement of cement. Moreover, use of pozzolans or use of blended cements, in general, reduces the quantity of CH due to the pozzolanic reactions which would otherwise react with sulfates to form gypsum.

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The abovementioned parameters, i.e. the cement composition and w/c ratio are the subject of this study. In order to investigate the effects of these two parameters against external sulfate attack, ordinary, blended and sulfate resisting cements were used to prepare cement mortars with two different w/c ratios, thus different permeabilities. These cement mortars were then immersed in a 5% Na_2SO_4 solution and compressive strength and length change (expansion) of mortar specimens were determined after various periods of immersion. For a better explanation of their performance against sulfate attack, X-ray diffraction (XRD), X-ray energy dispersion analysis and scanning electron microscopy (SEM) of cement pastes subjected to the same solution were also used.

2. Materials and experimental program

2.1. Cements

An ordinary Portland cement (OPC) and five different blended cements were produced with different proportions of clinker, natural pozzolan, fly ash and limestone. The chemical analysis of these raw materials is given in Table 1. The natural pozzolan was obtained from Bilecik and Yenişehir, the low-lime fly ash was obtained from the Seyitömer power-plant, and the limestone with a $CaCO_3$ amount of 95% was obtained from Bursa, Turkey. Labeling for all cements together with their ingredients is provided in Table 2. For comparison a sulfate resistant Portland cement (SRPC) with a different clinker was also obtained. The chemical composition and the major compounds of all Portland cements together with their physical properties are presented in Table 3.

2.2. Mixtures

Mortar mixtures were prepared using the cements mentioned above and the Rilem Cembureau Sand meeting the requirements of ASTM C 778. In all mortar mixtures cement:sand ratio was kept constant as 1:2.75 by weight. Seven mixtures were first prepared with a w/c ratio of 0.485, and flow and consistency characteristics of these mixtures were determined. Later, another set of seven mixtures were prepared by changing the w/c ratio in order to obtain similar consistency of the blended cements with the previously determined consistency of the OPC and SRPC mortar mixtures. The flow characteristics as determined by ASTM C 1437 and w/c ratios of all 14 mixtures are presented in Table 4. As seen in Table 4, the mixtures can be approximately grouped into two different w/c ratios such as 0.485 and 0.560.

Table 1
Chemical composition of materials

Chemical analysis (%)	Materials used in OPC and blended cement production			
	Clinker	Natural pozzolan (NP)	Fly ash (FA)	Limestone
SiO_2	20.63	66.44	57.10	1.36
Al_2O_3	6.09	12.11	18.67	1.05
Fe_2O_3	3.74	1.78	9.75	0.61
CaO	65.28	5.13	4.71	52.84
SO_3	0.94	1.76	0.67	0.23
MgO	0.44	0.95	4.43	0.28
Na_2O	0.53	0.63	0.38	0.04
K_2O	0.48	2.71	2.05	0.13
Loss on ignition (LOI)	0.36	7.15	2.13	41.93

Table 2
Material proportions of OPC and blended cements

Cement		Material (%)			
Label	Name	Clinker	NP	FA	Limestone
OPC	CEM I 42.5R	96.5	0.0	0.0	3.5
BC	CEM II/B-M(P-V) 42.5N	70.8	10.8	14.9	3.5
BC_{FA}	CEM IV/A-V 32.5R	64.7	0.0	31.8	3.5
BC_{NP}	CEM IV/A-P 32.5R	66.3	30.2	0.0	3.5
$BC_{NP–FA}$	CEM IV/B(P-V)32.5R	61.2	22.2	13.1	3.5
$BC_{FA–NP}$	CEM IV/B(P-V)32.5R	60.3	15.3	20.9	3.5

From each mixture, 25 × 25 × 285 mm prismatic mortar bars and 50 mm cubes were cast. The prismatic mortar bars were used for length measurements and the cubes were used to determine the compressive strength. After casting and finishing, the molds were covered with plastic sheets and stored for 24 h in a moist room (relative humidity: above 95% and temperature: 35 ± 3 °C). After the initial curing period, the specimens were demoulded and cured in lime saturated water at 23 °C until the mortar cube specimens gained compressive strength of 19.5 MPa or higher as described by ASTM C 1012. Upon reaching a compressive strength of 19.5 MPa (0-week), all prismatic mortar bars were stored in a 5% sodium sulfate (Na_2SO_4) solution (50 g/L). Length measurements of the prismatic specimens were performed at 1, 2, 3, 4, 8, 13, 15, 17, 26, 52 and 78 weeks after immersing the specimens into the sulfate solution. As the bars were cracked (Fig. 1) length change measurements was not possible for OPC mortar bars with a w/c of 0.560 at the end of 26 weeks, and for OPC mortar bars with a w/c of 0.485 at the end of 52 weeks. In the same way, after reaching compressive strength of 19.5 MPa, cube specimens were stored in a 5% Na_2SO_4 solution at 23 °C. The compressive strength measurements of the cubes were performed after 4, 26 and 52 weeks. In addition to the cement mortars, cement pastes that has equal w/c ratios with mortars were also prepared and subjected to the same sulfate solution. They were cast in plastic cylinders and demoulded the next day. After demoulding, the paste samples were cured in lime-saturated water for an additional 27 days. The paste samples were then exposed to 5% Na_2SO_4 solution at 23 °C. After the sulfate exposure period X-ray diffraction analysis (XRD) and scanning electron microscopy (SEM) was conducted on the paste samples. XRD

Table 3
Chemical composition, major compounds, and physical properties of cements

		OPC	BC	BC_{NP}	BC_{FA}	$BC_{NP–FA}$	$BC_{FA–NP}$	SRPC
Chemical analysis (%)								
SiO_2		20.12	29.17	34.57	29.43	34.38	33.58	19.65
Al_2O_3		5.75	8.73	8.48	9.84	8.96	10.02	4.27
Fe_2O_3		3.26	4.56	3.49	5.44	4.18	5.05	4.55
CaO		63.44	47.47	42.89	45.84	41.08	41.07	63.10
SO_3		2.71	2.51	2.49	2.50	2.47	2.49	0.95
MgO		0.98	1.54	1.15	1.78	1.38	1.70	2.48
Na_2O		0.43	0.62	0.56	0.55	0.59	0.62	0.63
K_2O		0.49	0.95	1.07	0.95	1.09	1.08	0.45
LOI		2.13	3.17	5.12	2.38	4.13	4.08	3.54
Compound composition (%)[a]								
C_3S		57.9	42.5	39.8	38.8	36.7	36.2	69.6
C_2S		13.4	9.8	9.2	9.0	8.5	8.4	3.8
C_3A		9.5	6.9	6.5	6.3	6.0	5.9	3.6
C_4AF		11.0	8.1	7.5	7.4	7.0	6.9	13.8
Physical properties								
Sp. gravity		3.18	2.87	2.89	2.75	2.78	2.75	3.21
Blaine fineness (cm^2/gr)		3629	4062	4432	4772	5000	4676	3090
Comp. strength (MPa)	2 d.	22.0	16.7	16.0	17.9	14.6	12.9	22.8
	7 d.	45.1	32.5	32.9	31.1	29.3	24.2	39.8
	28 d.	55.8	49.0	49.0	50.8	46.8	42.6	53.7
	180 d.	60.9	55.3	55.2	61.6	56.0	53.8	67.6

[a] Compound compositions were calculated using compound composition of clinker (Bogue's equations) and mineral addition content.

Table 4
Flow properties of mortar mixtures

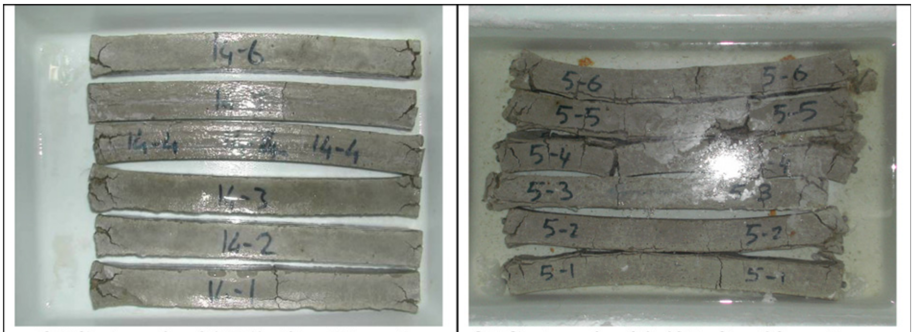
Label	Group 1 (w/c = 0.485)		Group 2 (w/c ≈ 0.560)	
	w/c	Flow (%)	w/c	Flow (%)
SRPC	0.485	81	0.570	Overflow
OPC	0.485	83	0.570	Overflow
BC	0.485	26	0.540	80
BC_{FA}	0.485	32	0.550	79
BC_{NP}	0.485	20	0.550	76
$BC_{NP–FA}$	0.485	21	0.570	80
$BC_{FA–NP}$	0.485	17	0.570	79

measurements were performed by a diffractometer using Cu Kα radiation with a wavelength of 1.54 Å, operating at 40 kV and 40 mA. Step scanning was used with a scan speed 2°/min and sampling width of 0.02° 2θ.

3. Results and discussion

3.1. Compressive strength

The compressive strengths computed as an average of three cubic specimens subjected to a 5% Na_2SO_4 solution are presented in Table 5. The 0-week in Table 5 is the time when the mortar cube specimens reached the compressive strength of 19.5 MPa or higher. As seen in that table, when subjected to continuous sulfate exposure all cements showed an increase in compressive strength up to at least 26 weeks. This was attributed to the hydration of calcium silicates and to the pozzolanic reactions of blended cements. However, after 26 weeks, the compressive strength of the OPC mortars began to decrease drastically for both w/c ratios. This was an expected result, as ettringite formation leading to an expansion, cracking and drastic



OPC at a w/c of 0.560 after 17 weeks **OPC at a w/c of 0.485 after 38 weeks**

Fig. 1. OPC mortar bars subjected to a 5% Na_2SO_4 solution.

Table 5
Compressive strength of mortars subjected to a 5% Na_2SO_4 solution

Test age (weeks)	Compressive strength (MPa)						
	OPC	BC	BC_{NP}	BC_{FA}	BC_{NP-FA}	$BCBC_{FA-NP}$	SRPC
w/c = 0.485							
0[a]	23.8	22.2	19.5	19.4	20.1	19.6	19.1
4	45.9	39.7	42.4	33.2	37.5	39.9	47.7
26	49.2	49.9	47.0	54.4	57.1	44.4	62.1
52	33.1	48.3	48.0	52.4	55.4	51.5	64.3
w/c ≈ 0.560							
0[a]	21.9	21.9	21.1	20.7	19.5	19.5	20.1
4	45.3	46.4	35.0	49.4	37.1	38.8	45.8
26	34.4	46.8	47.1	53.2	39.0	44.3	55.9
52	–[b]	53.0	47.9	52.0	42.6	48.4	63.5

[a] 0-week corresponds to the start of the sulfate exposure.

[b] Measurements could not be conducted as the specimens disintegrated.

reduction in the strength and gypsum formation leading to a reduction of stiffness and strength were the major form of deterioration of mortars containing cements with a high C_3A content [12,13]. SRPC cubes resulted in the highest strengths at all ages and did not show significant strength loss. This was possibly due to the filling of the pores by the reaction products of sulfate attack [14]. The beneficial effect of pore filling to strength can be considered for OPC mortars too. However, the reduction in strength of OPC mortars shows that the destructive effect of reaction products (gypsum and ettringite) dominated their contribution to strength. When the blended cements are considered, they do not show any significant strength loss until 52 weeks of continuous exposure. This result is consistent with the results of mortar bar expansions (Table 6), where the blended cements are found to be highly resistant to the sulfate exposure except of BC_{NP}. Although BC_{NP} specimens demonstrated relatively higher expansion (it will be discussed in Section 3.2), they did not deteriorate and develop any significant crack for both w/c ratios after the 52 weeks continuous exposure period. The continuous increase in strength of BC_{NP} cubes for both w/c ratios can be attributed to pozzolanic reactions and filling of the pores by the reaction products of sulfate attack. In literature, similar observations were also found by other researchers [14–16].

3.2. Expansion

The expansion test results of the mortar bars subjected to a 5% Na_2SO_4 solution are shown in Fig. 2 for all cements. When the dashed and solid lines corresponding to the w/c ratios of 0.485 and 0.560 respectively are compared, it can be easily observed that lowering the w/c ratio tends to reduce the expansion of most cement mortars. However, this effect is not the same for all cements. For the w/c ratios considered in this study, the sulfate resistance of OPC is highly sensitive to w/c ratio, compared with other types of cements where the effect of w/c ratio is minimal.

Cements satisfying the expansion limits of 0.10% and 0.05% at 26 weeks are considered as moderate sulfate resistant and high sulfate resistant, respectively and the expansion limits of 0.10% at 52 weeks are considered as high sulfate resistant by ASTM Subcommittee C01-29 [17]. A summary of the 26, 52, and 78-week expansions for all mortar mixtures are summarized in Table 6 and shown in Fig. 3. It can be seen from Table 6 and Fig. 3 that cement type is a factor in determining the performance of cement in a sulfate environment. It is clearly seen from Table 6 that OPC is not suitable for sulfate environments at both w/c ratios. Moreover, it can also be concluded that all blended cements with the Class-F fly ash showed better performance, when compared to the SRPC and the blended cement having only the natural pozzolan (Fig. 3). In order to quantify the effect of w/c ratio on the increase in expansion at a given age, change in the expansion of mortar bars when the w/c ratio is increased from 0.485 to 0.560 was calculated for all cement mortars as shown in Fig. 4. (Note that in this figure for OPC cement mortars extrapolation of the data towards 26 weeks were conducted as the OPC cement mortars cracked before 26 weeks.) As seen from this figure the effect of w/c ratio is relatively lower for all blended cements. However, expansion measurements did not dictate any trend as the age of exposure changed from 26 to 78-weeks.

All blended cements containing natural pozzolan and/or fly ash have a notable reduction in expansion at all test ages. Expansion reduction was drastic in mortars with BC_{FA} and BC_{FA-NA} cements. Both blended cements may

Table 6
Summary of the average 26, 52 and 78-week expansions

Label	w/c	Expansion (%)			w/c	Expansion (%)		
		26-week	52-week	78-week		26-week	52-week	78-week
OPC	0.485	0.328	–[a]	–[a]	0.560	–[a]	–[a]	–[a]
SRPC	0.485	0.075	0.116	0.168	0.560	0.073	0.168	0.230
BC	0.485	0.025	0.052	0.056	0.560	0.060	0.080	0.099
BC_{NP}	0.485	0.076	0.149	0.210	0.560	0.089	0.193	0.260
BC_{FA}	0.485	0.035	0.048	0.055	0.560	0.037	0.048	0.052
BC_{NP-FA}	0.485	0.057	0.089	0.110	0.560	0.074	0.098	0.122
$BCBC_{FA-NP}$	0.485	0.037	0.051	0.053	0.560	0.055	0.065	0.074

[a] Measurements could not be conducted as the specimens disintegrated.

be classified as high sulfate resistant cements for both w/c ratios. The pozzolanic activity of the fly ash and natural pozzolan binds it to CH released in the hydration of calcium silicates (C_3S and C_2S), so CH is no longer available for reaction with sulfates. This prevents the formation of gypsum. Pozzolanic reaction produces a secondary C–S–H that also decreases the capillary porosity of mortars and enhances significantly the paste–aggregate interface.

Moreover, the fly ash used in this study has a total SiO_2, Al_2O_3 and Fe_2O_3 content of 85.52%, CaO content of 4.71% and SO_3 content of 0.67. According to Tikalsky and Carrasquillo, all these chemical properties indicate that this

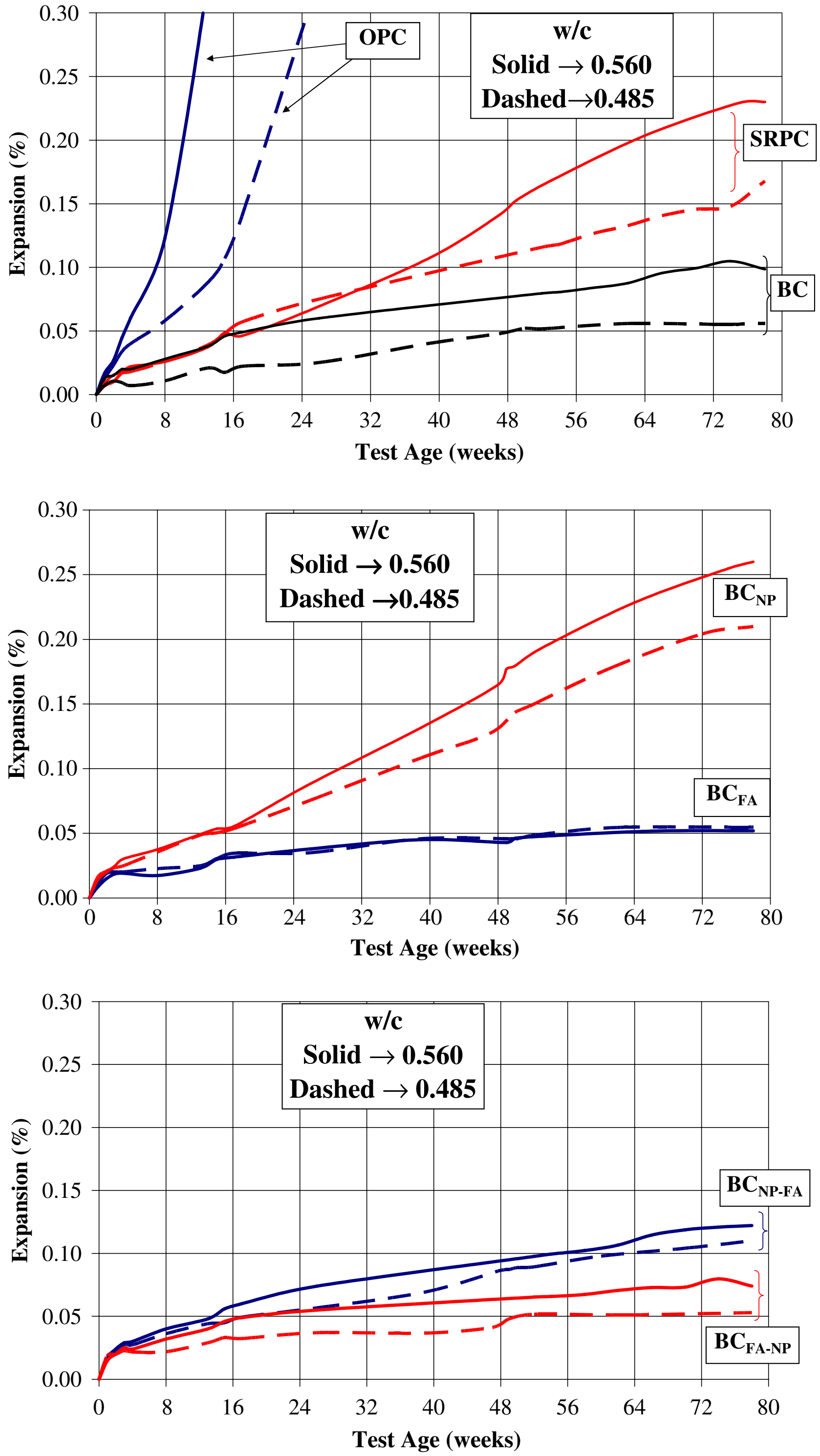


Fig. 2. Expansion of mortar bars.

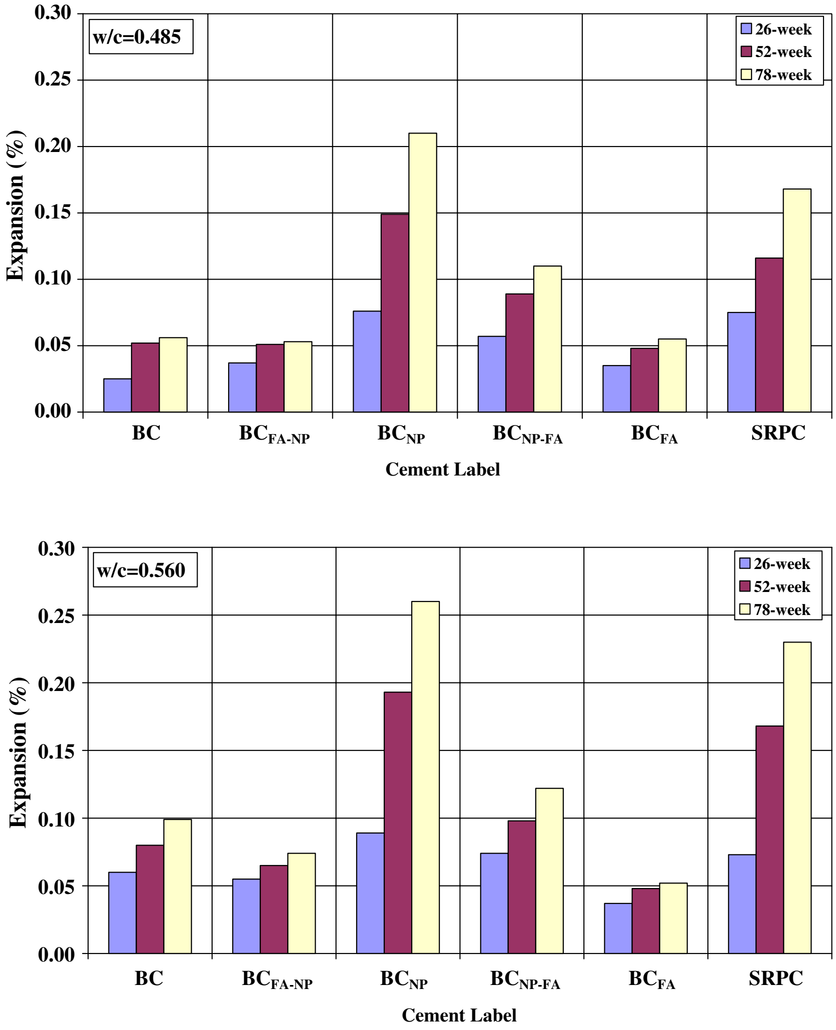


Fig. 3. 26, 52 and 78-week sulfate expansion of mortar bars.

class of fly ash will improve sulfate resistance [18]. The use of natural pozzolan improves the sulfate resistance of cements, principally, due to the CH reduction in mortars. Irassar et al. found that natural pozzolan addition reduce the expansion and strength loss of mortars with low C_3A (0–1%) portland cement [6]. However, the clinker used for the production of blended cement in this study has a high C_3A content. Therefore, blended cement with high natural pozzolan does not perform as well as the blended cement with high fly ash.

In the SRPC used in this study, the C_3S/C_2S ratio, which dictates the amount of CH in the hardened cement paste, is increased with the reduction of the C_3A content. The CH in the hardened cement paste reacts with sulfate ions to form gypsum. The deterioration of hardened mortar by the formation of gypsum goes through a process leading to a reduction of stiffness and strength loss; this is followed by expansion [12]. The volume of the resulting gypsum is also greater than the sum of its components. The expansion results show that SRPC with high C_3S/C_2S ratio of 18.3 has a poor sulfate resistance. The results of this study were also inline with the findings of Stephens and Carrasquillo [19], where they recommended testing the sulfate resistant Portland cements with a C_3A content of 4–5% before using them in a high sulfate environment.

3.3. XRD and SEM analysis of the cement pastes

In order to investigate the products of hydration, scanning electron microscopy (SEM) of cement pastes was performed. Cement pastes were first produced together with the mortars and these pastes were subjected to a 5% Na_2SO_4 solution. At the end of six months, there was visible cracking and disintegration of the cement pastes prepared from OPC when subjected to sulfates. SEM examinations of fractured pieces obtained from OPC cement pastes revealed formation of ettringite and gypsum (Fig. 5). EDX analysis of Section 1 in Fig. 5a showed only high calcium and sulfur peaks. It was then concluded that the phase was gypsum. EDX analysis of Section 2 in Fig. 5b showed a high calcium peak and smaller sulfur and aluminum peaks, confirming the identification of ettringite. Well-formed and randomly oriented rod-like crystals were observed in the pre-existing pores. These

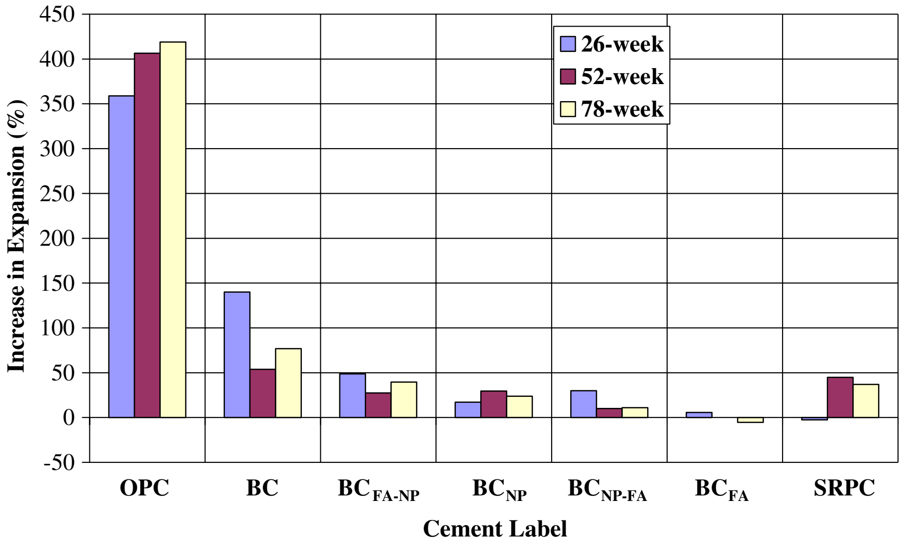
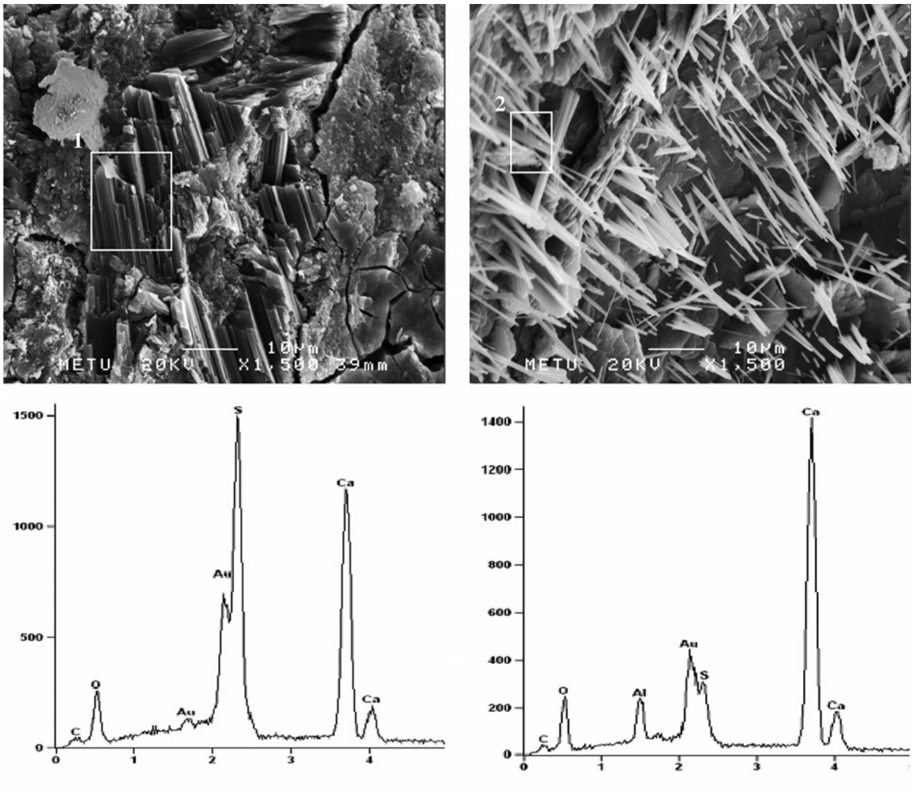


Fig. 4. Change in the expansion of mortar bars when w/c ratio is increased from 0.485 to 0.560.



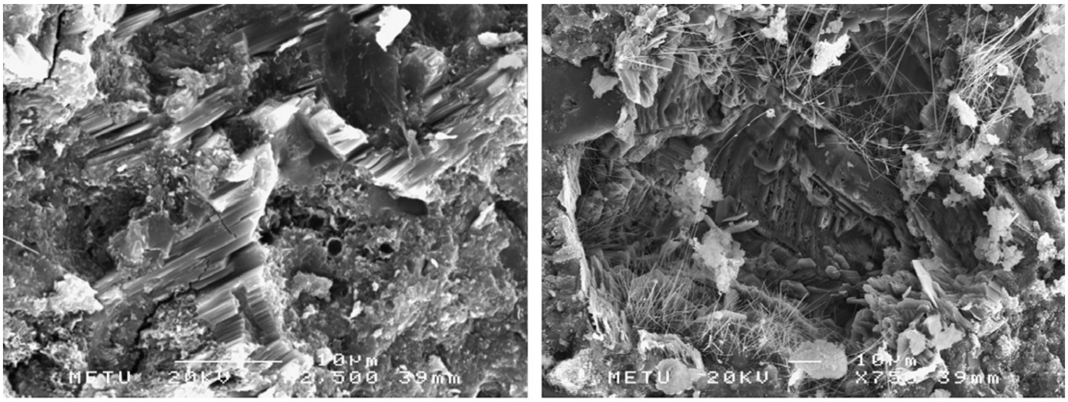
(a) Section 1 - Gypsum **(b) Section 2 - Ettringite**

Fig. 5. SEM and EDX analysis of disintegrated OPC paste at 78 weeks.

observations explained the results of expansion measurements.

Pastes with blended cements that had been exposed to a sulfate solution for a year were also examined in SEM but significantly lesser amounts of ettringite or gypsum was observed (Fig. 6). On the other hand, the expansion measurements revealed that the performance of SRPC was relatively poor when compared to the blended cements. This fact was explained by the SEM and EDX analysis of cement paste. Fig. 7 shows the relatively higher amounts of flower like ettringite needles in SRPC pastes.

To confirm the SEM results and semi-quantitatively identify the products of hydration, five of the cement pastes were examined by XRD after 78 weeks of immersion in a



(a) BC$_{FA-NP}$ **(b) BC$_{NP-FA}$**

Fig. 6. SEM analysis of two blended cement pastes at 78 weeks.

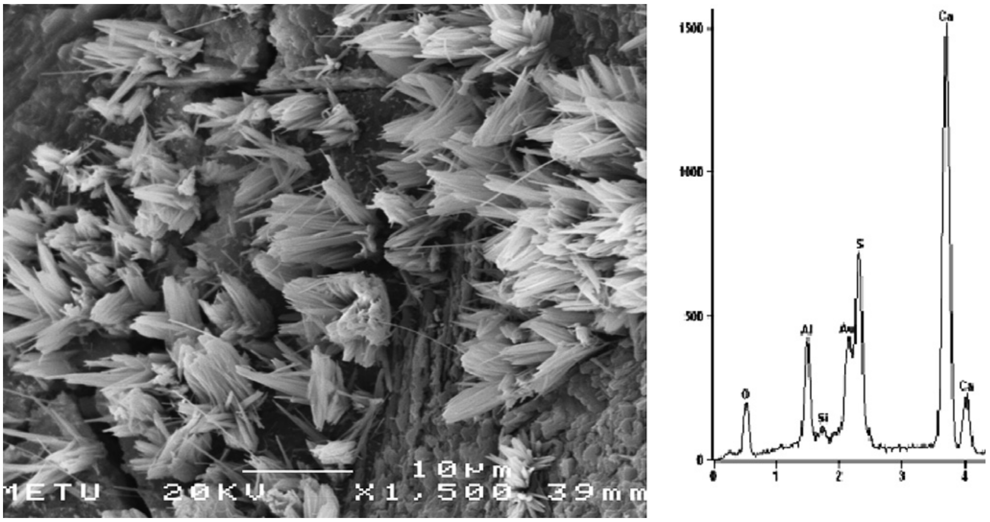


Fig. 7. SEM and EDX analysis of the SRPC paste at 78 weeks.

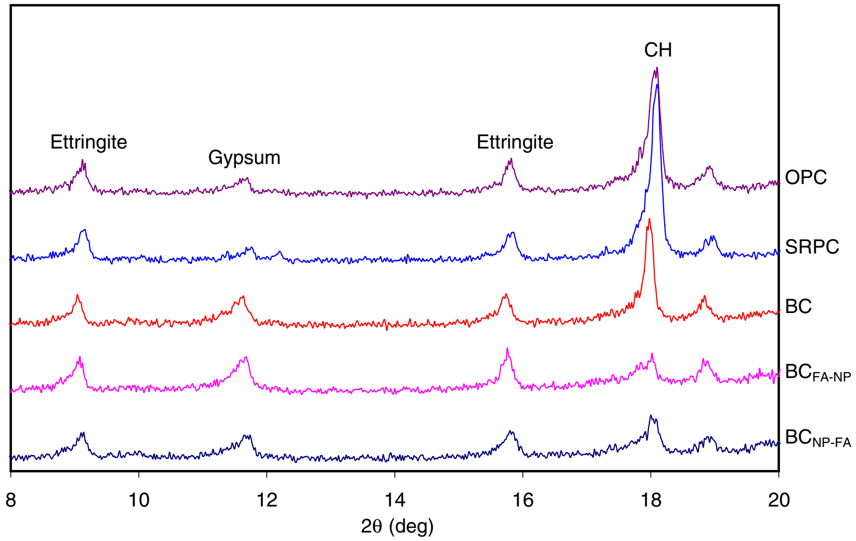


Fig. 8. XRD patterns of cement pastes at 78 weeks.

5% Na_2SO_4 solution. The analyzed samples were taken from the surface of the cement pastes. According to Fig. 8, ettringite and gypsum were identified in all cement pastes. After an exposure period of 78 weeks, the peak of calcium hydroxide (CH) was also detected in all cement pastes. However, the amount of CH was comparatively lower in blended cement pastes due to the pozzolanic reaction of fly ash and natural pozzolan with calcium hydroxide.

4. Conclusions

This paper discusses the results of an experimental program carried out to investigate the effects of w/c ratio and different mineral admixtures at different proportions on the sulfate resistance of cement mortars. Two different w/c ratios (0.485 and 0.560) and two different mineral admixtures (Class-F fly ash and a natural pozzolan) were used. As a result of this experimental study, the following conclusions were made:

- Compressive strength measurements monitored up to a year were not a reliable means for identifying the harmful effects of sulfate attack. Instead, expansion measurements as described in ASTM C 1012 were found to be more effective in identifying the proper cement against sulfate attack.
- Cement chemistry is an important parameter in coping with sulfate attack. Considerable amount of deterioration was observed in the mortar bars prepared with the OPC. For these cements, as the w/c ratio decreased the extent of deterioration drastically decreased. Even though, this was the case, OPC was still not suitable for sulfate environments regardless of the w/c ratio. Reducing the amounts of C_3A and C_4AF compounds prevent the formation of ettringite. However, the SRPC used in this study did not perform well against sulfate attack regardless of the w/c ratio. At both w/c ratios the performance of SRPC was considered to be effective only for moderate sulfate environments. Therefore, performance of an SRPC with a C_3A content of 3.6% needs to be tested before using in a high sulfate environment. For SRPC, the second important parameter seems to be the C_3S/C_2S ratio.
- Blended cements prepared with mineral admixtures (both fly ash and natural pozzolan) improved the resistance of mortars to sulfate attack due to the reduction in C_3A content. Moreover, the pozzolanic reactions of blended cements also reduced the CH content which was necessary for the formation of gypsum. Therefore, depending on the amount and effectiveness of the mineral admixtures, blended cements were considered to be effective for moderate or high sulfate environments. In this respect, the fly ash used in this study performed better than the natural pozzolan. Blended cement, particularly made with Class-F fly ash, performed much better than SRPC with a C_3A content of 3.6% in terms of expansion.
- The effect of w/c ratio is important especially for the low sulfate resistant cements with higher C_3A amounts. High sulfate resistant cements were not affected by w/c ratio to the same extent. As a result, the chemistry of cement appears to be more effective than the w/c ratio in offsetting the destructive effects of sulfate attack on concrete.

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