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The sulphate resistance of cements containing red brick dust and ground basaltic pumice with sub-microscopic evidence of intra-pore gypsum and ettringite as strengtheners

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ABSTRACT

This paper presents a laboratory study on the deterioration of blended cement combinations of plain Portland cement (PPC) with red brick dust (RBD) and ground basaltic pumice (GBP). One type of clinker, same Blaine values and two different proportions of additive by mass of clinker, were employed. In addition to these blends, Portland cements without additives were prepared as control specimens.

The compressive strength and the sulphate resistance of cements have been experimentally studied in this paper. A series of laboratory tests were undertaken on all specimens. A large quantity of sheet-like C–S–H was found in the mortars incorporating RBD and GBP. The results indicated that the increase in the additive content caused a significant increase in the sulphate resistance of the mortars. Hence, the studied RBD and GBP can be recommended for use as admixtures in cement production. The development of the particular microstructure including the secondary minerals in the plain and blended cements were studied via SEM analysis. SEM images revealed the presence of ettringite and Portlandite minerals, where the former was most probably responsible for the increase (together with the gypsum roses) as well as a decrease of strength based on its formation at different sites and crystal form. Portlandite was responsible for an increase in the specimen strength.

1. Introduction

The production of environmentally friendly cement-based materials has been one aim of concrete technology. Concrete materials should not only possess good workability in the fresh state and excellent mechanical properties and durability, but also offer environmental and economic benefits [1,2].

Possible technological benefits from the use of natural pozzolans in concrete include enhanced impermeability and chemical durability, improved resistance to thermal cracking, and increase in ultimate strength [3]. Natural pozzolans, which are a variety of pyroclastic rocks, are rich in silicate minerals and volcanic glass shards, and are used in powdered form as concrete additives. They react with calcium hydroxide, in the presence of water, to form compounds possessing cementitious properties [4].

Basaltic tuffs have been used in mixture with lime since historical times in most of the Mediterranean countries. Today, the cement industry in Turkey is one of the most well-established and developed industries with an increasing interest in the use of tuffs as source materials. Almost about one-third of the total production in the recent years was trass cement of a Portland-pozzolan cement mixture [5]. Turkey is rich in natural pozzolan, i.e., the basaltic pumice of the cement industry, where almost 155,000 km^2 of the country is covered by Tertiary and Quaternary-age volcanic rocks, among which tuffs occupy important volumes. Although there are ample geological studies conducted on these volcanic rocks, their potential as natural pozzolan is not well established [6].

Basaltic pumice as a pozzolanic additive contains more than 61% $SiO_2 + Al_2O_3 + Fe_2O_3$. These are the major components of cement that are used to improve the durability, workability and economy of concrete. The cements thus produced conform to the chemical requirements as well as the mechanical requirements concerning compressive and flexural strengths of the ASTM and the Turkish standards [7]. Previous studies showed that both natural and artificial pozzolan can be used effectively in cement

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production. Moreover, the European Cement Standards and the Turkish standards [8,9] allow the use of many different mineral admixtures in the production of CEM II–V. It is necessary to note that Portland composite cements contain at least two different mineral admixtures, at an allowable total amount of 50%, besides the Portland cement clinker. Despite the numerous studies conducted on the durability of mortars, work is under progress in determining the deleterious effects of the environment, namely the most destructive effects of the sulphates present in the soil, groundwater and seawater.

Concrete degradation due to sulphate-bearing environments has been studied, recently, with great concern [10,11]. Despite numerous investigations, there are still areas that require further research. Some properties of Portland cements and optimum blends of mineral additives are among the subjects, which require further research [10,12]. Mineral admixtures have shown to improve sulphate resistance of cements and the composition of clinker, fineness and type of grinding along with the admixture levels [10,13–19].

In a previous study of the first two authors, the sulphate resistance of blended cement incorporating basaltic pumice and ground blast furnace slag has been investigated in detail [20]. Another study carried out by the first author, sought to improve the concrete properties by the use of crushed fire brick and crushed basaltic pumice as aggregate admixtures [21]. In properly laid out brick processing plants, there has been a need to remove huge amounts of dust from the site. This removed quarry dust can amount up to 15% of the total brick production and might possess disposal and environmental problems necessitating the alternative use as an additive. However, studies concerning the use of red brick dust as an admixture are absent in the literature on cement. Hence, this led us to consider the use of red brick dust (RBD) and ground basaltic pumice (GBP) in the production of cement. The Blaine values of plain cement (clinker + gypsum) and blended cement (clinker + RBD + GBP + gypsum) were maintained constant at a value of approximately 300 m^2/kg. An addition of 20% and 30% of the clinker by weight of RBD and GBP was used for the Blaine values. Consequently, this study aimed to reveal the mechanical, as well as the microstructural characteristics of plain Portland cement (PPC) and blended cements, with admixtures of RBD and GBP via compressive strength and sulphate resistance tests that were confirmed by sub-microscopy (SEM micromorphology).

2. Materials and method

2.1. Materials

Red brick dust was obtained from Osmaniye (Southern Turkey), as the major waste material of the area containing considerable amounts of silicon dioxide (SiO_2) of pozzolanic character. The clinker used was obtained from the Adana cement plant, and the GBP samples were obtained from the Osmaniye region. The mixtures for all specimens, the chemical and mineralogical analyses and the physical characteristics of the materials used are given in Tables 1–3, respectively. The chemical analyses of the materials used were carried out according to the TS EN-196.

The widespread basaltic pumice cone deposits are of Quaternary age and are located in Osmaniye with the reserves estimated to be about 1 million tonnes. The pumice comprises an average of 85% volcanic glass and 15% phenocrystic feldspars along with minor spheroid hematite minerals, determined by microscopy. XRD tests show the presence of trace amounts of illite and kaolinite as clay minerals developed on feldspars. The high porosity (30–50%, determined in various basaltic pumice specimens by image analysis conducted by a Quantimet 520 – a Cambridge Leica Instrument) of the basaltic pumice is an advantage for easy and economical crushing [22].

Table 1
The compositions of specimens.

Specimens	Mixtures
A	Clinker + 5% gypsum
B_1	Clinker + 20% RBD + 5% gypsum
B_2	Clinker + 30% RBD + 5% gypsum
C_1	Clinker + 20% GBP + 5% gypsum
C_2	Clinker + 30% GBP + 5% gypsum
D_1	Clinker + 10% RBD + 10% GBP + 5% gypsum
D_2	Clinker + 15% RBD + 15% GBP + 5% gypsum
E_1	Clinker + 5% RBD + 25% GBP + 5% gypsum
E_2	Clinker + 25% RBD + 5% GBP + 5% gypsum

Both admixtures, the basaltic pumice and red brick dust are abundantly found, where the former covers large idle areas on the terrain and the latter is a cost free and environmentally friendly by-product. Both materials can be directly used in cement production without furnacing. Furthermore, they require minimum energies in the crushing process thus, being energy savers.

2.2. Method

The RBD of the Osmaniye Öncü Brick Factory and the GBP collected from the pyroclastic outcrops of Osmaniye, ground by the OYSA Iskenderun Cement Plant's closed circuit ball mill (300 + 10 m^2/kg Blaine value) as well as the clinker of the Adana cement plant were tested separately. The PPC and the blended cements were also mixed in the OYSA test ball mill with 5% gypsum and ground to the same Blaine value as the main ingredients. The sulphate resistance of the blended cements was experimentally studied on mortar specimens immersed in a 5% sodium sulphate solution for 36 months. SEM analysis was performed on the selected mortar specimens based on their compressive strength. The chemical analyses of all materials used were carried out according to the TS EN-196. However, RBD and GBP activity indices were determined according to the ASTM 989 and the behaviour in sulphate solution according to the ASTM 1012. The pH trend of the sulphate solution was observed periodically. The water/binder ratios varied from 0.5 to 0.55.

3. Experimental

3.1. The sulphate resistance of cement mortars

It is generally believed that the content of aluminum and sulphate of the alkali free admixtures may influence the rate of sulphate attack promoting an internal process. Thus, the internal sulphate attack has also been correlated with the presence of sulphate-rich constituents in the mixtures. Moreover, the magnitude of the consequent change was considered via the type of the cement, its dosage and the sulphate present in the clinkers. It is therefore important to determine to which extent the internal sulphate attack may worsen the sulphate resistance of the new alkali free accelerated systems and to compare these latter systems with the alkali-rich ones. Furthermore, an attempt was made to clarify the contribution of gypsum and ettringite to the extent of the damage and expansion. In this context, the confined crystal growth of ettringite results in expansion, which causes the formation of cracks. It appears that this expansion, due to the increasing fraction of ettringite, exhibits oriented crystal growth. Moreover, the ettringite-expansion relationship appears to be related to the curing conditions and depends on the chemical contents. The sulphate concentration of the solution, as well as the aluminate availability, appears to determine the formation of gypsum or ettringite [23].

The attack of sulphate on the completely immersed mortars, up to ages of 36 months, with pH control, and its effect on compressive

Table 2
Chemical and mineralogical analyses of the materials used.

Specimens	Oxides (%)								
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Nal_2O	L.I	T.A%
Gypsum	Crystal water: 19.4					42.8		–	
Adana clinker	20.2	5.5	3.8	64.7	1.9	0.8	0.2	0.1	0.60
RBD	14.5	21.4	36.5	6.4	7.4	1.4	9.2	0.2	1.02
GBP	53.8	14.1	12.1	9.2	8.9	–	1.3	0.3	1.10
	Cement modulus								
Samples	C_3S		C_2S			C_3A		C_4AF	
Adana clinker	63.9		9.7			8.1		11.5	

Table 3
Physical characteristics of the materials used.

Materials	Specific gravity (kg/m^3)	Blaine (m^2/kg)	Pozzolanic activity index with cement at 28 days, % (ASTM C 618)	Fineness residue on 90|am sieve (%)	Residue on 200|am sieve (%)
RBD	2988	300	78	0.5	0.09
GBP	2880	300	72	0.3	0.06
Adana clinker	3190	300	–	0.2	0.08

Table 4
Residue on the sieve (%) analysis of the blended and control cements.

Specimens	Residue on the sieve analysis (%)		
	45 μ	90 μ	200 μ
RBD	2.04	0.25	0.02
GBP	2.22	0.39	0.02
A	0.89	0.26	0.03
B_1	3.31	0.57	0.02
B_2	2.19	0.22	0.03
C_1	2.16	0.29	0.02
C_2	2.28	0.23	0.01
D_1	2.04	0.19	0.02
D_2	1.72	0.15	0.05
E_1	1.67	0.24	0.05
E_2	1.73	0.22	0.04

strength were determined on prismatic specimens containing different amounts of admixtures (denoted as A, B1, B_2, C1, C_2, D1, D_2, E1 and E_2, see Table 1). Over 200 standard prismatic specimens of 40 mm × 40 mm × 160 mm dimensions were cast for experimenting in accordance with the ASTM C1012, in order to determine the resistance of the blended cements against the deterioration of sodium sulphate.

3.2. Microstructure and mineralogy

The hydration products and the microstructure were identified by sub-microscopy (Scanning Electron Microscope – SEM). Selected cement prisms (specimens A – Portland cement with the lowest compressive strength and C_2 blend with the highest compressive strength), that were exposed to ultimate sulphate degradation for 36 months, were cut into cubes of approximately 10 mm^3 size and were placed in a vacuum desiccator for a minimum period of 3 days. Sample surfaces were coated with gold using a BIO-RAD polar Division SEM coating system. The microstructure of the specimens was investigated by a JEOL JEM – 840 Model Scanning Electron Microscope with a TRACTOR –TN 5502 model Energy Dispersion Spectrometer used for point and area chemical analyses. X-ray diffraction analysis was conducted, on the same specimens used for SEM, on a Bruker D8 diffractometer with General Area Diffraction Detector and a 800-mm pinhole collimator for diffractometry with a cobalt target generated at 40 keV and 20 mA and scanning angle from 3° to 90° – 2θ, for determining the total matrix mineral contents in ground specimens and the secondary formations of the in situ samples.

4. Results

4.1. The fineness of the blended and control cements

The fineness of the RBD, GBP, control cement (A) and blended cements, versus the duration of grinding, are shown in Table 4. A similarity was determined by the laser diffractometer in the particle size distributions of the blended and plain Portland cements (Fig. 1).

4.2. Development of compressive strength

The results of the compressive strength test obtained for the blended and control specimens are shown in Table 5, where one could note the clear increases of the compressive strength with the increases of the percentage of additions. The results obtained

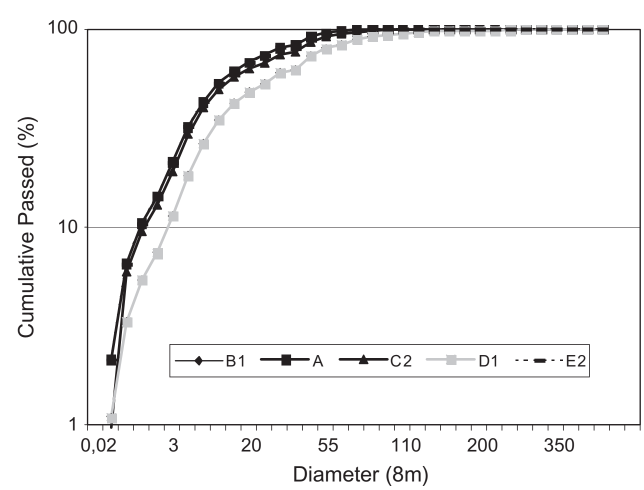

Fig. 1. Particle size distribution of the blended and control cements.

Table 5
The compressive strength of cement specimens stored in tap water (MPa).

Specimens	Time				
	3 days	7 days	28 days	90 days	24 months
A	20.8	34.0	43.2	53.7	58.2
B_1	18.9	31.1	45.4	50.2	61.3
B_2	16.8	30.2	42.6	51.4	62.6
C_1	19.9	33.4	44.2	52.4	68.7
C_2	18.5	34.7	47.5	57.3	72.0
D_1	17.2	32.9	40.2	50.4	70.0
D_2	18.5	34.8	45.6	56.5	71.4
E_1	16.9	30.1	43.4	50.9	63.4
E_2	17.4	33.2	46.9	55.6	70.2

Table 6
The compressive strength of cement specimens in sodium sulphate solution (MPa).

Specimens	Time					
	3 days	7 days	28 days	90 days	24 months	36 months
A	22	32.7	42.4	51.1	42.8	–
B_1	23.4	33.4	46.9	45.7	40.4	35.1
B_2	17.1	31.0	42.9	45.2	39.7	31.4
C_1	20.2	33.5	43.7	51.7	56.9	38.7
C_2	19.3	35.4	48.1	55.1	59.7	39.1
D_1	17.5	33.1	39.7	44.5	53.9	31.6
D_2	18.7	34.7	46.2	54.2	57.3	36.7
E_1	17.5	30.3	42.9	48.7	51.2	35.4
E_2	18.1	33.1	47.2	54.2	55.1	36.6

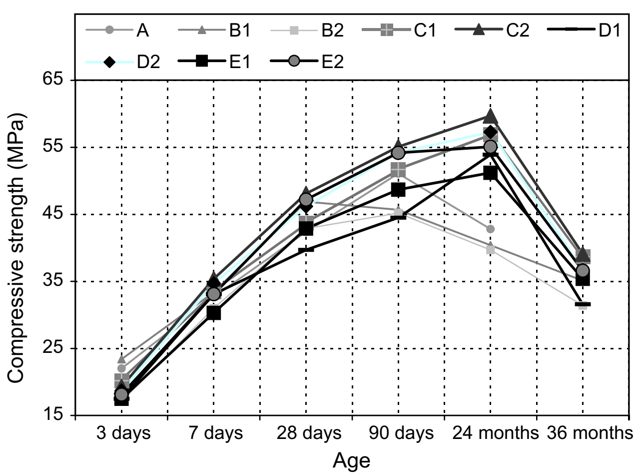

Fig. 2. The compressive strength of cement specimens in sodium sulphate solution (MPa).

for the compressive strengths of the specimens are all acceptable according to the TS EN-196 standards.

Fig. 4. Selected blended cement prismatic mortars (C_2) exposed to sodium sulphate solution for 36 months.

Fig. 5. Plain Portland cement (A) prismatic mortars exposed to sodium sulphate solution for 36 months.

As expected, the compressive strengths of most of the blended cements at early ages (after 3 and 7 days) and some after 28 days are lower than that of specimen A (Table 5). Table 5 shows the

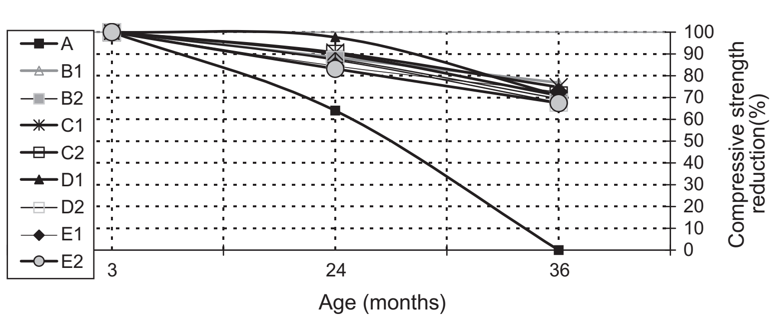

Fig. 3. Compressive strength loss in a sodium sulphate solution.

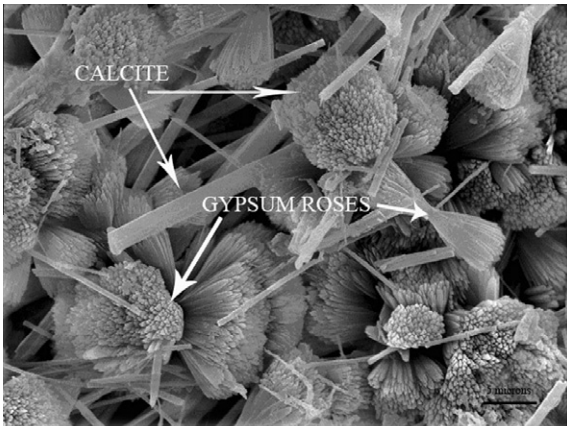

Fig. 6. Gypsum with Acicular Calcite in matrix of specimen C_2.

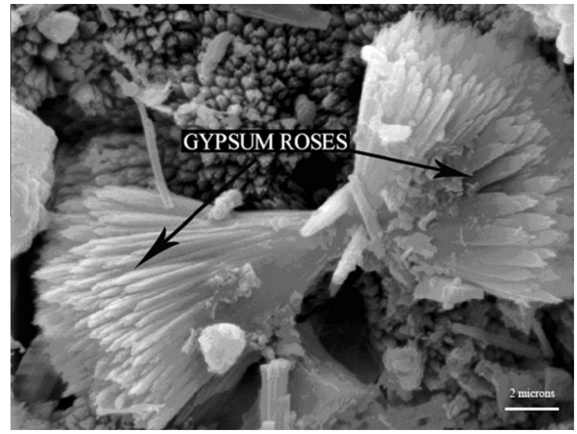

Fig. 7. Gypsum roses in specimen C_2.

compressive strength gain of the mortars from 3 days to 24 months. At the age of 28 days the specimen of plain Portland cement (A) showed a compressive strength increase of 75%. The compressive strength of the blended cement during this period increased by 70% for B–C and by 60% for D–E compared to the plain Portland cement. At the end of the test ages (24 months) specimen C_2 had 20% higher compressive strength than the control cement specimen. These results revealed a clear increase in the compressive strength with an increase in the percentage of the additives.

4.3. The sulphate resistance of cement mortars

The sulphate resistances of blended cement mortars reported by Binici and Aksogan [20] were significantly higher than the Plain Portland cement mortars both against sodium sulphate and magnesium sulphate attacks. Khandaker and Hossain [24], who reported a relatively reasonable compressive strength value, recommended the use of 20% volcanic ash or pumice as a cement additive.

The results of the sulphate resistance test obtained from blended and plain Portland cement samples, in accordance with

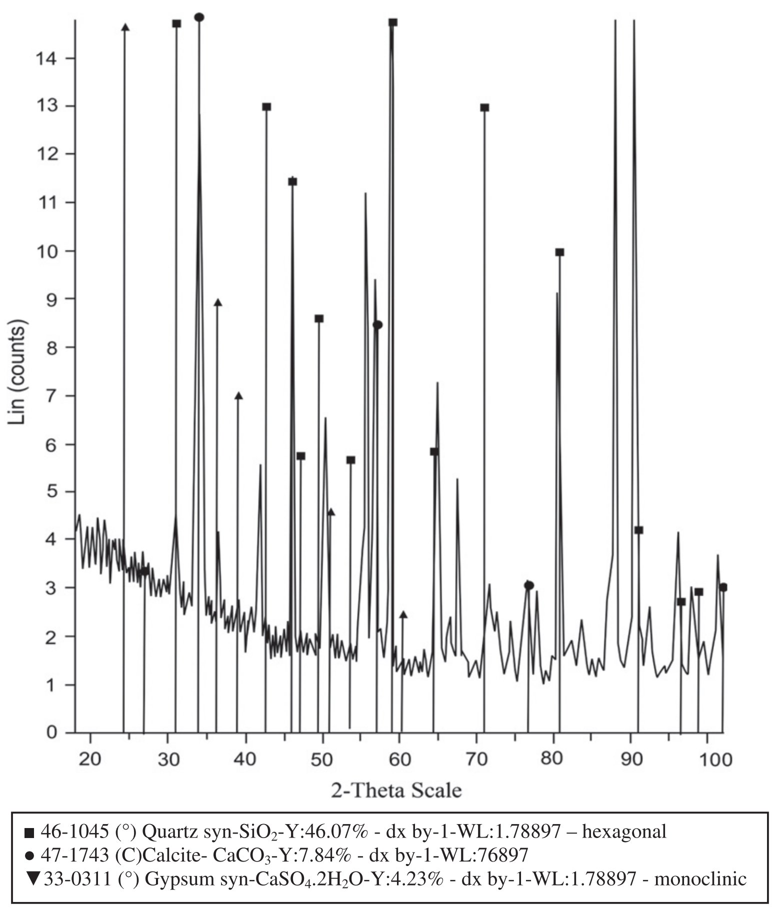

Fig. 8. Minerals of specimen C_2 by X-ray diffraction.

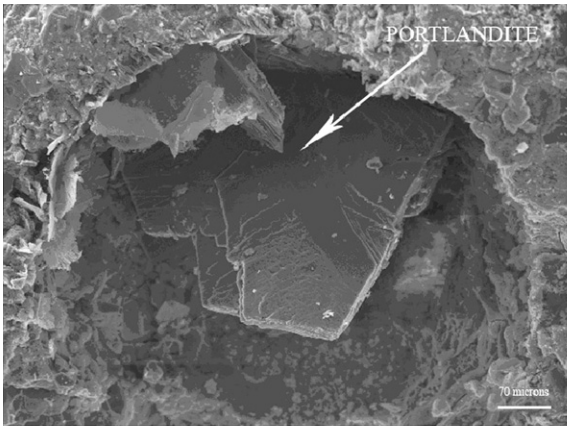

Fig. 9. Platy sheets of Portlandite with C–S–H structures (specimen A).

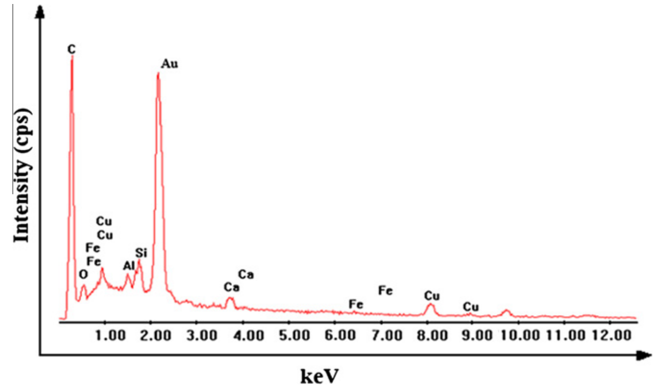

Fig. 10. EDAX of platy Portlandite showing presence of calcium (specimen A) with probable aluminum and silica from the matrix.

the ASTM C1012, are shown in Table 6. The results revealed the increasing resistance with the addition of RBD and GBP in the cement blends. As previously stated, both natural and artificial pozzolans can contribute to enhancing the chemical resistance of concrete [20]. Ground granulated blast furnace slag cement concrete is acknowledged to have high resistance to the aggressive actions of sulphates and seawater [25]. A Similar observation was obtained earlier [26], where metakaolin blended cement mortars were determined to be insignificantly affected by sulphates. Salah and Al-Dulaijan [27] also reported that the use of silica fume and fly ash cements improved concrete durability in terms of sulphate attack.

The data developed in the present study indicate that the type of cement had a significant effect on the sulphate resistance. At 36 months, the maximum deterioration was noted in the control cement mortar specimens (Figs. 2 and 3).

As shown in Fig. 2, the strength reduction in the control cement after 24 months of exposure was less than 65%. After 36 months, the rates of deterioration for the blended cement mortar specimens were different for different specimens. The specimen con-

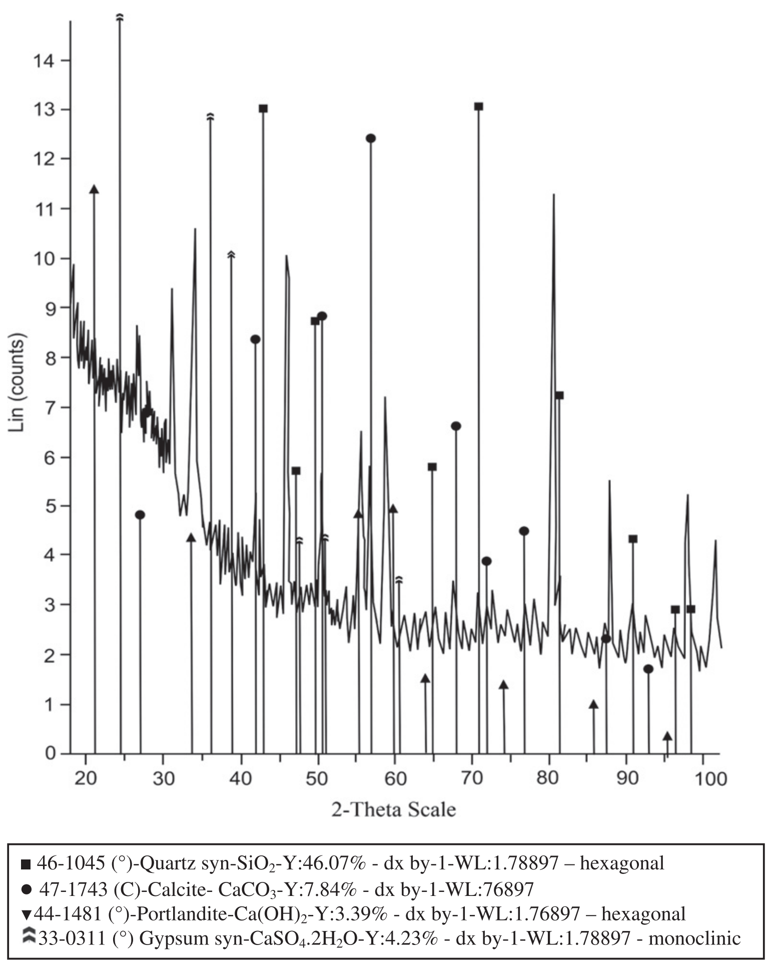

Fig. 11. Minerals of specimen A by X-ray diffraction.

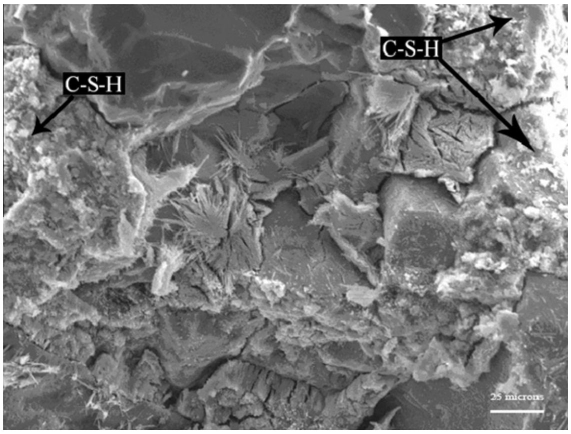

Fig. 12. Radial fibrous clusters ettringite and of C–S–H structures (specimen A).

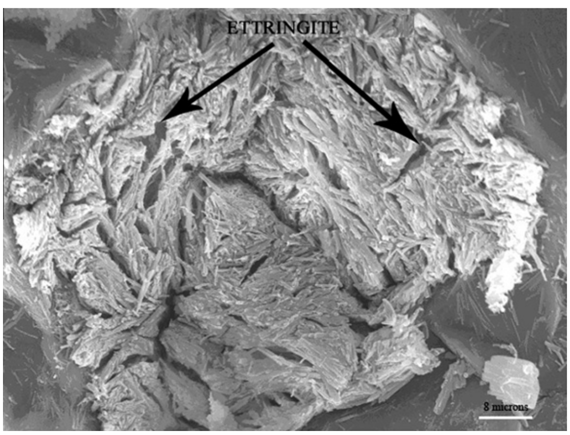

Fig. 13. Ettringite and fibrous aggregates filling pore space (specimen A).

taining only 30% GBP (C2) showed the best sulphate resistance. On the other hand, the specimen containing only 30% RBD (B2) showed the lowest sulphate resistance. However, specimens containing 15% RBD and 15% GBP (D2) showed nearly as high sulphate resistance as the C2 specimen. These results revealed that a good blend would be around 15% for each of the additives.

The visual examination of the specimens after 36 months of exposure also revealed deterioration in the form of disintegration in the control specimens. However, there was no such deterioration in the blended cement mortars (Figs. 4 and 5). This type of failure was observed, also in the previous study of the first two authors [20] and in some other studies [27–29].

4.4. Microstructure and mineralogy

A previous study [30], reported that the primary factor responsible for the progressive damage of the cement mortar due to the erosion of sulphate, was the constant hydration processes. Other factors responsible for this process, were stated to be the nucleation and growth of delayed ettringite and the enhanced damage process caused by the expansive forces attributed to delayed fibrous ettringite.

Despite the development of the weakeners determined by scanning electron microscopy, the blended cement specimen (C_2) has attained the highest compressive strength after 3 days in addition to cementitious properties, reflected by the highest compressive strength obtained after the 36-month curing with sulphate solutions (Table 6). Secondary calcite (acicular) and secondary gypsum,

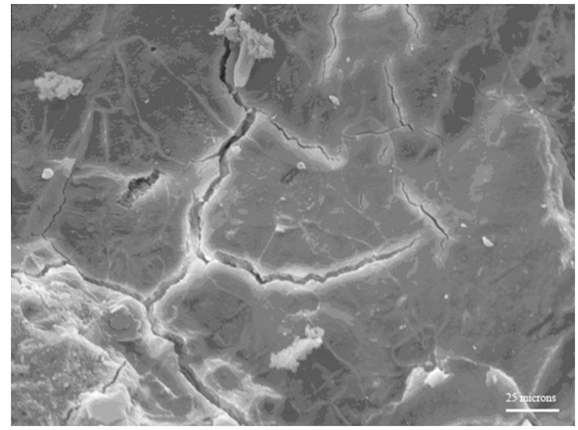

Fig. 14. Patches of C–S–H structures on coating surface on specimen and along cracks (specimen A).

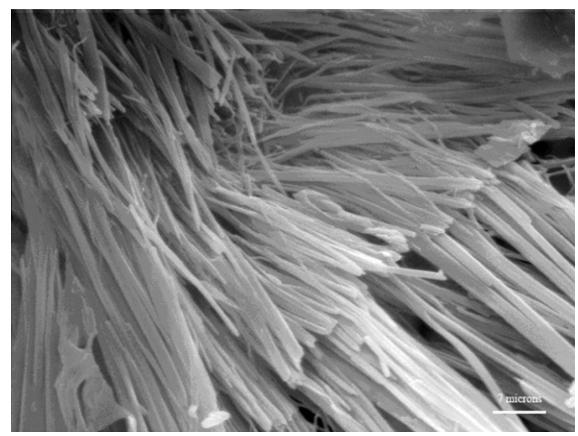

Fig. 15. Fibrous ettringite crystals filling pore spaces (specimen A).

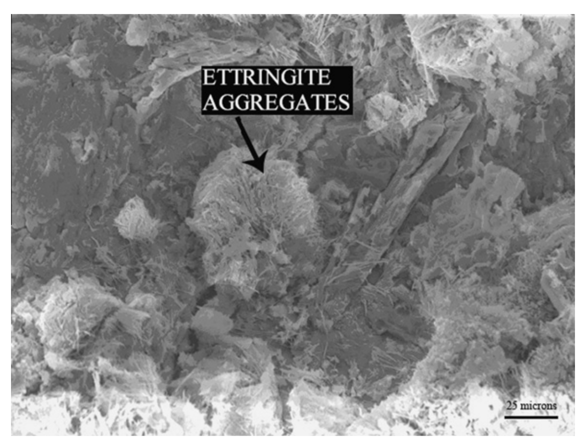

Fig. 16. Randomly oriented and aggregates of ettringite in matrix (specimen C_2).

as radial clusters (gypsum roses) have been determined to develop in the matrix and pores (intra-pore development) of the blend specimen C_2. They document the occurrence of a re-precipitation process during hydration leading to an increase of strength (Figs. 6–8).

In-situ developed mica-like platy sheets of Portlandite (Figs. 9–11), along with CSH structures of short fibres with preferred radial orientation, were occasionally determined to form in pores and matrix most probably following hydration in the non-resistant (Ta-

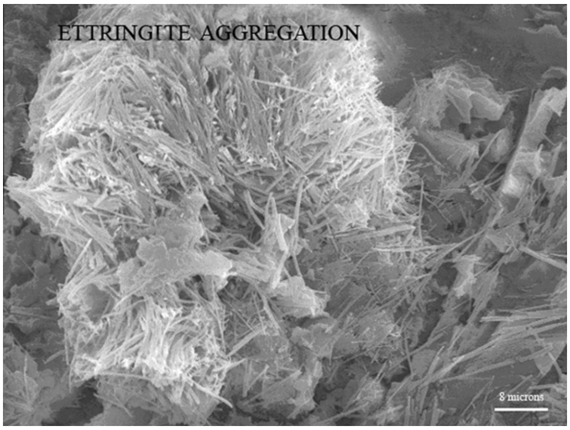

Fig. 17. Aggregates of clewing ettringites (specimen A).

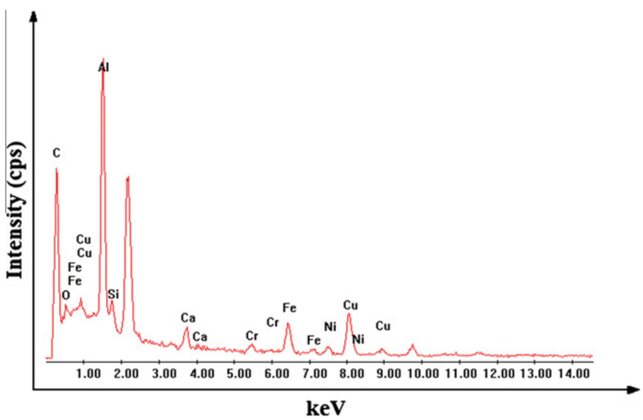

Fig. 18. EDAX of ettringite aggregates with high aluminum (specimen C_2).

ble 5) plain Portland cement specimen (A). Intra-pore Portlandite may have also contributed to the increasing strength of specimen A after the 28 day curing period.

Ming-hua et al. (2008) stated that under seawater conditions, the durability of concrete materials were to be one of the major factors to regard in designing structures. Further, the decrease of durability in structures was reported to be enhanced, by the progressive development of the micro-damage, via the erosion of chlorine and sulphate ions. The consequence was determined via the reduced properties of, namely the modulus, strength, and toughness of the material [31].

Cracks were determined to develop topo-chemically with bladed-fibrous radial ettringite clusters aggregating with C–S–H structures in the specimen A matrix and pore spaces, being most likely responsible for the low resistance to sulphate solutions (Figs. 12 and 13) [32,33]. Cracks may have developed elongated along crack boundaries and were also observed to develop with rare patches of fibrous C–S–H structures, where the original C–S–H structure network is coated and masked by the matrix surface (Fig. 14). C–S–H structures are also present along the cracks, i.e., elongated pores. Well developed fibrous ettringite grains (Fig. 15) and clewing hair-like aggregates were determined to form in pores (intra-pore formation) (Figs. 16–18) of the specimen indicating the heterogeneity in the progress of the hydration process and the possible increase of the strength in the C2 specimen.

5. Conclusions

From this study we can conclude that:

1. Although the probable increasing compressive strength and sulphate resistance in the concrete (specimen C2) may be attributed to the development of the intra-pore gypsum roses and ettringite during hydration, there seems to be need for further studies concerning the development of the process of hydration during curing.
2. Deterioration, after 36 months of exposure, manifested in the form of disintegration and cracking was more obvious in the control cement mortar specimens.
3. The incorporation of red brick dust and/or basaltic pumice improved the sulphate resistance of cement mortars. Actually, an appropriate amount of additives, providing high sulphate resistance, seem to be around 15% for each additive.
4. SEM examination confirms that ettringite is not a pronounced feature in the microstructure of the blended cement mortars exposed to sodium sulphate solution. In comparison to that of the control cement mortars, ettringite has formed in pores of specimen A revealing the probable effects on the compressive strength, with a significant increase in the first 28 days. Portlandite forming in the pores of specimen A may also have an effect on increasing the strength as well for the same treatment period. However, the ettringite forming in matrix and pores as bladed-fibrous radial cluster aggregates may be responsible for the decrease of strength.
5. The additives used in the present study increased the strength of cement and provided a decrease in the production cost and an improvement in the quality of the cement. Furthermore, the basaltic pumice is more easily ground than Portland cement clinker, which is a further advantage.

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