

Performance of mortars incorporating fly ash, silica fume, blast furnace slag at different temperature in magnesium sulfate solution

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ABSTRACT This study presents the performance of five different mortars at room temperature, 40°C and 5°C in magnesium sulfate solution. Two groups of mortars are prepared of only cement but vary in ratio water/ cement (W/C) (0.52 and 0.625). The other three groups of mortars produced incorporating fly ash (FA), silica fume (SF) and blast furnace slag (BFS). These mortars were immersed for 600 days in magnesium sulfate solution. All mortars measured compressive strength, flexural strength during the 600 days. Water absorption and sorption of mortars were determined at 28 days. Microstructures of mortars were studied by scanning electron microscope (SEM) and microscope. Chemical analyzes were done with X-ray diffraction (XRF). Mortars sulfate attack affected mineral additives but cement prevent the mortars better than these with mineral additives. Mortars water absorption and sorption are important parameters on sulfate attack. Mortars corrupt at room temperature more than the other mediums.

Keywords: Magnesium sulfate; Brucite; Ettringite; Absorption; Sorption; Mineral additives; Silica fume; White materials.

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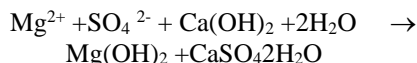
1. INTRODUCTION

Sulfate attack is the term used to describe a series of chemical reaction between sulfate ions and the components of hardened concrete, principally the cement paste, caused by exposure of concrete to sulfates and moisture [1]. The sulfate attack can be divided into two groups of sulfate, 1-internal and 2-external sulfate attack. Internal sulfate attack refers to a situation where the source of sulfate is internal to concrete. The source of sulfate can be the cement, supplementary materials such as fly ash or slag, the aggregate, the chemical admixtures, or the water. Two examples of such internal sulfate attack by an excess of cement

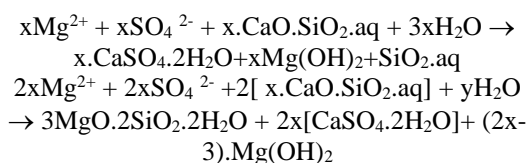
sulfate and the so-called delayed ettringite formation (DEF). External sulfate attack is caused by a source external to concrete. Such sources include sulfates from ground water, soil, solid industrial waste, and fertilizers, or from atmospheric sulfate (SO₃), or from liquid industrial wastes [1]. These two types of sulfate attack convert hardness concrete component into the other products, such as gypsum (CaSO₄·2H₂O), ettringite (6CaO·Al₂O₃·SO₃·32H₂O) and thaumasite (CaSO₄·CaSiO₃·15H₂O). These three kind of products are the main formation besides other products which occur to produce a kind of sulfate ion, such as brucite (Mg(OH)₂), magnesium silicate (MgO·SiO₂·H₂O) and hydrous silicate.

New product formation in hardness concrete consists of expansion, cracking and spalling. Sulfate attack decreases the durability of concrete, hereby long-term durability of structures is reduced and at the same time increases the cost of caring for structures, so the durability problems of concrete has drawn the attention of researchers.

The proposed mechanism of magnesium sulfate attack, sulfate ions firstly attack calcium hydrates and rapidly form brucite (Mg(OH)₂) and gypsum. Areas of concrete are touched by a solution of sulfate ions occur brucite layer and that behind gypsum layer [2].



When penetrated sulfate ions attack calcium silicate (C-S-H), the main corruptive product in hardness concrete begins to dangerously corrupt and C-S-H change magnesium silicate (M-S-H). hardness concrete transforms into a mud like substance.



The sulfate attack corrupts concrete material and decreases the service life. Researchers are looking at how to protect concrete from sulfate ions with mineral additives such as fly ash, silica fume and granule blast furnace slag [3-9]. The laboratory studies show some different results. Separate sulfate ions, mineral additive, water cement rate, temperature, ions concentration produce an unlikely situation. Skalny, Amoudi, Thomas, Akoz have reported on the resistance imparted by mineral additives increasing the sulfate resistance of concrete [1,4,8,10,11]. The other researchers, Lee said that silica fume had decreased the resistance of concrete against sulfate, the larger amount SF content the greater was the strength loss exhibited [9]. Some researcher represented that mineral additive change CH to C-S-H for magnesium sulfate direct influence C-S-H formation, mineral additive decreases sulfate resistant of mortars [14]. Cavdar has reported an increase in the resistant of hardness concrete mineral additives must be restricted to approximately 25-30% by mass to erosion [12].

The quality of concrete, specifically a low permeability, is the best protection against sulfate

attack. Adequate concrete thickness, high cement contents, low water/cement ratio and proper compaction and curing of fresh concrete are among the important factors that contribute to low permeability [1]. The most important role is permeability of hardness concrete and mortars in a sulfate environment [1,7,13,17]. Permeable concrete is vulnerable to attack by almost all classes of aggressive agents [15].

At medium temperature, have shown an occurrences of sulfate attack on concrete which is progressive. If medium temperature is high, thaumasite can occur greatly [16]. The formation of favored at low temperatures and a temperature of around 5°C is most favorable. Nevertheless, a formation of this phase at temperatures up to about 25°C is possible. The rate of thaumasite formation drops off markedly at somewhere between 15°C and 20°C [16].

The researchers have tried to develop various approaches to estimate long-term durability of concrete structures subject to sulfate attack. The researchers improve some equations such as $DC = [0,11 S^{0,45}][0,143t^{0,33}][0,204.e^{0,145C_3A}]$. Where DC is the degree of sulfate-induce corrosion, S is sulfate concentration, t is immersion period (days), C₃A is the presentence of tricalcium aluminate. The others produced the equation connect to diffusion coefficient. $X_s = 1.86 \cdot 10^6 C_3A (\%) \cdot ([Mg] + [SO_4]) D_i \cdot t$ Where D_i is diffusion coefficient, X_s is the location of the visible degradation zone [1]. These kind equations were constantly improved by the researchers.

In this study, the varied medium temperature effect of different mineral additives, fly ash, silica fume, granule blast furnace slag on magnesium sulfate attack was investigated.

2. EXPERIMENTAL

2.1. Materials

2.1.1. Cement component

The material used in mortars specimens are provided from locally resourced Turkish sources supplies. The cement is ordinary Portland cement, CEM 42,5 R. Granule ballast furnace slag was obtain from Karabuk Iron-steel Factory. Silica fume and fly ash are from Elettroferrocrom in Antalya and Thermal Power Plant. Five types of mortars were produced. Cement was incorporated in fly ash, silica fume and blast furnace slag with ratio 20 % weight of cement. Table 1. demonstrates the chemical properties of cement and materials. Table 2. shows cements and additive materials mechanical and physical properties.

Table 1. Chemical properties of cement and additive materials.

Contents	Cement	Fly ash	Silica fume	Granule blast furnace slag
CaO	54.7	1.3	0.5	25.3
SiO ₂	24.9	57	95.3	43.3
Al ₂ O ₃	5.3	1.1	0.7	13.6
Fe ₂ O ₃	3	4.3	0.9	0.3
MgO	1.6	16.3	1.4	10.5
SO ₃	3.3	0.4	0.4	3.7
Na ₂ O	0.9	0.4	0.3	0.2
K ₂ O	1.1	2.9	0.8	1.1
Cl ⁻	0.04	0.05	0.1	0

Table 2. Cements and additive materials mechanical and physical properties.

	Cement	Fly ash	Silica fume	Granule blast furnace slag
Density(g/cm ³)		3.05	2.4	2.2
Fineness(cm ² /g)		3832	3520	150000
Loss on ignition (%)		3.5	1.67	3.34
Flexural strength (N/mm ²)	2 day	3.9		
	28 day	8.2		
Compressive strength (N/mm ²)	2 day	25.9		
	28 day	46.3		

2.1.2. Specimens preparation

The compositions of the mortars are shown Table3. All mixtures are used at the same aggregate, shown grading and physical properties in Table 4 and the ordinary Portland cement, CEM I 42,5 R. This cement is a mixture of three mineral additives, fly ash, silica fumes and granule blast furnace slag in some mortars. Mortar samples were prepared by Hobart mixer and mortar was placed on the impact test bench and were blocked in a mould to be 4*4*16 cm, 60 drops (twice). After 24 hours of curing at 20 °C by 90% relative humidity, the samples were removed from the molds and placed in standard curing condition in saturated lime water at 20±1°C. Five main different types of mortars

were prepare to mix with cement, fly ash, silica fume and granulated blast-furnace slag, using only cement mortars consisting of 480 g cement, and 250 g water (W/C=0,52), mortars, named PCM-1 were used in 400 g cement and 250g water, W/C ratio is 0,625. Four mortars, FAM, SDM, BFSM and PCM-2 were kept at the same level water/binder ratio, 0,52. In additive mortars, with fly ash, silica fumes and blast furnace slag was incorporated with a ratio 20% weight of cement. In total five sets of mortars were prepared. Table 3 is shown at PCM-1, without mineral additive and 400 g cement, FAM, fly ash additive, SDM, silica fumes additive and BFSM, granule blast furnace slag additive mortars and PCM-2, mortars in 480 g cement.

Table 3. Mixture proportion of mortar specimens (1 m³).

Materials	Mortar specimens code of series				
	PCM-1	FAM	SFM	BFSM	PCM-2
Aggregate (kg/m ³)	1543	1455	1448	1465	1474
Cement(kg/m ³)	400	400	400	400	480
Water(kg/m ³)	250	250	250	250	250
Fly ash(kg/m ³)	0	80	0	0	0
Silica fume(kg/m ³)	0	0	80	0	0
Blast furnace slag(kg/m ³)	0	0	0	80	0

Table 4. Grading and physical properties of aggregate.

Aggregate type	Percentage passing						Density (g/cm ³)	Water absorption (%)
	Sieve size (mm)							
	8	4	2	1	0.5	0.25		
Fine aggregate	100	97	70	44	22	10	2.62	2.3

2.1.3. Specimens curing

After 24 hours, the specimens were removed from the molds and marked for later identification. All mortars were cured in lime saturated water 20±1°C on one mount. Three specimens from all groups were measured for flexural and compressive strength for seven days.

After one month, three mortars of groups were calculated for flexural and compressive strength, water absorption and sorption coefficient. The remaining specimens were divided into three groups and placed in a magnesium sulfate solution at room temperature, at 40°C and at 5°C for a period of 600 days.

Table 5. Specimens flexural and compressive strength.

Days	Mortar specimens									
	PCM-1		FAM		SDM		BFSM		PCM-2	
	7 days	28 days	7 days	28 days	7 days	28 days	7 days	28 days	7 days	28 days
Flexural strength (N/mm ²)	4.9	6.3	4.9	6.7	5.9	7.1	5.9	7.4	5.9	7.1
Compressive strength (N/mm ²)	30.5	37.9	33.2	42	38.9	46.6	31.3	53.1	33	53.2

Table 6. Compressive strength of mortars in sulfated medium (MgSO₄), different temperature.

Medium	Date (mount)	Compressive strength (MPa)				
		PCM-1	FAM	SDM	BFSM	PCM-2
Room temperature	3	40.8	44.6	48.6	55.4	54.6
	6	36.4	44.2	47.8	55.6	54.1
	9	28.6	38.3	45.4	55.1	53.3
	12	18.8	32.4	42.3	53.2	51.8
	20	9.6	24.6	39.2	46.6	47.2
40°C	3	41.2	45.4	48.9	55.2	54.9
	6	38.3	45.1	48.8	55.4	54.6
	9	35.9	43.4	47.5	54.8	53.8
	12	31.8	38.6	44.2	49.8	52
	20	22.1	28.4	39.2	47.5	48.8
5 °C	3	40.2	44.3	48.4	55.1	54.2
	6	37.6	43.4	48.1	53.6	53.7
	9	31.5	39.6	44.4	46.7	49.6
	12	22.4	34.3	39.8	44.2	48.8
	20	15.4	26.5	34.8	40.1	47.8

2.1.4. Testing program

Specimens from all groups were tested on the 7th and 28th day flexural and compressive strength. After placed in three different environments of magnesium sulfate solution, three specimens from all series also were tested for flexural and compressive strength on 90th, 180th, 270th, 360th and 600th day. When mortars were taken from the curing pool, water of mortars were wiped and kept humid. Firstly, flexural strength was measured, after all pieces of mortars were tested by a mortars compressive test machine.

After one month, standard curing, specimens were measured for water absorption and sorption coefficient. For sorption, prisms of 40x40x160 mm were cast from each mixture, 28 days. Measurement of capillary sorption, three mortars specimens from each one group was stood in a oven at 70°C until a constant mass formed. The bottoms of the mortars were opened and 3 mm above of mortars were covered by paraffin

Table 7. Flexural strength of mortars in sulfated medium (MgSO₄), different temperature.

Medium	Date (mount)	Flexural strength (MPa)				
		PCM-1	FAM	SDM	BFSM	PCM-2
Room temperature	3	6.8	7.3	7.8	8.2	7.9
	6	6.1	7.2	7.8	8.1	7.9
	9	5.2	7	7.7	8.1	7.8
	12	3.4	6.5	7.4	7.8	7.7
	20	2.2	5.2	6.2	6.8	6.9
40°C	3	6.9	7.4	7.9	8.1	7.8
	6	6.6	7.4	7.8	8	7.7
	9	5.8	7.3	7.6	7.9	7.6
	12	4.9	6.9	7.2	7.6	7.5
	20	3.8	5.8	6.6	6.9	6.9
5 °C	3	6.8	7.2	7.7	8	7.8
	6	6.4	7	7.6	7.9	7.7
	9	5.1	6.9	7	7.3	7.1
	12	4.4	6.6	6.9	7.6	7
	20	3.6	5.6	6.5	7	6.9

The mortars were then placed in a pan. The water level in the pan was maintained at 3 mm above the base of the specimens throughout this experiment. All specimens were weighted at specific times such as 1, 2, 4, 6, 12 and 24 hours. The coefficient (k) was obtain by $(Q/A) = k \cdot \sqrt{t}$ where Q= the amount of water adsorbed in (cm³), A is the cross section of specimen that was in contact with water (cm²), t is time (s), sorption coefficient is cm/s^{1/2}.

When mortars specimens were placed in three different solution pools, white materials (brucite and gypsum) began to change at different rates. Standing for 60 days, three mortars, PCM-1 in the different medium were cut into small nine pieces. New formations were observed under the microscope, taking 10 measurements from each one and calculated average thickness. Used Nikon SMZ 1000 type microscope and taking photography. NIS-elements program calculated white materials on mortars surface.

After placing in a magnesium solution, changes in all mortars were observed and photographed, some formations were investigated by microscope. Standing for 600 days in a sulfate solution, mortars specimens

were investigated by Scanning Electronic Microcopy (SEM) and X-Ray Diffraction (XRD). 5x5x3 mm small pieces were cut from the mortars. The pieces were dried in an oven and covered with gold than after investigated with SEM.

At room temperature, of 40°C and at 5°C, the pH of standing pools was measured together with temperature and conductivity. A group of mortars were put into the standard pool and this pool's pH, temperature and conductivity were also measured.

2.1.5. Solutions sulfate concentration

During the experimental program, magnesium sulfate solution concentration was adjusted to be added MgSO₄·7H₂O, 4.5 % of water weight. This medium is renewed with each mount.

3. RESULTS

Table 5. shows the compressive and flexural strength at the 7th and 28th of standard curing. Value of compressive and flexural strength at room temperature, 40°C and 5°C in magnesium sulfate solution are reported in Table 6 and Table 7. Fig.1, Fig.2 and Fig.3 are drawn according to Table.5, Table.6 and Table.7.

Fig.1. indicates the change of compressive strength at room temperature in magnesium sulfate solution. The change of compressive strength were calculated by the compressive strength at the 28th days (R_{c28}) were eliminated from the compressive strength at the 90th, 180th, 270th, 360th and 600th days (R_{cx}), and divided by the compressive strength at the 28th days and multiply by 100 ($100 \times (R_{c28} - R_{cx}) / R_{c28}$). The same calculations were made regarding the changes of the compressive and flexural strength in Fig.2, Fig.3, Fig.4, Fig.5 and Fig.6.

In Fig.1, during the first 90 days there are steady but slow increases in the all mortars, about %8 ratio, hereby R_{c28} . The next 180 days, compressive strength decreases slowly but next following days, compressive strength decreases quickly. Ratio of changing compressive strength of PCM-1 is 75% during 600 days. The mortars occurred high level corruption in PCM-1. There are high ratio of W/C (0,625), water absorption (10,5%) and sorption coefficient ($21,8 \times 10^{-3}$) in PCM-1. PCM-1 also has the less weight of cement and compressive strength ($R_{c28}=37,9 \text{ N/mm}^2$). Fig.1. indicated that mortars, to be high W/C, water absorption and sorption, affected much more than the others in magnesium sulfate solution. The corruption of FAM is second, ratio of 48% according to fig.1. FAM has second high water absorption and sorption but FAM has same ratio of W/C, with BFSM, SDM and PCM-2. FAM, used fly ash mineral additive, fineness of fly ash is smaller than blast furnace slag and silica fume (Table.2). If fineness is low at the mineral additives, the mortars have much water absorption and sorption on the 28 days. This situation is demonstrated Fig.7 and Fig.8. Third corruption has

in BFSM, ratio of 12%. Fourth corruption has in SDM, ratio 10%. Value of changing compressive strength in SDM is very near the value in BFSM. The best performance is shown in PCM-2. There is no any mineral additive in PCM-2, but has maximum cement content, 480 kg/m³ and low water absorption (9,3%) and sorption ($17,2 \times 10^{-3} \text{ cm/s}^{1/2}$) (Table 8). When PCM-2 is compared PCM-1, low ratio of W/C, water absorption and sorption increase the resistant of mortars on sulfate attack. Fig.1, Fig.7 and Fig 8 exhibit that the higher cement content causes less water absorption and sorption at the same ratio of W/C and aggregate. The arrangement of changing compressive strength in fig.1. has a similar arrangement water absorption and sorption, Fig.7 and Fig.8. This evidence shows that water absorption and sorption in mortars is the best parameter against magnesium sulfate attack.

In Fig.2 and Fig.3 are shown a shape like Fig.1 but the ratios of changing compressive strength are different. While the ratio of changing compressive strength in PCM-1 at room temperature is 75%, it is 42% at 40°C and 60% at 5°C. The same situation is shown in the ratio of changing compressive strength at FAM, BFSM, SDM and PCM-2. The high corruption occurred at room temperature in magnesium solution after than at 5 °C. The best performance of mortars is at a high temperature at 40°C. This evidence was derived from occurrences between new materials formation and pH at different temperature medium. Fig.9. shows four types curing conditions of mortars, at room temperature, 40°C, 5°C in magnesium sulfate solution and standard curing pool, no sulfate, were detected on the first day, nearly equals values were at the same levels, more with values of pH at 40° (Table 9). Values of pH at 5°C in magnesium solution had increased during 30 days. Values of pH at 5° were higher than the others mediums. If pH is high, concrete and mortars decomposes less. Fig.10. white material on surface of mortars can be seen where a white color is easily identified under the microscope. Fig.11.. depicts how this thickness was measured by Nis-elements D, computer program. Results of the measurement, thickness of white materials at 40° in magnesium sulfate solution was higher and quicker than in the other mediums. This situation indicates warm water increased the reaction between cement materials and sulfate. The thickness of white materials was the thinnest at a low temperature of water. Thickness of white materials is given in Table 10 for each medium at 90 days. Fig.12. gives results of XRD. This analyze was done on white materials, taken from surface on mortars in magnesium sulfated solution. In this analyze many peaks relating to brucite and gypsum were found.

The ratio of compressive strength, pH of solution and speed of white materials formation were analyzed with an expectations high corruption at 40° in magnesium solution for low value of pH, but thickness and quick white

materials formation reduce permeability and starts corruption later. High pH prevented the mortars at 5°C, so the mortars deteriorated at room temperature in magnesium sulfate solution, firstly.

Fig.13, , Fig.14 and Fig.15 show SEM micrograph of PCM-1, at 600 days, at room temperature, 40°C and 5°C in magnesium sulfate solution. Fig.16. indicates the result of XRD.

Which mortars had the greatest damages from sulfate, new materials formation increased in Fig.13, Fig.14, and Fig.15. How sulfated solution entered into void and crack, SEM micrograph were showed. New material formation cause expansion and cracking in mortars, are shown at SEM analyze. Ettringite, thaumasite and gypsum were found by XRD.

Table 8. Mortar specimens sorptivity coefficient and water absorption at 28 days.

Specimens type	Water absorption (%)	Sorptivity coefficient (cm/s ^{1/2})
PCM-1	10.5	0.02361
FAM	10.3	0.021844
SDM	9.8	0.019322
BFSM	9.9	0.018959
PCM-2	9.3	0.017284

Table 9. Curing pool pH, temperature and mV values.

Time (day)	Room temperature with MgSO ₄			40°C with MgSO ₄			5°C MgSO ₄			Standard curing		
	pH	°C	mV	pH	°C	mV	pH	°C	mV	pH	°C	mV
1	7.32	20.4	0.01	7.22	39.3	0.8	7.36	17.7	-0.01	7.81	19.8	-110
9	9.16	22.7	101	8.27	40.1	-0.59	8.75	4.9	-0.88	9.02	20.4	-107
24	8.77	20.9	0.94	7.92	39.6	-0.36	10.25	4.5	-185	8.94	20.3	-103
30	8.73	18.1	-100	7.85	39.5	-0.47	10.33	2.4	-187	8.76	19.4	-100

Table 10. White materials thickness is on PCM-1 mortars at 90 days in magnesium sulfate solution.

Group name	White materials thickness (micron)		
	Room temperature	40 °C	5°C
PCM-1	78.54	136.87	44.72

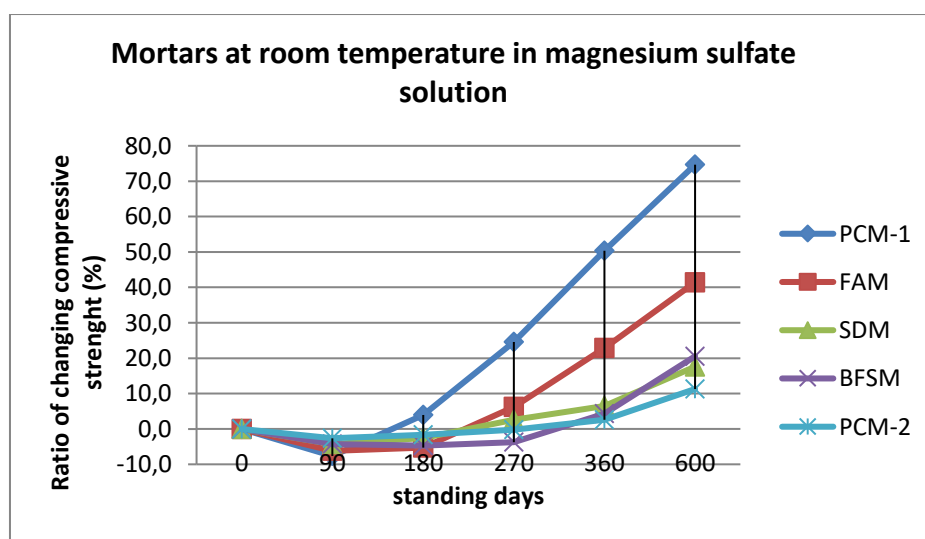


Fig.1. Mortars ratio of changing compressive strength at room temperature in magnesium solution

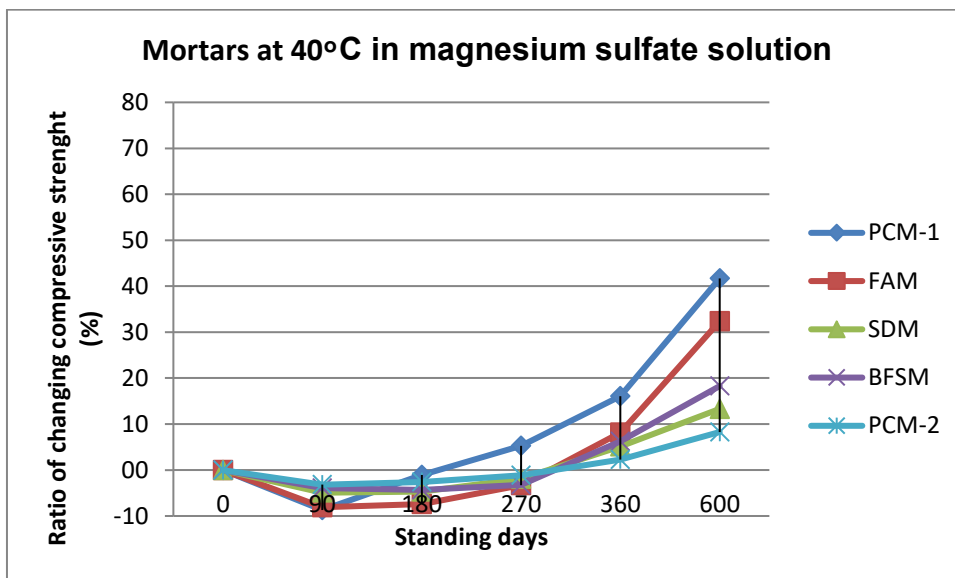


Fig.2. Mortars ratio of changing compressive strength at 40°C in magnesium solution

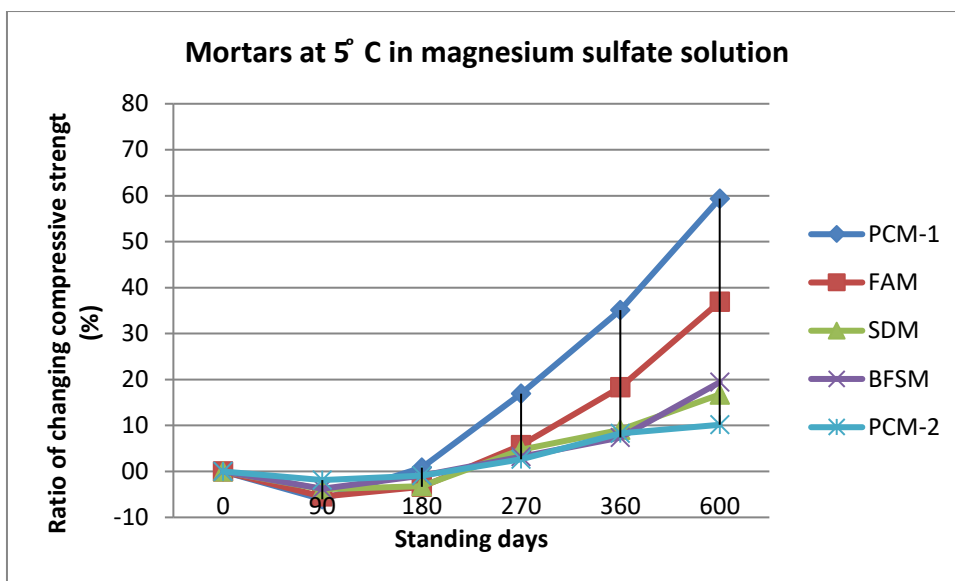


Fig.3. Mortars ratio of changing compressive strength at 5°C in magnesium solution

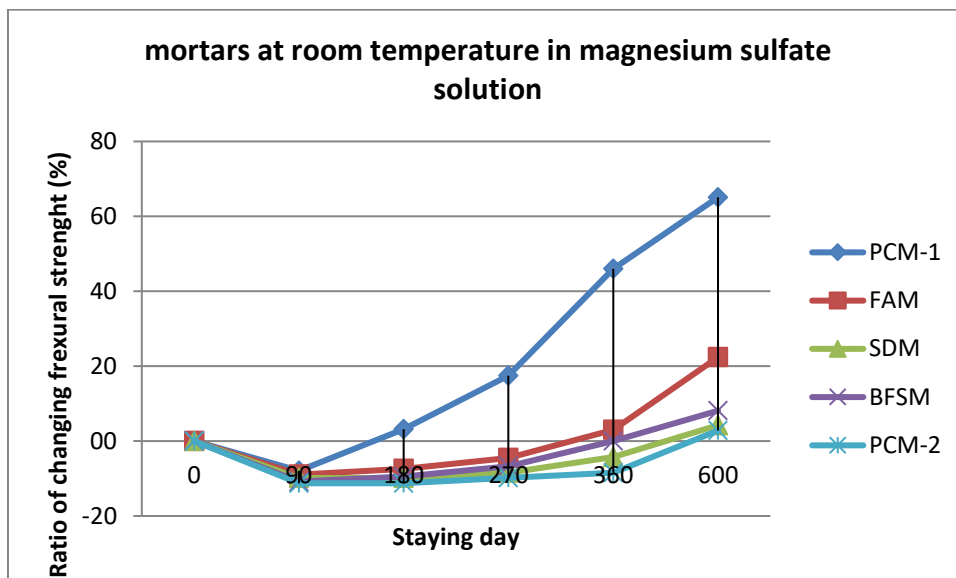


Fig.4. Mortars ratio of changing flexural strength at room temperature in magnesium solution

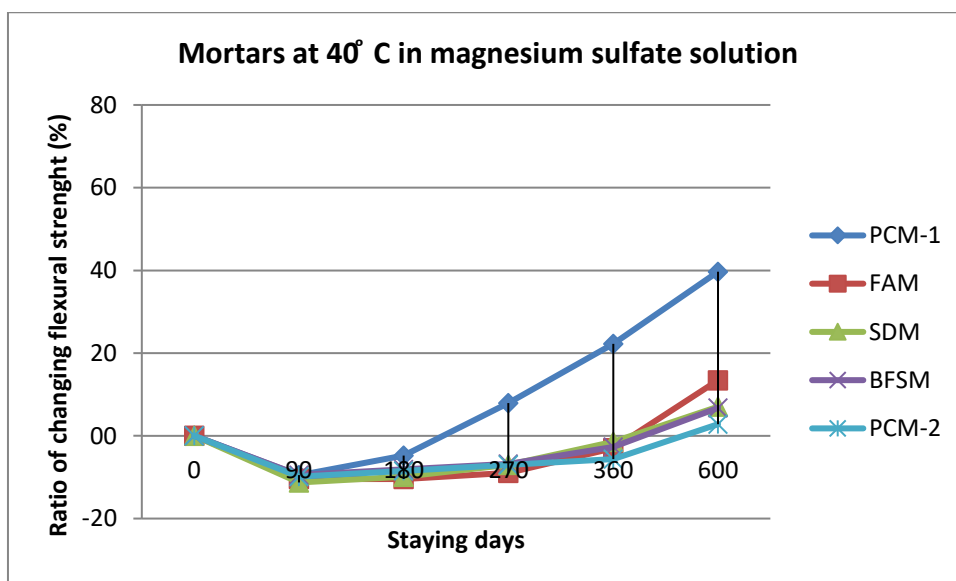


Fig.5. Mortars ratio of changing flexural strength at 40°C in magnesium solution

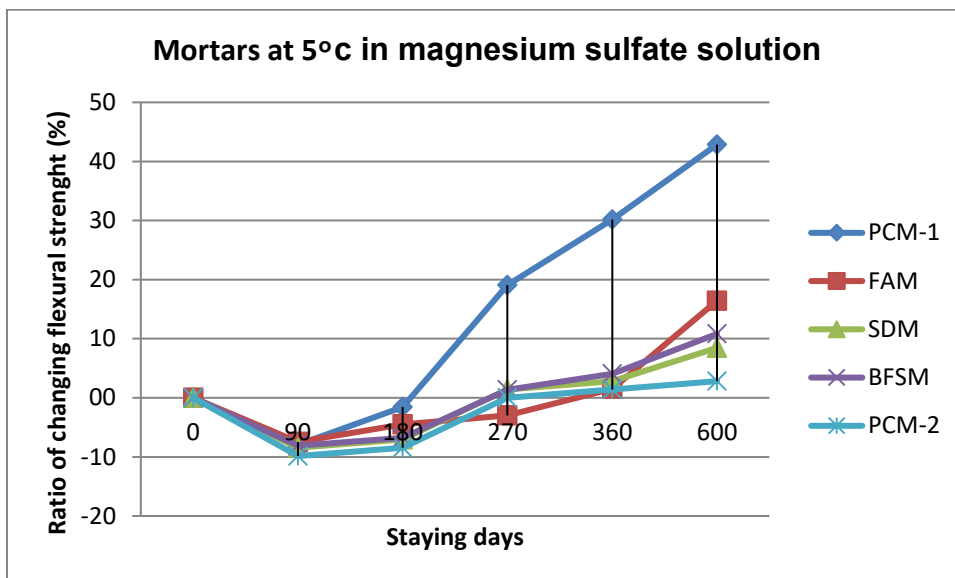


Fig.6. Mortars ratio of changing flexural strength at 5°C in magnesium solution

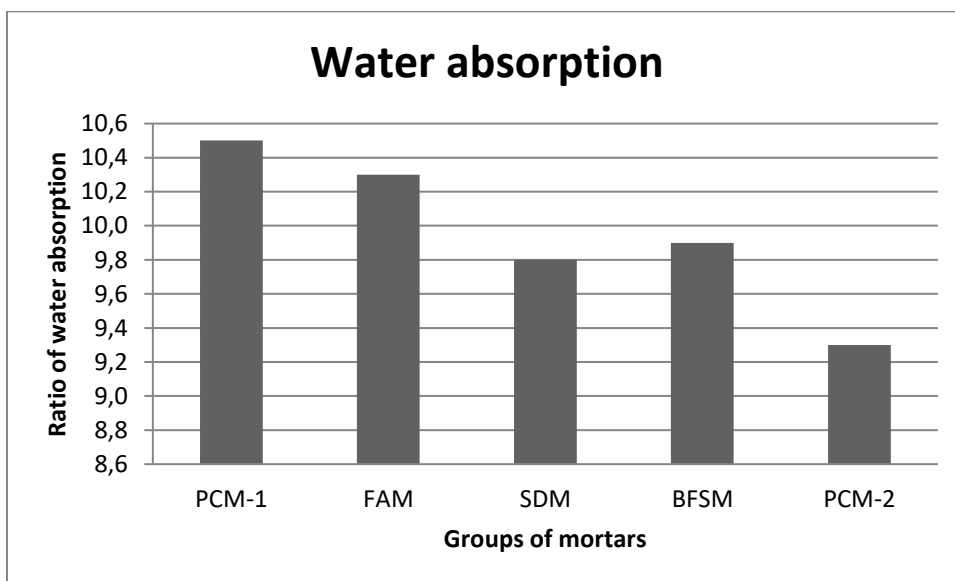


Fig.7. Group of mortars water absorption levels

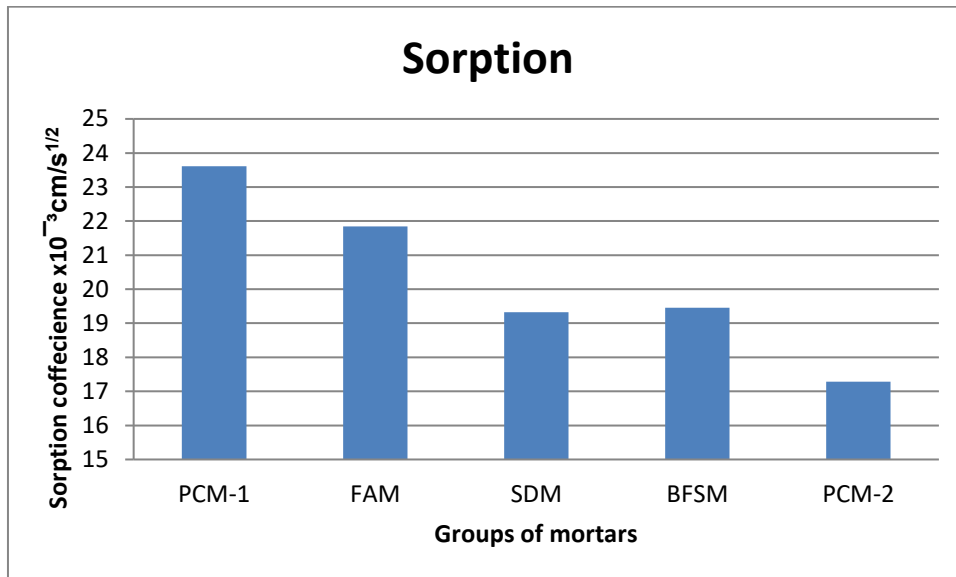


Fig.8. Group of mortars sorpton coefficient levels

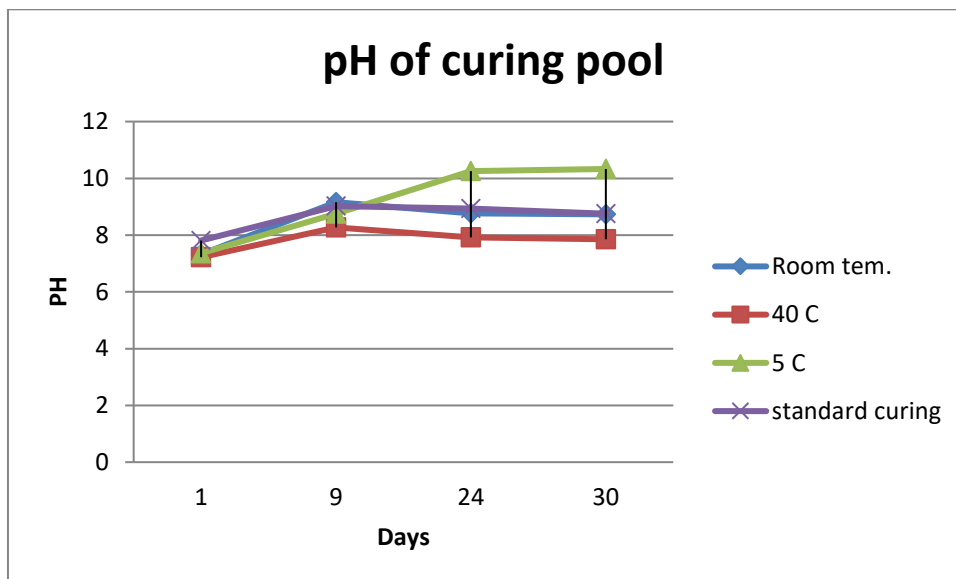


Fig.9. pH value of curing pool

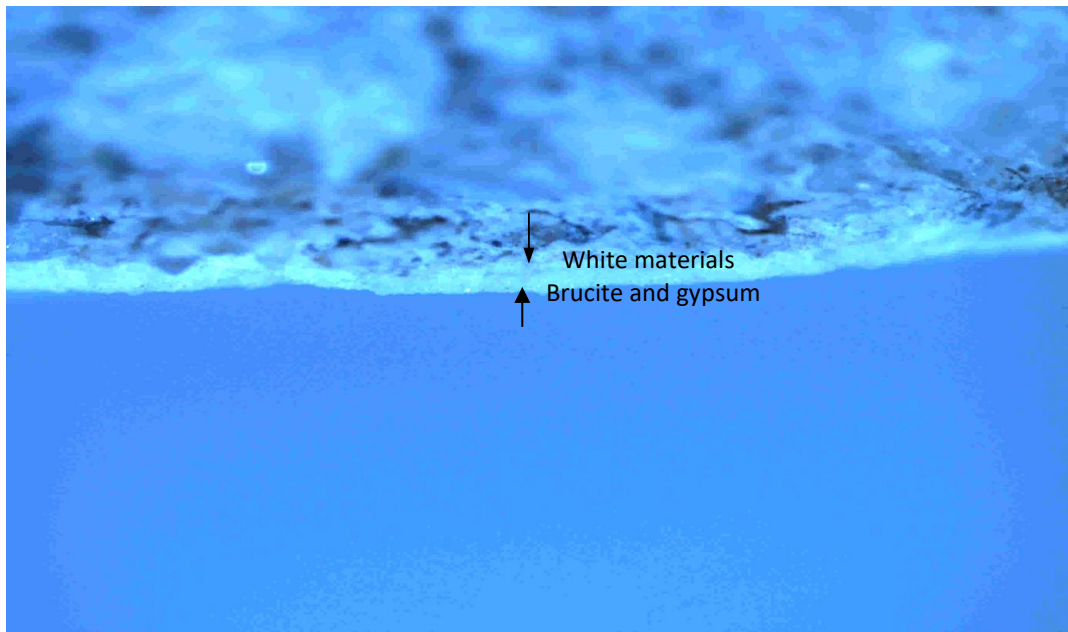


Fig.10. Occurrences of white materials on mortars

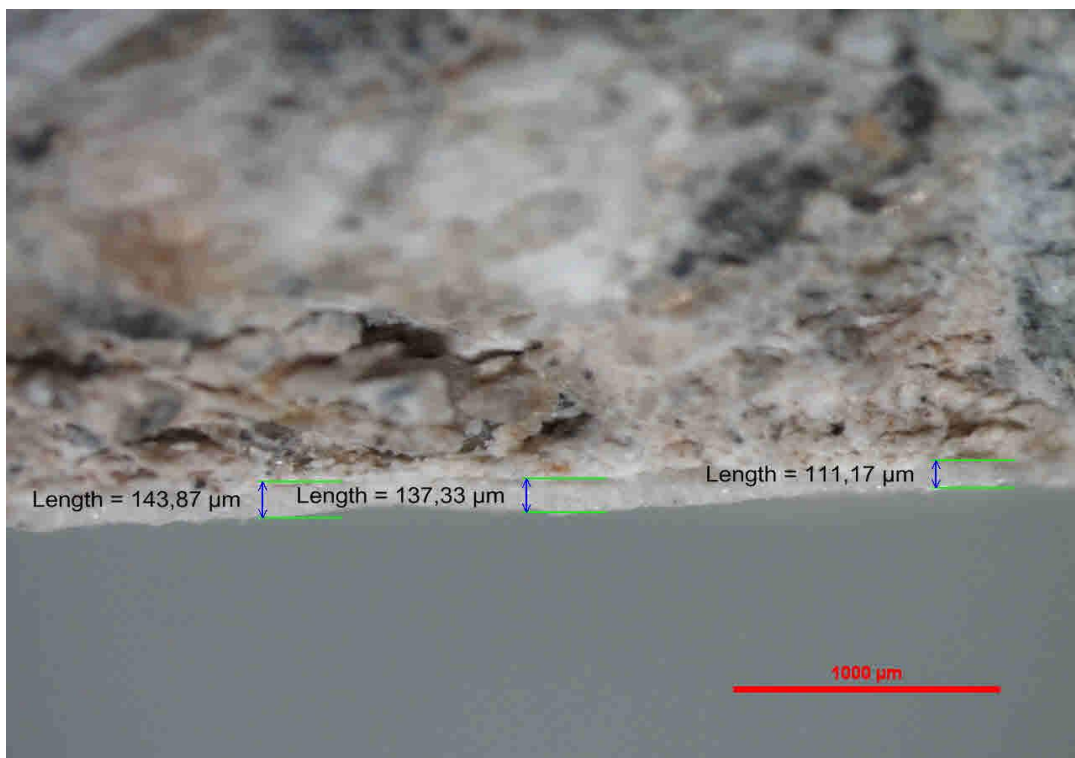


Fig.11. Thickness of White materials, standing 60 days at 40°C in magnesium sulfate solution on mortars.

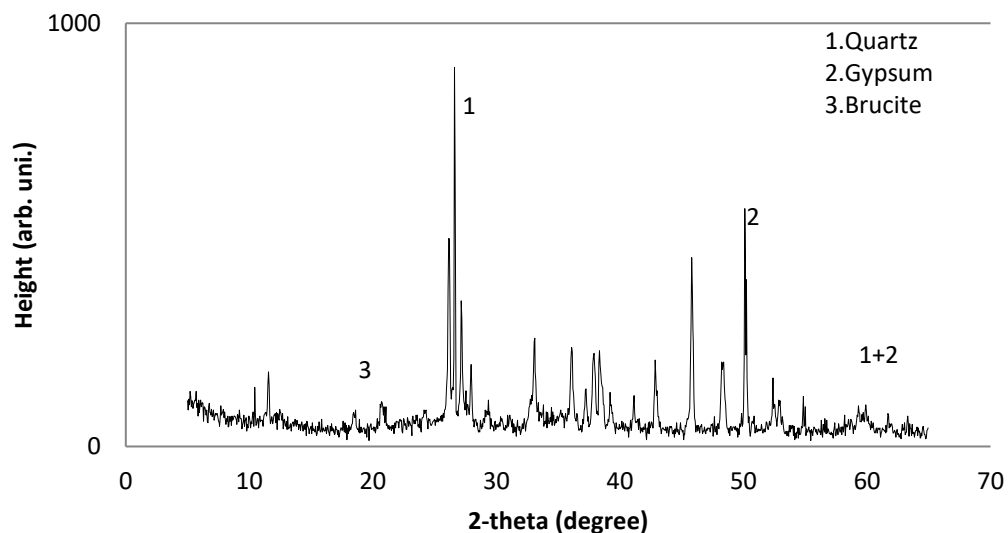


Fig.12. XRD of white materials

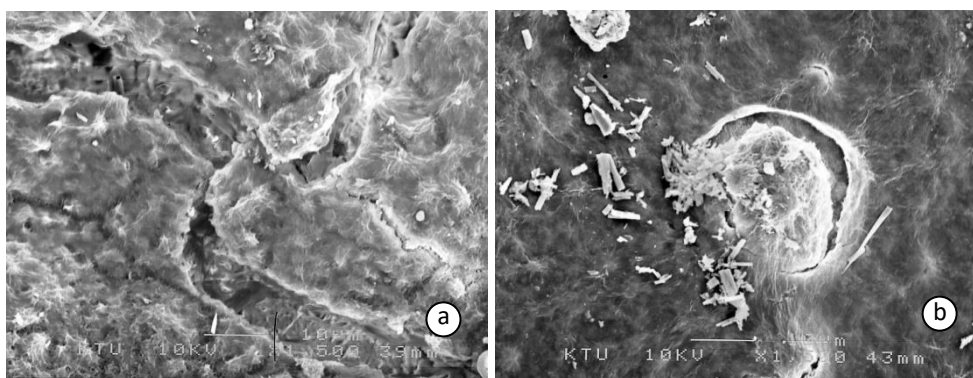


Fig.13. SEM micrograph of PCM-1, at 600 days, at room temperature in magnesium sulfate solution. (a) SEMx1500, (b) SEMx1500

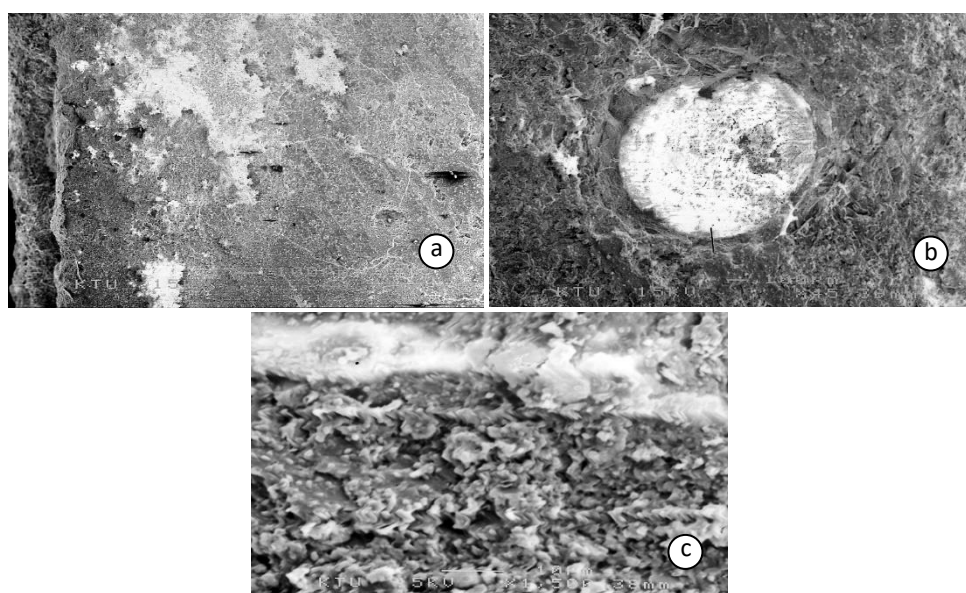


Fig.14. SEM micrograph of PCM-1, at 600 days, at 40° in magnesium sulfate solution. (a) SEMx60, (b) SEMx45, (c) SEMx1500

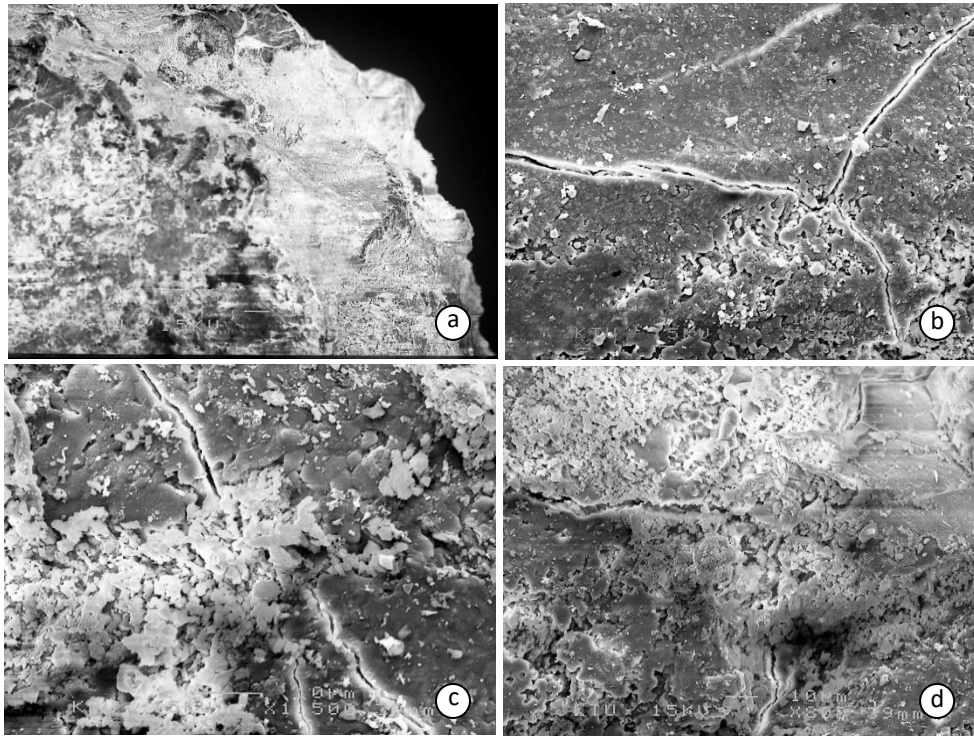


Fig.15. SEM micrograph of PCM-1, at 600 days, at 5° in magnesium sulfate solution. (a) SEMx60, (b) SEMx800, (c) SEMx1500, (d) SEMx800

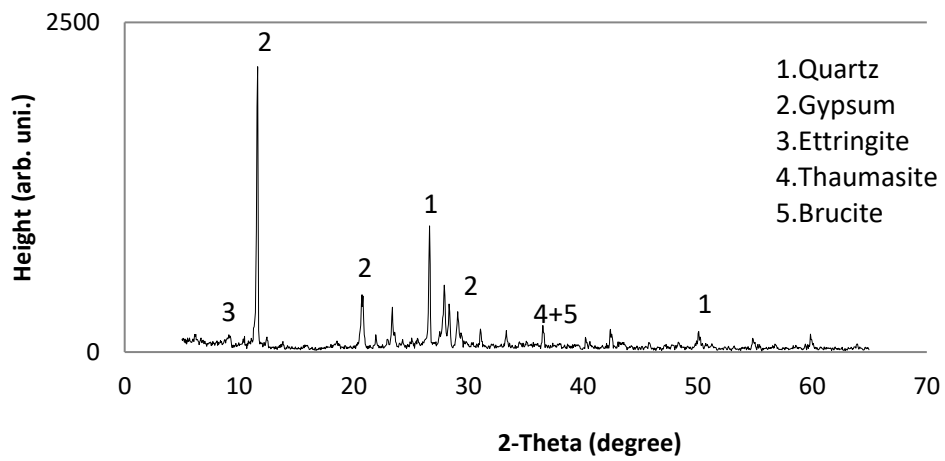


Fig.16. XRD of PCM-1 at room temperature.

4. CONCLUSION

The following conclusions can be made based on the test results of this study. Using the same types of aggregated and water/cement ratio, water absorption and sorption are important in magnesium sulfate attack. Mineral additive affected the absorption of mortars and prevent the mortars from magnesium sulfate attack. At the same ratio of W/C, high cement content mortars show the best performance in magnesium sulfate solution. With a less water /cement ratio reduce absorption therefore resistance of mortars increases against magnesium sulfate attack. Different temperature levels influence sulfate attack. The speed of occurring brucite and gypsum on mortars and pH levels of sulfate solution are the other parameters influencing magnesium sulfate attack. Mortars are affected at room temperature more than at 40°C and 5°C. Corruptions reduce at the same proportion of magnesium sulfate solution at high temperature. High temperature affected thickness and speed of formations, on white materials.

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