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EFFECTS OF $MgSO_4$ AND Na_2SO_4 SOLUTIONS ON CONCRETE MORTARS FROM CEMENT TYPES PKC 32.5 AND PC 42.5

ABSTRACT

Hydration products of various types of cement differ in both quantitative and qualitative aspects. This study aims to determine the nature and severity of such difference under certain Sulphate conditions. Investigated to this end were changes at intervals in physical and chemical behaviors of standard sand mortars from cement types PKC 32.5/B and PC 42.5 in Na_2SO_4 and $MgSO_4$ sulphate conditions. Mortar specimens cast in 40x40x160 mm mould were let to cure for 14 and 28 days in fresh water in addition to medium containing 5% and 10% Na_2SO_4 and $MgSO_4$ sulphate solutions. Several physical and chemical analyses and measurements with XRD and SEM instruments revealed that solution with $MgSO_4$ had a more detrimental effect than did Na_2SO_4 solution and that specimens in $MgSO_4$ solution had relatively lower compressive strength. In the study mortars from cement type KPC 32.5/B proved higher resistance to Sulphate medium than did mortars from cement type PC 42.5.
Keywords: Mortar, Pozzolan, Compressive Strength, Sulphate Resistance, Cement

$MgSO_4$ VE Na_2SO_4 ORTAMLARIN PKÇ 32.5 VE PÇ 42.5 ÇIMENTOLU HARÇLARA ETKİLERİ

ÖZET

Çimentoların hidratasyon ürünleri miktar ve özellikleri bakımından birbirinden farklıdır. Bu farklılığın sülfatlı ortamlarda yönünü ve şiddetini ortaya koymak bu çalışmanın amacını oluşturmaktadır. Bu çalışmada, PKÇ 32.5/B ve PÇ 42.5 çimentolarla üretilen standart kumlu harçların Na_2SO_4 ve $MgSO_4$ çözeltileri içindeki zamana bağlı fiziksel ve kimyasal değişimleri incelenmiştir. Söz konusu harç numuneler, 40x40x160 mm'lik prizmatik kalıplarla hazırlanmış olup 14 ve 28 gün süreyle içilebilir su ortamı yanında %5 ve %10'luk Na_2SO_4 ile MgSO4 çözeltilerinde kür edilmişlerdir. Araştırmada, $MgSO_4$'lı çözeltinin zararlı etkisinin Na_2SO_4'lı çözeltilere göre daha fazla olduğu ve $MgSO_4$ çözeltisindeki numunelerde basınç dayanımlarının daha düşük çıktığı, PKC 32.5/B çimentoların PÇ 42.5'tan daha fazla sülfatlara dayanıklı olduğu, yapılan fiziksel, kimyasal, XRD ve SEM analizlerle saptanmıştır.
Anahtar Kelimeler: Harç, Puzolan, Basınç Mukavemeti, Sülfat Dayanımı, Çimento

1. INTRODUCTION (GİRİŞ)

Concrete throughout its service life is subject to several chemical and/or physical impacts such as acts of nature, Sulphate attack, carbonization and acidic waters, which may in turn lead to wearing.

Sulphate attack was first ascertained in 1908 by the "United States Bureau of Reclamations (USBR)" [1] and the related researches have been held ever since.

In order to determine the Sulphate resistance of concrete, the latter is examined in two features, namely the nature of cement and the permeability of the concrete. On this account, ASTM stipulates as the basis "the V type cement with a content of C_3A and $2C_2A+C_4AF$ respectively no more than 5% and 25% [2].

Concrete, under certain chemical and/or physical impacts, tends to have more cavities in texture and the reinforcement may rust away, which in turn may lead to wearing and rather serious internal stress. Certain substances penetrating into concrete, e.g. Water, carbon dioxide, oxygen, sulphates, acids and chlorine are particularly responsible for various chemical reactions inside, which degrades the concrete seriously hampering its service life.

Water penetrating into concrete causes the hydroxide and salts in the texture of hardened cement mortars (paste) to dissolve and diffuse on the surface of the concrete, where they form a thin whitish layer called "flowers of sulphur".

Penetrating in hardened concrete, sulphates bring about detrimental effects causing it to expand and split and lead to various chemical reactions. Concrete is also effected by chlorine medium. Main detrimental factor from seawater to concrete structures result from the chlorine contained in salt water. Chlorine here accelerating the corrosion of reinforcement leads to the disintegration of concrete. Acid aggression in concrete results in dissolution of calcium hydroxide and calcium hydrated silica gel (C-S-H) contained in hardened concrete. A thin soft layer forms on the surface. The concrete develops more pores losing strength and durability [3].

In their research on the impact by sulphates from the mixing water on the cement mortar (paste) features, Kılınç and Uyan studied the effect of $MgSO_4$ and Na_2SO_4 solutions in the water on yield strength, flexural strength and compressive strength of the mortar (paste). The research ascertained the damaging effect of magnesium Sulphate on both bending and compressive strength and established that sodium sulphate had a more detrimental impact than did magnesium Sulphate in terms of Sulphate attack. Another striking finding was that the damaging effect by sodium Sulphate on compressive strength dependent on water/cement ratio.

Also found out in this research was that the adverse effects of harmful substances in the mixing water should be determined not only with 7-day results but with 28-day and 90-day results [4].

Atahan, Pekmezci, Uyan and Yıldırım studied the impact of Sulphate medium on the durability of concrete. Their research also emphasized the importance of cement dosage and water/cement ratio for the durability of the concrete [5].

Biricik and Aköz conducted a research on the effects of sodium Sulphate solution on mortars with and without stem ash as additive. In the research sodium Sulphate solutions with 10000 mg/l and 40000 mg/l SO_4^{2-} concentration were employed to investigate the Sulphate resistance of wheat stem ash, whose pozzolanic behavior had been experimentally ascertained.

The experiments were accomplished with standard mortars as well as with substituted mortars added with wheat Stem Ash at 8%, 16% and

24% by cement weight. Ash in the admixture proved to contribute to Sulphate resistance of the mortar even at rather high concentrations upto 180 days [6].

The effects of magnesium Sulphate, sodium Sulphate and sodium sulphur in the mixing water on fresh and hardened cement mortars were investigated by Uyan in a research, were Sulphate solutions prepared at various concentrations were used as mixing water. The results were analyzed on a large scale with a view to the impacts by Sulphate salts on fresh and hardened cement mortar characteristics [7].

The concentration of dissolved sulphate in water as well as passive water as opposed active waters are also significant factors in the determining the given effect. Normally, dissolved Sulphate concentrations exceeding 210 mg/l in water are presumed as the initiation of deterioration. Limit on Sulphate concentration in swamp like water is taken as 225 mg/l. As the regenerative action in stagnant waters is less than in freshwater, damaging effect is relatively smaller. With Sulphate concentrations up to 0,10 g/l in water, practically no effects are considered whereas in case of concentrations between 0,15 g/l and 1,00 g/l the effect is considered to exist and with the figure between 1,00 g/l and 2,00 g/l the effect is regarded as significant. If it exceeds 2,00 g/l it is a taken a serious effect. These figures are taken 50% more for estimations. The values given for sulphates such as CaSO4, Na_2SO_4 an K_2SO_4 can be taken half as much for $MgSO_4$ because the damaging effect of $MgSO_4$ occurs twice as much as the others do [8 and 9].

2. RESEARCH SIGNIFICANCE (ÇALIŞMANIN ÖNEMİ)

Amount and properties of hydratation products differs from each other. Having intensity and direction of that difference in sulphate based environment would barely show the necessity in which the cement sample has to be used under which circumstances. In this study, time dependent physical and chemical changes of PKÇ 35.5/B and PÇ 42.5 standardized mortars are of major interest.

3. MATERIAL (MALZEME)

Cement: Cement types PC42.5 from Iskenderun OYSA Cement plant and PKC/B 32.5 from Adıyaman Cement Plant (TS 12143) were utilized in the experiments. Physical features and chemical composition of bath types are given in Table 1.

Standard sand: For all mixtures in the research standard sand from Pınarhisar Cement Plant consistent with TS 819 [10].

Solutions: Composition and characteristics of Merc branded Na_2SO_4 and $MgSO_4$ are given in Table 1. Concentration of Sulphate solution was 33,800 mg/l with the pH value varying between 6 and 8 [11 and 12]. The aggressive (corrosive) volume versus sample volume ratio was kept constant at less then 1 cm^3 cement per 1 ml of solution [13].

4. METHODS AND DISCUSSION (YÖNTEMLER VE TARTIŞMA)

Experimental: Standard mortar samples were material ratios were as follows cement type PKC 32.5/B and type PC 42.5: 450 g standard sand: 1350 g. Amount of water: enough to allow for a dispersion of 105% to 115%

Shaking table acc. to TS 3322 was used to set S/C ratio in consistency control. Based on 225 g water addition to standard mortars, water content was increased with 5% increments each time and the diffusion percentage values of mixtures on the shaking table noted down. This process was continued until the diffusion rates reached 105% to 115%. Calculation of diffusion was worked out on the formula

$$\% = \frac{Ds - Di}{Di} x100$$

where D stands for the initial and final diameter of diffusion of the mortar in cm.

Mortars were mixed with laboratory type automatic mixer and compacted in automatic shaker. 24 hours after having been taken out of moulds, the specimens were placed in sulphate solutions, which formed the essence of the experiment.

Three cubic samples of mortars (40x40x160 mm) prepared at laboratory conditions acc. to design mixture as in Table 2 were cured for 14 and 28 days in %, 5% and 10% $MgSO_4$ and Na_2SO_4 solutions. Water/cement ratio was kept constant at 0.50 for all mortar mixtures. Samples taken out an completion of curing period were subjected to compression test (Figures 1, 2 and 3). The samples chosen for XRD and SEM analyses were soaked acetone to prevent any chemical reaction during storage period.

As can be seen in Figure 1 and 2 both 14 and 28 day compressive strength values of PKC 32.5/B samples held in both water and 5% MgSO4 medium appeared to be lower than those of the ones held in 5% Na_2SO_4 medium. As for PC 42.5 cement samples, those held for 28 days in 5% $MgSO_4$ medium had higher strength than the ones held in 5% Na_2SO_4 medium. PKC 32.5/B and PC 42.5 cement samples kept in 10 % $MgSO_4$ for 14 and 28 days displayed lower strength than those held in 10% Na_2SO_4 medium.

Related literature may read that sodium Sulphate has more damaging effect than magnesium Sulphate and lead to lower strength of samples [4]. This is considered to depend on the water/cement ratio in the composition of mortar sample and cement type and features.

Control sample with cement type PKC 32.5/B displayed a 14 day-strength of 24,2 MPa, yet lost 17% strength. When curing period was prolonged to 28 day, it was noted that with respect to 35,3 MPa strength control sample lost 9% and 16% strength respectively in 5% Na_2SO_4 solution and 5% $MgSO_4$ solution. Compressive strength of samples with PKC 32.5/B kept in solutions with 10% additive concentration proved to be lower than that of the samples in 5% solutions (Figure 1 and 2).

Among samples with PC42.5 cured for 28 days, compressive strength of those in 5% Na_2SO_4 solution diminished 20% when compared to that of the control sample whereas the loss in the strength of samples in $MgSO_4$ solution reached 34%. Strength losses in the samples 25% and 30% respectively in 10% Na_2SO_4 solution and $MgSO_4$ solution (Figure 1, 2 and 3).

Table 1. Chemical composition of cement types PKC 32.5/B and PC 42.5.
(Table 1. PKC 32.5/B ve PC 42.5 çimentolarının kimyasal kompozisyonu)

Components and characteristics		İskenderun Cement Plant(PÇ 42.5 (Literatüre)	Adıyaman Cement Plant PKÇ 32.5/B
CaO		62.84(62.97)	52.01
SiO_2		18.80 (19.80)	24.23
Al_2O_3		5.21 (5.61)	5.11
Fe_2O_3		2.59 (3.42)	5.25
MgO (Lim. ≤5%)		1.37 (1.81)	3.33
SO_3 (Lim. ≤3,5%) [14]		2.58 (2.86)	2.28
Insoluble residue (Lim. ≤5%)		0.72 (0.36)	-
Chlorine Cl^- (Lim. 0,10 ≤%) [14]		0.020	-
Loss of ignition (Lim. 5 ≤%) [14]		2.80	-
Physical Features			
Specific gravity g/cm^3		3.10 (3.15)	-
Blaine specific surface ≥3000 cm^2/g [14]		3980 (3410)	3750
Normal consistency (%)		29.4	26.0
Setting time (dk)	Start (dk)	145 (130)	-
	Completion (dk)	185 (160)	-
Compressive Strength			
Compression strength MPa	7 day	39.8 (39.9)	26.7
	28 day	49.5 (46.4)	38.4

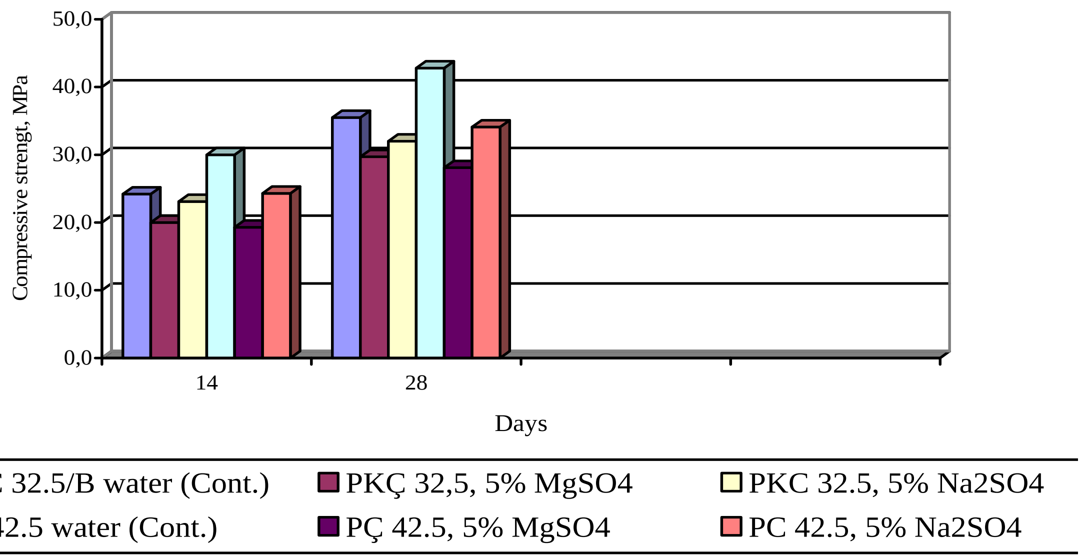

Figure 1. Compressive strength of mortar samples cured in water and 5% $MgSO_4$ and Na_2SO_4 solutions
(Şekil 1. Su ile %5'lik $MgSO_4$ ve Na_2SO_4 çözeltilerinde kür edilen harç numunelerinin basınç dayanımları)

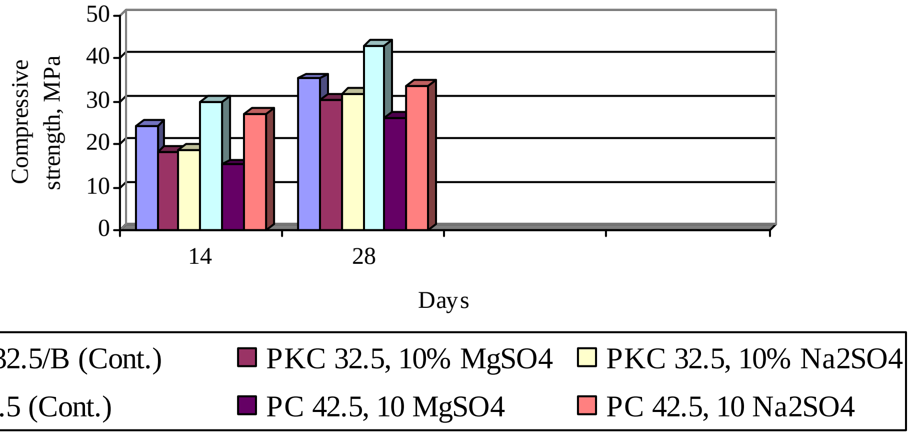

Figure 2. Compressive strength of mortar samples cured in water and in 10% $MgSO_4$ and Na_2SO_4 solution
Şekil 2. Su ile %10'luk $MgSO_4$ ve Na_2SO_4 çözeltilerinde kür edilen harç numunelerinin basınç dayanımları

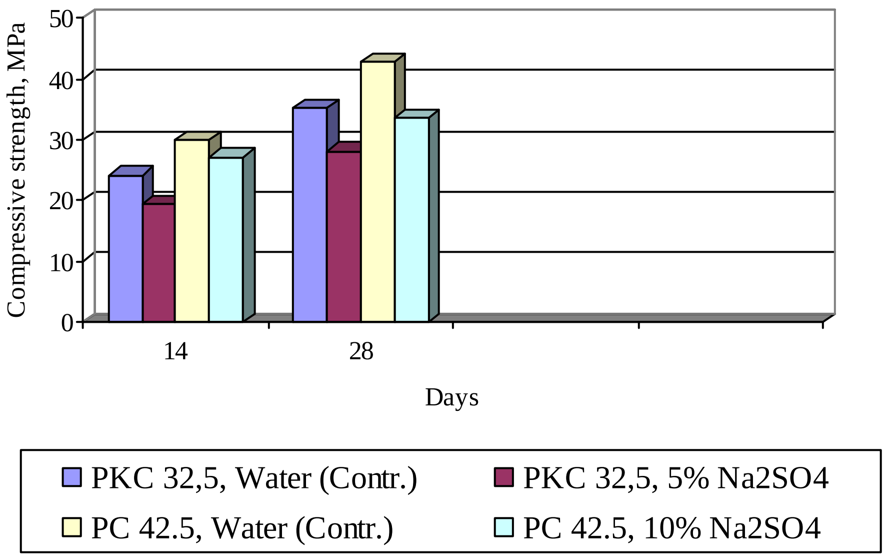

Figure 3. Compressive strength of mortar samples cured in water and 5% as wellas 10% $MgSO_4$ and Na_2SO_4 solutions
(Şekil 3. Su ile %5'lik ve %10'luk $MgSO_4$ ve Na_2SO_4 çözeltilerinde kür edilen harç numunelerinin basınç dayanımları)

Chemical Composition: Data from the chemical analyses obtained from Iskenderun OYSA Cement Plant are given in Table 3. When Al_20_3 content, which determines the rate and the nature of the reaction in cement, was examined for PC 42.5 cement type, the samples held in 10% solutions displayed lower values than those held in 5% solutions, which indicates that 10% $MgSO_4$ solution is more detrimental than 5% one (Table 2).

A similar comparison could be mode as to MgO. As MgO brings about volumetric expansion in cement, it is kept at lower contents. When checked in Table 3, MgO values in PKC 32.5/B and PC 42.5 samples in 5% and 10% Na_2SO_4 solutions appear almost near the values of control solution values. On the other hand, MgO contents of samples in 5% and 10% $MgSO_4$ solutions occurred approximately twice to three times as high. As to the same analyses for SO3, cement types PKC 32.5/B and PC 42.5 in 5% and 10% solutions displayed relatively higher values when compared with the control sample. The increase in SO_3 content denotes Ettringite formation [$Ca_6.Al_2$ $(SO_4)_3.26H_2O$] of the cement (Figure 4, 5, 6, 7, 8 and 9). SO_3 content, however, never accedes the limit values. Ettringite is a chemical formation which inhibites the reaction acting upon the development of cement and concrete.

Tablo 2. Chemical composition of samples on completion of 28 day curing period [15]
(Tablo 2. Numunelerin 28 günlük kür süresi sonunda kimyasal analizleri [15])

Mortar samples with cement type PC 42.5					
	Curing conditions				
	Water (Cont)	MgSO4 solution		Na2SO4 solution	
	0 %	5 %	10 %	5 %	10 %
CaO	39.34	38.06	36.93	35.90	39.59
SiO_2	31.43	29.48	34.45	36.36	30.26
Al_2O_3	2.93	3.14	2.90	3.09	2.80
Fe_2O_3	2.03	2.04	2.00	1.97	2.07
SO_3 (Lim.≤% 3.5) (TS 25) [14]	1.09	2.05	2.00	1.66	2.36
MgO (Lim. ≤ %5) (TS 25) [14]	0.47	1.75	1.47	0.45	0.50
K2O	0.19	0.26	0.24	0.24	0.22
Mortar samples with cement type KPC 32.5/B					
CaO	31.41	29.34	33.62	29.91	32.15
SiO_2	39.42	41.23	34.09	40.30	34.61
Al_2O_3	3.32	2.95	3.39	3.09	3.12
Fe_2O_3	3.74	3.63	4.30	3.69	3.93
SO_3 (Lim.≤%3) (TS 25) [14]	1.17	1.53	2.34	1.96	2.43
MgO (Lim. ≤%5) (TS 25) [14]	1.89	2.52	2.73	1.62	1.65
K2O	0.14	0.09	0.08	0.11	0.08

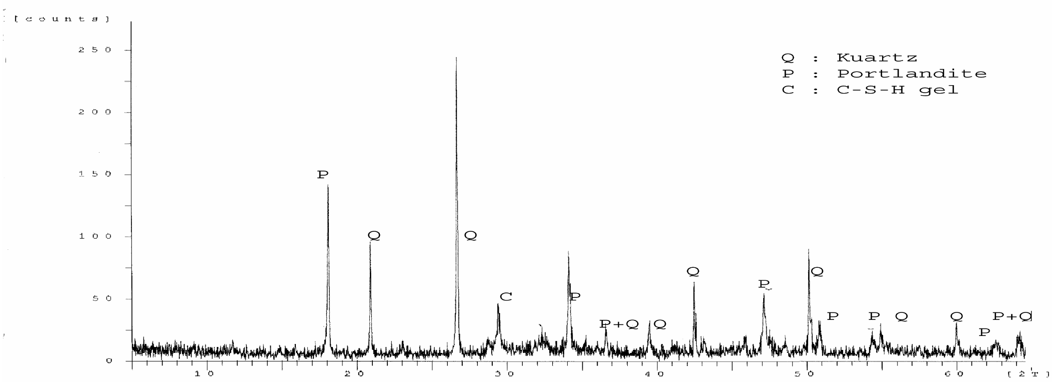

Figure 4. XRD analysis of control mortar samples with cement type PC 42.5 cured in water
(Şekil 4. Su içinde kür edilen PC42.5 çimentolu kontrol harç numunelerinin XRD analizi)

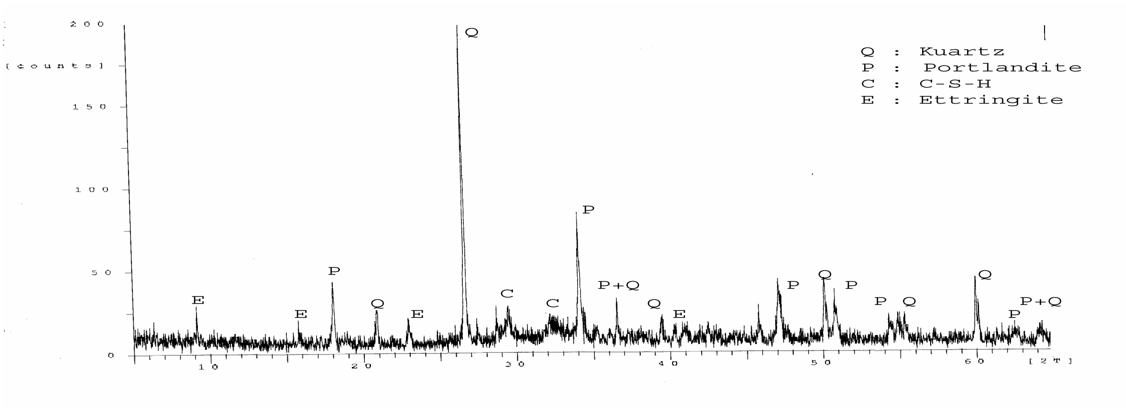

Figure 5. XRD analysis of mortar samples with cement type PC 42.5 cured in $MgSO_4$ solution
(Şekil 5. $MgSO_4$ çözeltisinde kür edilen PC42.5 çimentolu harç numunelerinin XRD analizi)

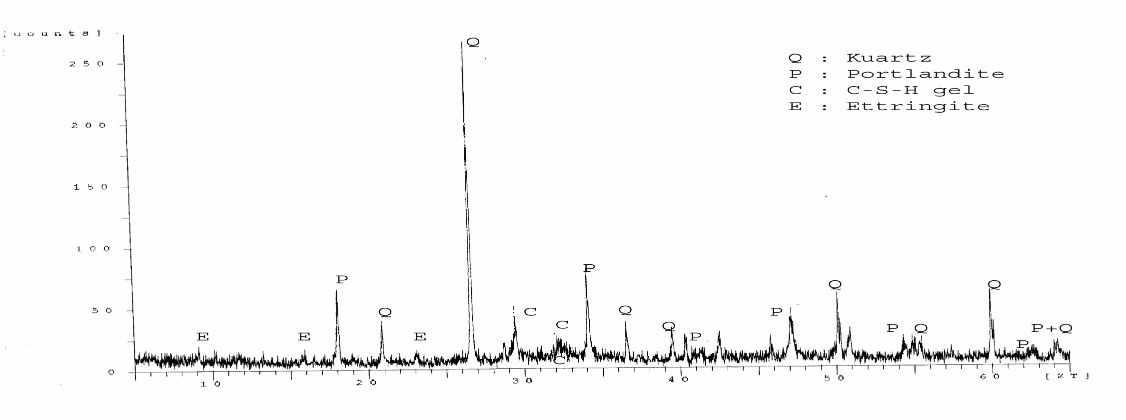

Figure 6. XRD analysis of mortar samples with cement type PC 42.5 cured in Na_2SO_4 solution
(Şekil 6. Na_2SO_4 çözeltisinde kür edilen PC42.5 çimentolu harç numunelerinin XRD analizi)

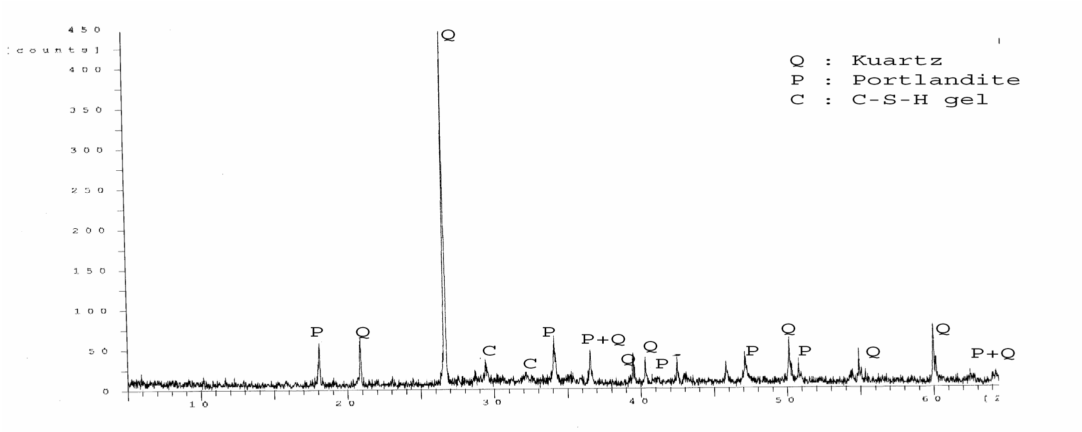

Figure 7. XRD analysis of control mortar samples with cement type KPC 32.5/B cured in water
(Şekil 7. Su içinde kür edilen PKC 32.5/B çimentolu kontrol harç numunelerinin XRD analizi)

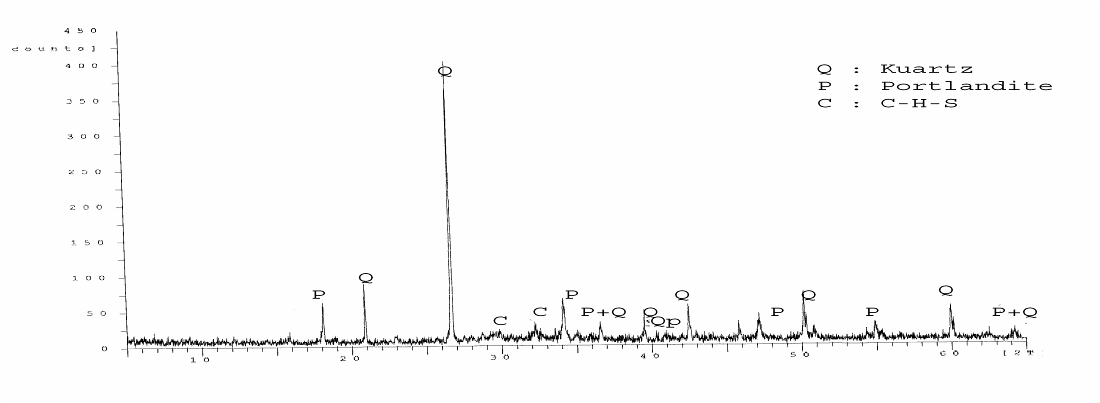

Figure 8. XRD analysis of control mortar samples with cement type KPC 32.5/B cured in $MgSO_4$ solution
(Şekil 8. $MgSO_4$ çözeltisinde kür edilen PKC 32.5/B çimentolu harç numunelerinin XRD analizi)

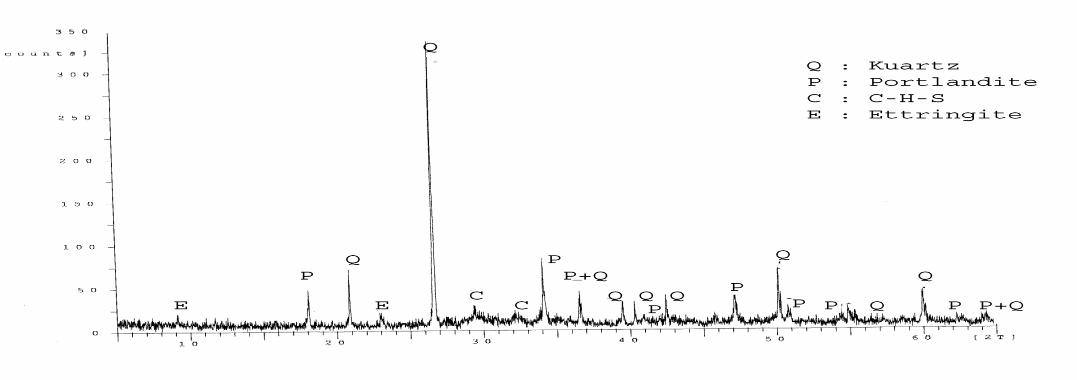

Figure 9. XRD analysis of control mortar samples with cement type KPC 32.5/B cured in Na_2SO_4 solution
(Şekil 9. Na_2SO_4 çözeltisinde kür edilen PKÇ 32.5 çimentolu harç numunelerinin XRD analizi)

Microscopic Analyses: Sulphate attack to cement, concrete or mortars can be checked up with a scanning electron microscope (SEM) inspection. For such analysis surface of cement mortar samples were plated with gold so that their micro structural composition was examined in SEM (LEO 435 VP) by method of secondary electron image (Fig. 10 to 15). Micro structural inspections of mortar samples after 28 days displayed aggregate grains and main hydration products of cement namely calcium silicate hydrate, calcium hydroxide and calsium sulpho aluminate formed after subjection to both water and Sulphate medium. No significant microstructural differences occurred between PKC 32.5/B samples kept in water and those subjected to Sulphate conditions. As to PC 42.5 samples, there were more remarkable sulpho aluminate (Ettringite) formation in those subjected to $MgSO_4$ and Na_2SO_4 conditions than those in water [16].

The sample mainly incorporates C-S-H phase in the form of rather vulnerable crystalline crusts similar to C-S-H Type I. This phase covering the surface of slag grains in cement Type PKC 32.5/B formed a bond in the form of rather vulnerable crystalline between hydration products. The layer of slag formation is seen to be rich in Al_2O_3 and MgO, which causes vulnerability. This web like structure rich in silicates consists of Ca, Si and Al arranged in flat plates (Figures 3, 4 and 5).

Figure 10. SEM structural analysis of control sample with cement type PKC 32.5/B cured in water
(Şekil 10. Su içinde kür edilen PKÇ 32.5 çimentolu kontrol numunesinin SEM mikroyapı incelemesi)

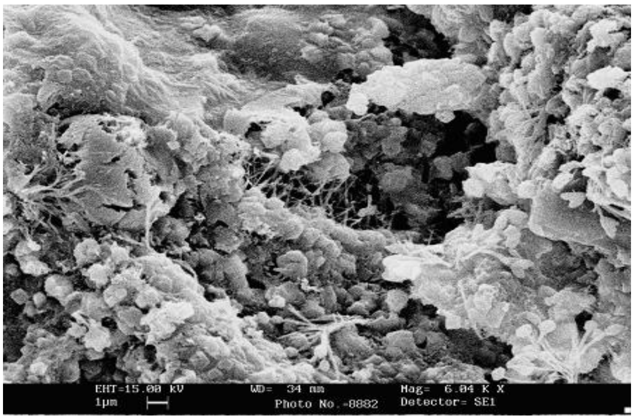

Figure 11. SEM microstructural inspection of control sample with cement type PKC 32.5/B cured in $MgSO_4$
(Şekil 11. $MgSO_4$ içinde kür edilen PKÇ 32.5/B çimentolu numunelerin SEM mikroyapı incelemesi)

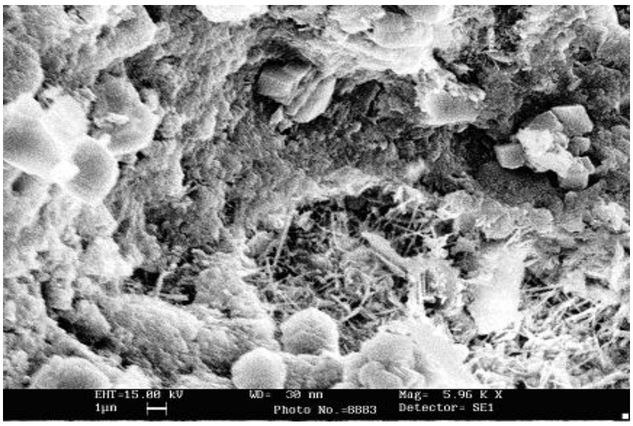

Figure 12. SEM microstructural inspection of samples with cement type PKC 32.5/B cured in Na_2SO_4
(Şekil 12. Na_2SO_4 içinde kür edilen PKÇ 32.5/B çimentolu numunelerin SEM mikroyapı incelemesi)

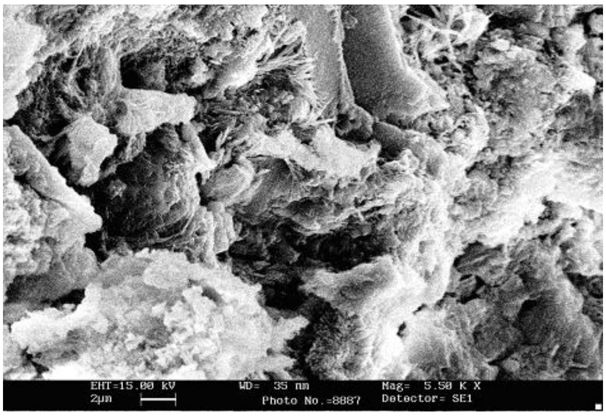

Figure 13. SEM microstructural inspection of control samples with cement type PC 42.5 cured in water
Şekil 13. Su içinde kür edilen PÇ 42.5 çimentolu kontrol numunelerin SEM mikroyapı incelemesi

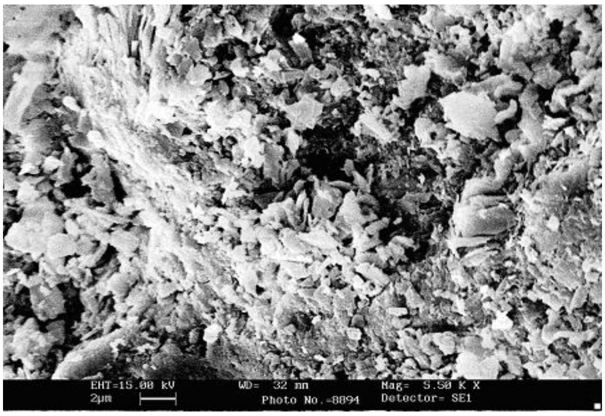

Figure 14. SEM microstructural inspection of samples with cement type PC 42.5 cured in $MgSO_4$
(Şekil 14. $MgSO_4$ içinde kür edilen PÇ 42.5 çimentolu numunelerin SEM mikroyapı incelemesi)

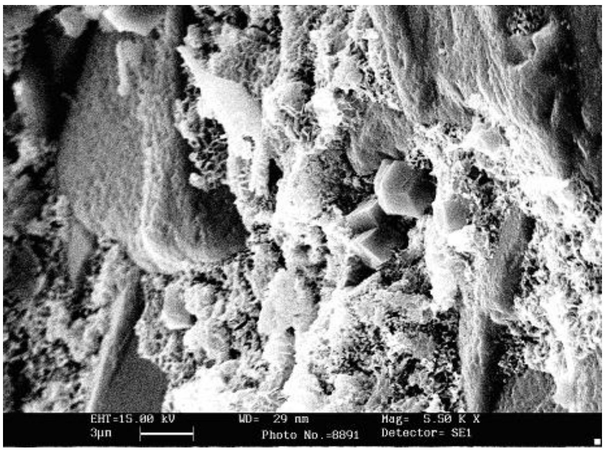

Figure 15. SEM microstructural analysis of samples with cement type PC 42.5 cured in Na_2SO_4
(Şekil 15. Na_2SO_4 içinde kür edilen PÇ 42.5 çimentolu numunelerin SEM mikroyapı incelemesi)

Compared micro structurally to mortars with cement PC 42.5, samples from type PKC 32.5/B cured in Na_2SO_4 contains more of portlandite developing homogenously. No uniformity occurs in portlandite web of samples with PKC 32.5/B cured in Na_2SO_4, portlandite bond seems to have interruptions, still effective in well developed parts. The same is valid for the samples with PC 42.5. Portlandite formation in the mortar samples, slowing rate of reaction, dissolution of portlandite by water forming cavities in the texture all denote a background to contribute to corrosion in reinforcement of concrete.

5. CONCLUSION AND SUGGESTIONS (SONUÇ VE ÖNERİLER)

Cements have so far improved both in quality and in production techniques. As part of such developments, an investigation is aimet at revealing durability features of mortars with PKC 32.5/B cement.

This experimental study sheds light on performance differences between standard mortars from PC 42.5 and those from PKC 32.5/B under aggressive conditions such as Na_2SO_4 and $MgSO_4$. To this end, physical tests and chemical analyses of cement samples were carried out, compressive strength of 40x40x160-mm cubic mortar samples was tested, mineralogical analyses were performed with X-Ray diffractometer to inspect internal texture and finally micro structural analyses were accomplished with a scanning electron microscope (SEM).

PKC 32.5/B is a kind of cement comprised of pozzolana. Relatively more remarkable formation of Ettringite on completion of 28-day curing time in $MgSO_4$ and Na_2SO_4 in PC 42.5 type cement which is supposed to be free from $Ca(OH)_2$ (free lime) proves that Sulphate environment has less damaging effect on the PKC 32.5/B cement.

PK 42.5 is a type of cement, whose composition and reaction features contribute to its durability; however, cement type PKC 32.5 is more resistant to Sulphate environment [15].

Where Sulphate solutions occur in the environment, PKC 32.5/B displays better durability features. As to the solutions, $MgSO_4$ has a more damaging effect than does Na_2SO_4. It is suggested that compression volues be read after repeated experiments with prolonged curing time in solutions.

XRD analyses of all samples displayed portlandite Ca $(OH)_2$ Table 4). Portlandite formation in PKC indicates poor ratio of additives in PKC production process. If the percentage of additives were adequate, pozzolana would join together the $Ca(OH)_2$ forming in the texture. Dissolutions of $Ca(OH)_2$ in cements under the effect of water and moisture causes the alkaline level in the medium to diminish even more. With low alkaline content, carbonation intensifies accelerating corrosion in reinforcing bars of the concreter [3].

ASTM [2] stipulates V Type cement as the basis for sulphate resistance, with the prerequisite that C_3A component and 2 C_3A+C_4AF content should not exceed respectively 5% and 25%, as can be seen in Table 1 PC 42.5 cements for exceed these values whereas PKC 32.5/B Type cement meets the requirement, which is another evidence that PKC 32.5/B cements are more resistant to sulphates than cement type PC 42.5.

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