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Effects of pozzolanic additives on the properties of portland cement containing clay waste

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Abstract

This paper presents the effects of pozzolanic additives on the properties of Portland cement containing clay waste. Twelve different types of cement mortars were produced by adding fly ash and natural pozzolan (in the range of 5 % - 20 %) to ordinary CEM I 42.5 Portland cement containing 10 % and 15 % of clay waste. The cement mortars were subjected to compression and flexural tests. The durability of mortars were also evaluated by the measurements of the weight losses of the samples exposed to sodium sulfate or magnesium sulfate solutions. The scanning electron microscopy (SEM) analysis of the samples exposed to the sulfate solutions was also performed. The results showed that the clay waste could be used as cement additive up 15 % with the use of 20- 30 % pozzolanic material. Fly ash and natural pozzolan addition improved the resistance of mortars to sulfate attack.

Keywords: Cement; Clay waste; Pozzolanic additives; Durability; Sulfate attack

1. Introduction

In the Borax Plants, millions tons of waste such as clay waste and sludge were formed every year during the benefication of borax. These wastes are not utilized and they usually are accumulated in solid or liquid waste areas in the plants [1]. The wastes are very huge problems for industrial countries. If chemical wastes, which deteriorate ecologic balance because of the boron content and are threat to the health of human and environment, do not deposited adequately, they cause some environmental problems. On the other hand, these depositions have a cost unless the wastes are utilized in any way.

Cement is a hydraulic binder which is produced by grinding gypsum which is added in 3-5 % to clinker. Clinker principally comprises of tricalcium silicate ($3CaO.SiO_2$ (C_3S)), dicalcium silicate ($2CaO.SiO_2$ (C_2S)), tricalcium aluminate ($3CaO.Al_2O_3$ (C_3A)) and tetracalcium aluminoferrite ($4CaO.Al_2O_3.Fe_2O_3$ (C_4AF)). Also, it contains very little compounds such as MgO, K_2O, Na_2O, TiO_2 and Mn_2O_3. Cement industry consumes too much energy. The cement plants also generate the huge amount emissions of dust and CO_2 during the process. The reduction of energy cost and emissions can be supplied by utilization of clay waste as cement additive [2, 3]. Thus, the recycling of this clay waste will contribute to the reduction of the amount of waste deposition and the environmental sustainability.

In our previous study, the utilization of clay waste containing boron as cement additives was examined and the effect of clay waste on the physical and mechanical properties of cement was investigated. The results showed that the clay waste could be used as cement additive up to 5 and 10 % [4]. The utilization ratio of clay waste in cement can be increased by adding pozzolans to cement. The C_3S and C_2S react with water in cement to form a calcium silicate hydrate (C-S-H) by releasing lime as $Ca(OH)_2$ (CH). These are the two principal solid phases in the hydrated cement paste. The C-S-H occupies 50 to 60 percent of the volume of solids in completely hydrated Portland cement paste. The CH constitutes 20 to 25 percent of the solid volume in the hydrated paste. The strength-contributing potential of calcium hydroxide is limited in compared with C-S-H due to a considerably lower surface area. When pozzolanic materials are added to cement, they react with CH in the presence of moisture to form secondary calcium silicate hydrate [2, 5]. Therefore, in this study deals the effects of pozzolanic additives on the properties of Portland cement containing clay waste to increase the utilization ratio of clay waste in cement by pozzolans like fly ash and natural pozzolan which are more active from pozzolonic point of view. Mortar samples containing 10 % and 15 % clay waste with pozzolans were prepared to test for determining the compressive strength. Pozzolans are used to improve the mechanical strength as well as durability [6-9]. The durabilities of these mortar samples against the sulfate attack were investigated by measuring their mass loss in sodium sulfate solution and magnesium sulfate solution. The deteriorations due to the sulfate effects are also determined using SEM.

2. Materials and Method

2.1. Cement components

In this study, ordinary CEM I 42.5 Portland cement (OPC) meeting the requirements of TS EN 197-1 [10] was used and it was provided from SET Cement Plant (Ankara, Turkey). The additives used in this study were clay waste (CW), fly ash (FA) and natural pozzolan (NP). CW was provided from Etibank Kırka Borax Plant. The X-ray diffractogram (XRD) of the CW was obtained using a Rigaku Rint 2200 diffractomer with Cu K α radiation. The XRD of the CW indicated that it consisted of montmorillonite ($(Na, Ca)_{0.33}$ $(Al, Mg)_2$ (Si_4O_{10}) $((OH)_2H_2O)$, dolomite ($CaMg(CO_3)_2$) and tincal ($Na_2B_4O_7.10H_2O$). The fly ash (FA) was obtained from Kütahya-Tunçbilek Power-Plant, and natural pozzolan was from The Central Anatolia. The chemical analyses of cement components were carried out by X-ray fluorescense analyzer (XRF Rigaku RIX 2080). The chemical and physical properties of the materials are given in Tables 1 and 2. The strength activity indexes of pozzolans used were analyzed according to ASTM C 311 [11] and ASTM C 618 [12] (Table 3). FA and NP conform to the requirement of ASTM C 618 (min. % 75).

2.2. Cement types

In our previous study, the experimental results showed that the clay waste could be used as cement additive up to 5 and 10 %. To increase the utilization ratio of clay waste in cement by pozzolans like fly ash and natural pozzolan, the mortar samples containing 10 % and 15 % clay waste with 10 %, 20 % and 30 % pozzolans were prepared. Twelve different types of cement mortars were used in the experimental process. These were produced by adding clay waste, fly ash and natural pozzolan to ordinary CEM I 42.5 Portland cement. The terminology, proportions and mineral compositions of the studied cements are given in Table 4.

2.2. Method

Mortar mixtures were prepared according to TS EN 196-1 [13] using the cements in Table 4 (w/c=0.5, cement: sand = 1:3). Two series of specimens were produced. From each mixture, (40 x 40 m x 160) mm prismatic mortar bars were cast. After casting and finishing, the molds were covered with plastic sheets and stored 3 days in a moist room with relative humidity above 90 % and temperature of 23 ±2 ^{0}C. After the initial curing period, the specimens were demoulded and cured in lime saturated water at 23 ±2 ^{0}C until the time of testing.

Compressive strength and flexural strength measurements were performed at ages of 7, 28 and 90 days on specimens of the first series according to TS EN 196-1. Each value is the mean of three measurements.

After the 28 days curing, the specimens of the second series were stored in a 5 % sodium sulfate (Na_2SO_4) solution or in a 5 % magnesium sulfate ($MgSO_4$) solution. The concentrations of the solutions were checked periodically, and in the case of decreasing the solutions were replaced with fresh ones. The effect of sulfate attack on the mortar bars was determined by the mass loss of the specimens. The mass losses of the specimens were measured at regular intervals up to 180 days. The presented results are the average mass loss of three mortar bars.

Scanning electron microscopy (SEM) was performed on some samples to determine the structural changes formed during sulfate attack. The microstructure of fractured surface of samples was examined by scanning electron microscopy (Jeol JSM 6080).

3. Results and Discussion

3.1. Compressive strength

The compressive strengths of the mortar bars at various ages are presented in Table 5. When CW percentage increased from 10 % to 15 %, the loss of strength was about 7 %. Thus, the strengths of the mortars containing 10 % CW had higher than those of the mortars containing 15 % CW at all ages. This is a result of replacing a part of cement by the equivalent amount of CW. The mortars with a high FA content (CW3, CW5, CW9, CW11) showed better performance when compared to those with a high NP content (CW4, CW6, CW10, CW12). The difference in the strengths of these increased with increasing pozzolan addition. This situation occurs because FA has a higher pozzolonic activity and a better physical property than NP (Table 3).

The compressive strength of the mortar increased with increasing pozzolan amount. At pozzolan amount higher than 20 % of the mortar, the compressive strength reduced gradually. A large difference was not observed in the compressive strength between the use of 10 % pozzolan and the use of 30 % pozzolan. So, considering the cost of cement, the use of 30 % pozzolan appears to be more suitable economically.

Specimen CW3 had the highest compressive strength at 90 days. The increase in the compressive strengths between 7 days and 28 days was about 30 %. On the other hand, this increase between 7 days and 90 days was about 60 %. This situation indicated that the pozzolanic reactions in the mortar containing CW were progressing due the use of pozzolanic additive. Hence, the strength of mortar samples should be determined according to the strengths at 90 days instead of those at 28 days.

The compressive strengths of the mortars with pozzolanic additive equated to that of the OPC at 28 days (46.3MPa) except for a few sample. However, their strengths increased after 28 days and were higher than that of OPC.

3.2. Flexural strength

Table 5 shows the flexural strengths of the mortar bars at various ages. The results of flexural strength tests were in agreement with those of compressive strength tests. The flexural strength decreased by increasing CW ratio. This strength increased by increasing total pozzolan ratio. The flexural strength had a maximum value with the additions of FA and NP in the ratio of 20 %. After this ratio, it decreased gradually. All these results can be explained in a way similar to the compressive strength results of the mortar samples as reported above.

3.3. Mass Loss

Durability experiments were performed with Na_2SO_4 and $MgSO_4$ solutions in order to test the durabilities of the samples prepared by using CW, FA and NP and cured for 28 days. The weight losses of the samples were measured after they had been put into these solutions for 60, 90, 120, 150 and 180 days. The mass loss test results of the mortar bars subjected to a 5 % Na_2SO_4 and 5 % $MgSO_4$ solutions for different days are given in Tables 6 and 7. As known, Na_2SO_4 and $MgSO_4$ compounds are salts having an expansive effect on concrete, mortar and cement. The both salts used in the experiments react with lime ($Ca(OH)_2$) in cement to form $CaSO_4$ releasing Na^+, Mg^{2+} and OH^-, respectively, by the following reactions:

$$Na_2SO_4 + Ca(OH)_2 + 2H_2O \longrightarrow CaSO_4.2H_2O + 2NaOH \quad (1)$$

$$MgSO_4 + Ca(OH)_2 + 2H_2O \longrightarrow CaSO_4.2H_2O + Mg(OH)_2 \quad (2)$$

Subsequently, $CaSO_4$ reacts with tricalcium aluminate (C_3A) in the cement to form tricalcium sulfoaluminate hydrate (ettringite) (Candlot's salt: $3CaO.Al_2O_3.3CaSO_4.32H_2O$), causing crystal growth, as follows:

$$3(CaSO_4.2H_2O) + 3CaO.Al_2O_3 + 26H_2O \longrightarrow 3CaO.Al_2O_3.3CaSO_4.32H_2O \quad (3)$$

Therefore, a great volume increase appears during the formation of salt and the concrete may be cracked due to the internal tension occurring in the course of this volume expansion [5, 14-16].

The mass loss increased with the period of exposure in all specimens. The values measured after 180 days are about four times those after 60 days (Tables 6 and 7). It was also observed that they deteriorated and developed significant cracks after 180 days exposure to sulfate attack.

The mass losses in the specimens after 180 days of exposure to a 5 % Na_2SO_4 and 5 % $MgSO_4$ solutions are given in Figs. 1 and 2. For both sulfate solutions, when CW percentage increased from 10 % to 15 %, an increase in the mass loss was about 30-35 %. This showed that CW had a negative effect on the durability of mortar. This may result from the fact that the CW has no a pozzolonic activity, and thus $Ca(OH)_2$ which is necessary for the formation of $CaSO_4.2H_2O$ (gypsum) is not reduced by pozzolonic reaction. Almost a linear decrease in mass loss with the amount of pozzolan was determined in all specimens. In the case of using 30 % pozzolan, the reduction in the mass loss was maximum. Finally, the use of pozzolanic additives increased the sulfate resistance of mortar specimens containing clay waste. Fly ash and natural pozzolan improved the resistance of mortars to sulfate attack due to the reduction in C_3A content (Table 4). Also, their pozzolanic reactions reduced CH amount which was necessary for the formation of gypsum.

As seen in Figs. 1 and 2, $MgSO_4$ has a large effect on the durability of mortar in comparison with Na_2SO_4. For all specimens, the deterioration was greater in $MgSO_4$ than in Na_2SO_4. The mass losses in the specimens after 180 days of exposure to $MgSO_4$ solution are 30 % higher than those after 180 days of exposure to Na_2SO_4 solution. In other words, the durability performance of the mortars in the Na_2SO_4 solution was better. The cementitious hydration product C-S-H is stable since the formation of sodium hydroxide, which is a by-product of the reaction, ensures the continuation of high alkalinity in the system. In the case of magnesium sulfate attack, the formed magnesium hydroxide is insoluble and reduces the alkalinity of the system. In the absence of hydroxide ions in the solution, C-S-H is no longer stable and is also attacked by the sulfate solution. So, the magnesium sulfate attack is more serious on the mortar [5].

The differences in the mass losses were observed according to the use of FA and NP. While the specimens with NP showed better performance for Na_2SO_4 attack, the specimens with FA showed better performance for $MgSO_4$ attack (Figs. 1 and 2).

3.4. SEM Analysis

Scanning electron microscopy (SEM) was performed on some samples to identify the products formed during sulfate attack. SEM micrographs of the samples subjected to 5 % Na_2SO_4 attack for 180 days are given in Figs. 3 and 4 SEM micrograph of the sample CW1 revealed the formation of ettringite surrounding the mass of C-S-H, and a lot of cracks in structure (Fig. 3). In the SEM micrograph of CW5, the lesser amount of ettringite with rod-like crystals was observed. The formation of ball-like ettringite also appeared in the pores. It can be thought that due to low CaO content [6], large minerals do not form in this cement's mortar. Thus, there were not dense cracks, and were fewer and shorter cracks (Fig. 4). These observations are in good accordance with the results of mass loss.

The SEM micrographs of the samples subjected to 5 % $MgSO_4$ attack for 180 days are given in Figs. 5 and 6. The SEM micrographs of the sample CW1 showed the formation of massive ettringite covering over the C-S-H structure, and a number cracks (Fig. 5). In Fig. 6, a ball-like ettringite and an ettringite with pin-like crystals were observed. These results indicate that $MgSO_4$ has a different sulfate effect according to Na_2SO_4. The crack morphology of the sample subjected to the $MgSO_4$ is different from that of the sample subjected to Na_2SO_4 (Figs. 3 and 4).

4. Conclusions

In this paper, the effects of pozzolanic additives on the properties of Portland cement containing clay waste were investigated. The major conclusions derived from the present work are as follows:

1. When clay waste percentage increased from 10% to 15%, the strength was decreased. The compressive strength and flexural strength of the mortar increased with increasing pozzolan amount. At pozzolan amount higher than 20% of the mortar, the compressive strength reduced

gradually. However, the use of 30% pozzolan appears to be more suitable economically. The use of clay waste at a higher ratio is possible with fly ash and pozzolan.

2. The mass loss increased with increasing from 10 % clay waste to 15% clay waste, and thus clay waste showed negative performance against sulfate attack.

3. The mass loss decreased with increasing pozzolanic additives proportion. Fly ash and natural pozzolan improved the resistance of mortars to sulfate attack due to the reductions in C_3A and CH contents.

4. There was no relation between sulfate resistance and pozzolan type. However, the performance of natural pozzolan was observed to be effective for Na_2SO_4 attack, and also the performance of fly ash was observed to be effective for $MgSO_4$ attack. The magnesium sulfate attack is more serious on the mortar.

5. It is suggested that the clay waste can be used as cement additive up 15 % with the use of 20-30 % pozzolanic material.

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Chemical composition	Weight (%, w/w)	Physical and mechanical properties	
	61.17	Specific gravity (g/cm^3)	3.13
CaO	1.15	Blaine (cm^2/g)	3075
MgO	23.24	Compressive Strength (MPa)	
SiO_2	3.96	2 days	23.3
Al_2O_3	4.16	7 days	37.6
Fe_2O_3	0.07	28 days	46.3
Na_2O	0.49	Flexural Strength (MPa)	
K_2O	2.66	2 days	1.80
SO_3	0.010	7 days	3.4
Cl^-		28 days	5.7
Ignition loss	1.19	Initial setting time (hrs.min)	2. 45
Insoluble residue		Final setting time (hrs.min)	3. 55
	0.27	Volume expansion (mm)	1.5

Table 1. Chemical and physical properties of ordinary CEM I 42.5 Portland cement

Chemical composition (%, w/w)	CW	FA	NP
SiO_2	15.47	56.24	58.12
Al_2O_3	2.55	16.06	19.05
Fe_2O_3	1.74	15.16	9.11
CaO	22.86	2.32	5.39
MgO	18.26	4.85	3.75
Na_2O	5.13	0.35	0.24

K_2O	1.33	1.49	1.72
SO_3	-	1.01	0.84
Cl^-	-	0.01	0.016
Ignition loss	23.88	1.84	1.4
Insoluble residue	-	0.27	0.27
Specific gravity (g/cm^3)	2.12	2.23	2.24
Blaine (cm^2/g)	2830	3975	3850
Residue on 90 µm (%)	0.4	3.7	3.8
Residue on 200 µm (%)	0.9	0.3	0.2
Strength activity index			
7-d (%) for -90 µm	-	89	83
28-d (%) for -90µm		100	91

Table 2. Chemical and physical properties of clay waste and pozzolanic additives

Sample	7 days activity (%)			28 days activity (%)			90 days activity (%)		
	Original	Pass 200µm	Pass 90µm	Original	Pass 200µm	Pass 90µm	Original	Pass 200µm	Pass 90µm
FA	80	85	89	83	96	100	101	111	115
NA	74	81	83	79	87	91	87	93	100

Table 3. Activity index of pozzolans

Cement type	Proportion (%)				Mineral composition (%)			
	OPC	CW	FA	NP	C_3S	C_2S	C_3A	C_4AF
CW_1	90	10	0.0	0.0	41.4	11.3	9.1	10.2
CW_2	80	10	5.0	5.0	39.3	9.2	7.6	8.1
CW_3	70	10	15	5.0	37.4	9.0	7.1	7.7
CW_4	70	10	5.0	15	37.4	9.0	7.1	7.7
CW_5	60	10	20	10	36.1	8.6	6.5	7.3
CW_6	60	10	10	20	36.1	8.6	6.5	7.3
CW_7	85	15	5.0	5.0	40.4	10.8	8.7	9.9
CW_8	75	15	5.0	15	38.9	8.8	7.4	7.7
CW_9	65	15	15	5.0	37.0	8.4	6.7	7.2
CW_{10}	65	15	5.0	15	37.0	8.4	6.7	7.2
CW_{11}	55	15	20	10	35.6	8.1	5.9	6.6
CW_{12}	55	15	10	20	35.6	8.1	5.9	6.6

Table 4. The terminology, proportions and mineral compositions

Cement type	Total pozzolan (%) (FA+NP)	Compressive strength (MPa)			Flexural strength (MPa)		
		7 days	28 days	90 days	7 days	28 days	90 days
CW_1	0.0	30	35	42	3.2	5.1	5.5
CW_2	10	34	47	54	3.3	5.4	5.8
CW_3	20	38	49	60	3.5	5.6	6.0
CW_4	20	37	48	58	3.4	5.3	5.9
CW_5	30	37	46	53	3.4	5.1	5.6

CW_6	30	34	43	51	3.3	5.0	5.4
CW_7	0.0	28	33	37	3.0	4.9	5.3
CW_8	10	29	44	50	3.2	5.2	5.6
CW_9	20	36	47	54	3.3	5.4	5.8
CW_{10}	20	34	46	53	3.2	5.3	5.7
CW_{11}	30	35	44	50	3.0	5.0	5.5
CW_{12}	30	33	41	47	2.8	4.8	5.1

Table 5. The results of compressive and flexural strength tests for mortar mixtures

Cement type	Total pozzolan (%) (FA+NP)	Mass loss (%)				
		60 days	90 days	120 days	150 days	180 days
CW_1	0.0	0.40	0.52	0.72	1.01	1.52
CW_2	10	0.35	0.45	0.61	0.85	1.28
CW_3	20	0.27	0.35	0.50	0.70	1.12
CW_4	20	0.28	0.37	0.52	0.69	1.05
CW_5	30	0.21	0.27	0.39	0.55	0.92
CW_6	30	0.20	0.28	0.38	0.56	0.84
CW_7	0.0	0.54	0.70	0.96	1.34	2.01
CW_8	10	0.43	0.56	0.78	1.10	1.65
CW_9	20	0.32	0.41	0.57	0.81	1.41
CW_{10}	20	0.35	0.45	0.63	0.88	1.34
CW_{11}	30	0.28	0.36	0.52	0.73	1.10
CW_{12}	30	0.29	0.38	0.50	0.75	1.13

Table 6. The results of Na_2SO_4 durability test for mortar mixtures

Cement type	Total pozzolan (%) (FA+NP)	Mass loss (%)				
		60 days	90 days	120 days	150 days	180 days
CW_1	0.0	0.37	0.44	0.63	1.38	2.60
CW_2	10	0.31	0.38	0.53	0.87	1.55
CW_3	20	0.25	0.30	0.42	0.71	1.22
CW_4	20	0.26	0.31	0.44	0.72	1.29
CW_5	30	0.20	0.24	0.34	0.59	1.06
CW_6	30	0.18	0.22	0.31	0.56	1.10
CW_7	0.0	0.52	0.62	0.87	1.72	3.45
CW_8	10	0.45	0.54	0.76	1.23	2.22
CW_9	20	0.33	0.41	0.58	0.93	1.70
CW_{10}	20	0.32	0.41	0.60	0.96	1.71
CW_{11}	30	0.26	0.32	0.45	0.72	1.38
CW_{12}	30	0.27	0.33	0.49	0.81	1.45

Table 7. The results of $MgS0_4$ durability test for mortar mixtures

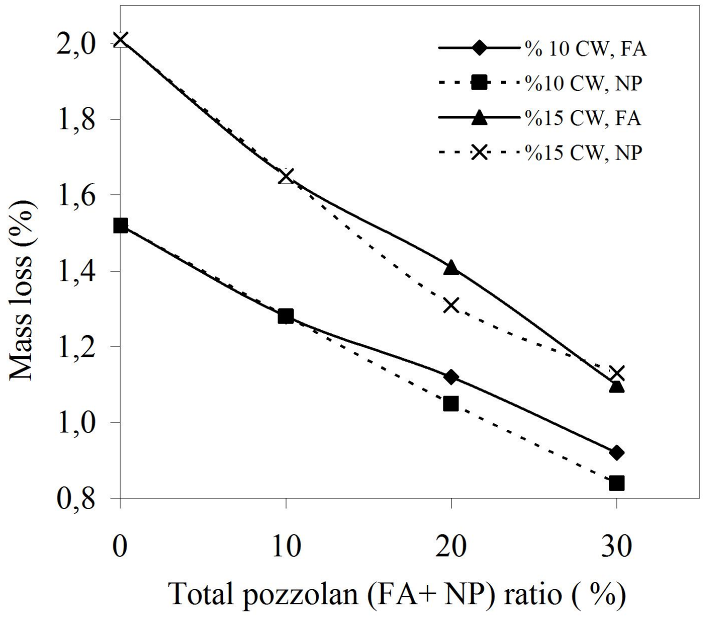

Fig. 1. Mass losses in the cement mortars after 180 days of exposure to 5 % Na_2SO_4 solution

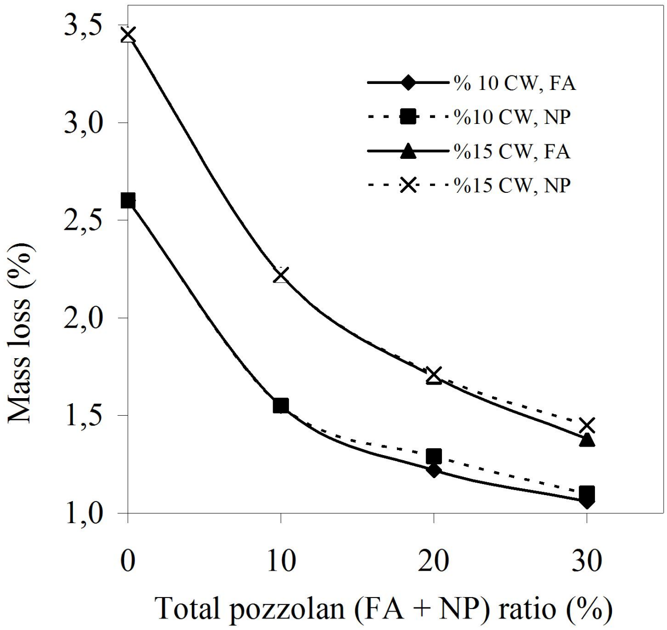

Fig. 2. Mass losses in the cement mortars after 180 days of exposure to 5 % $MgSO_4$ solution

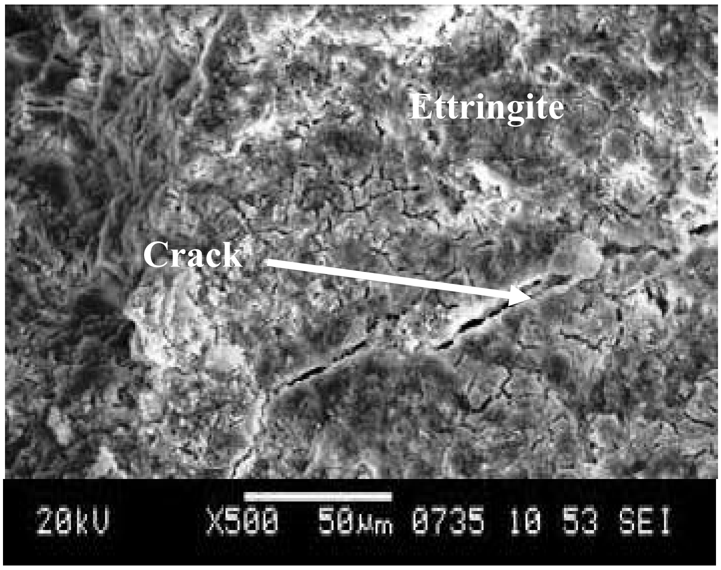

Fig. 3. SEM micrograph of CW1 subjected to 5 % Na_2SO_4 attack for 180 days

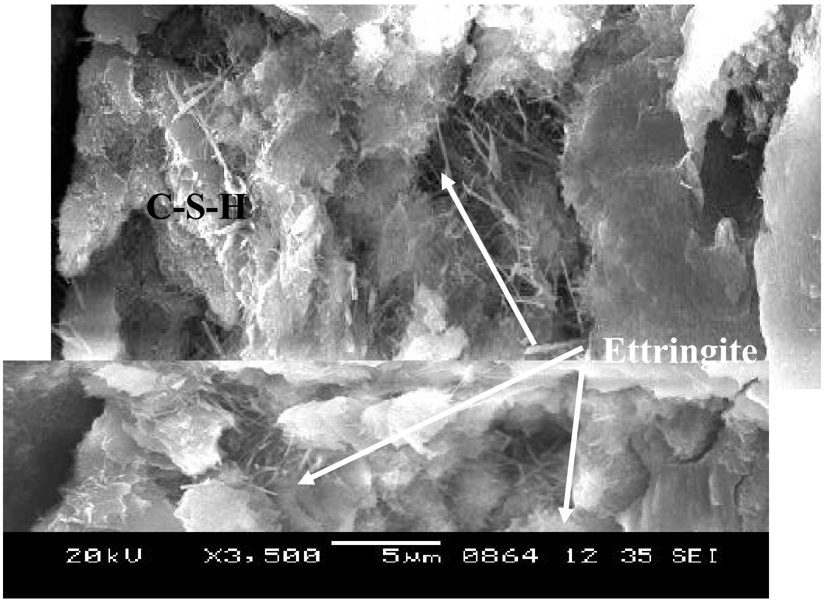

Fig. 4. SEM micrograph of CW5 subjected to 5 % Na_2SO_4 attack for 180 days

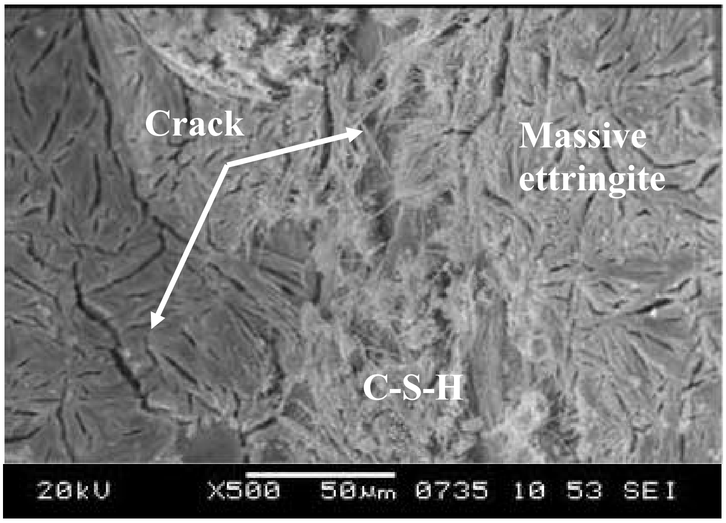

Fig. 5. SEM micrograph of CW1 subjected to 5 % $MgSO_4$ attack for 180 days

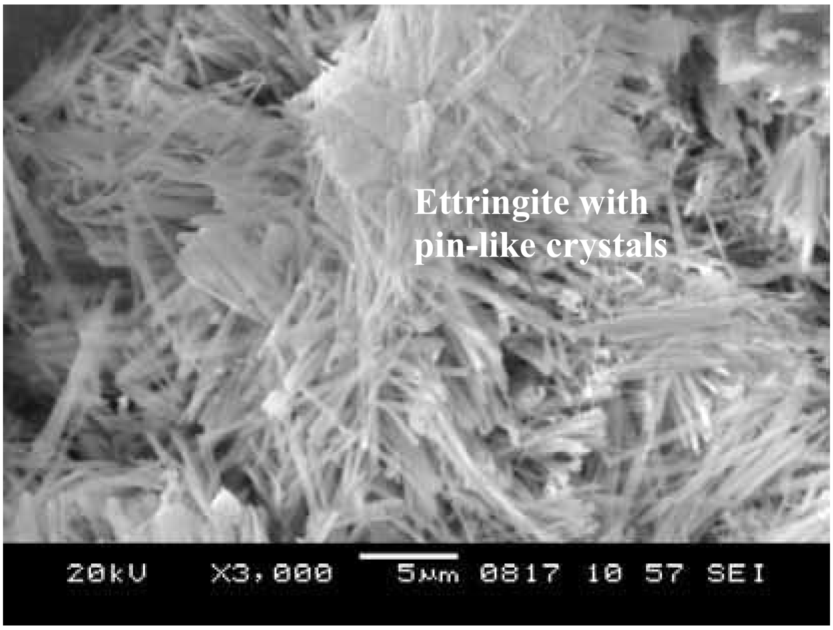

Fig. 6. SEM micrograph of CW5 subjected to 5 % $MgSO_4$ attack for 180 days