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Research Article

Sulphate Resistance of Boron Active Belite Cement Concrete

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Keywords

Concrete,
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Sulphate effect

Abstract

The sulphate resistance of variable concretes, including Boron active belite cement (BABC), CEM I 42.5 R and CEM II 32.5 B-S cements, were determined in the present study. The compressive strength, ultrasonic velocity, Schmidt, and unit weight tests were applied to steam cured and water cured samples. Three Na2SO4 solutions of %5, %10 and %20 were prepared for each type of concrete except for the control group and the samples were exposed to the effect of sulphate solutions for 24 weeks. As a result, weight increase was determined in CEM I 42.5 R cement samples the least, and in BAB cement samples the most. As expected, pronounced chemical effect was not observed in samples of cement CEM II.32.5 B-S. On the other hand, while decreases or slight increases were observed in the ultrasound measurements of CEM I 42.5 R cement samples after sulphate test, critical increases were observed in BAB and CEM II 32.5 B-S cement samples. Under sulphate effect, water and steam cures had explicit effects on pressure resistances.

1.Introduction

Boron is a chemical element with symbol "B" and atomic number 5. Boron is concentrated on Earth by the water-solubility of its more common naturally occurring compounds, the borate minerals. These are mined industrially as water-soluble mineral sediment, such as borax and kernite (razorit). Important boron reserves of the world are found mainly in Turkey, USA and Russia. Among those, the share of Turkey is about 72 % [1].

Boron minerals are most commonly used in glass industry. Boric oxide, borosilicate glasses are essential components of textile glass fibers and insulation glass fibers. Boric oxide is an extra strong flux and glass former. In other words, it prevents devitrification. It increases the resistance against thermal shocks because it decreases dilatation [2, 3].

Boric oxide (B2O3) has long been used in cement production. Some researchers around the world have determined that they observed improvements in the quality of cement that they produced with pure boric oxide. In this regard, studies have been started in Turkey and colemanite has been determined the most favorable boron mineral in cement production [4-8]. The most suitable boron mineral in cement production is colemanite. Colemanite is a borate mineral found in evaporite deposits of alkaline lacustrine environments. Colemanite is a secondary mineral that forms by alteration of borax and ulexite. Colemanite contains 28% CaO, 6,5% SiO2, 42% B2O3 and 23,5% H2O. It includes all necessary raw materials for cement, and does not include any chemical component or alkalis that would negatively affect the cement quality [1, 4-12].

According to the Yeşilmen and Gürbüz [13] BAB cement offers the advantage of high durability, low heat of hydration, and improved long term strength along with reduced energy and emissions during its production process compared to conventional cement. The production of BAB cement is yet to be continued in industrial scale. For this work required cement, has been taken with special permission from Turkish National Boron Institute.

Studies in Turkey on cement and concrete with boron additives gained importance in recent years with these developments. Today, boron and derivatives are added or used in the production of 250 different items used in different industries. Most important Boron minerals can be given as Borax (Tincal), Kernit, Uleksit, Colemanite, Pandermite and Hydroboracid. Studies on using these waste materials that can be harmful for the environment also, are going on. Studies are performed and good results are obtained from using these wastes especially in concrete production by partial replacement with cement. Thereupon these results, researches started on using these wastes at the production of clinker [14]. In general, belite cement has lower early and higher late strengths than Portland cement. High belite cements (HBC) are those in which there is reversal in the preponderance of alite by belite without sacrifice of early age strength in mortar and concrete.

Topçu et al [14], determined that, replacement of tincal wastes with cement in concrete production at around 3-5 % cause slowdown in setting, but more dense internal structure can be obtained and cement economy can be achieved.

Saglik et al. [15-16] compared compressive strength of concrete specimens cast using fresh BAB cement and ordinary Portland cement (OPC). The results indicated the 28-day strength of concrete cast using two cement types was almost the same (i.e., around 2% difference) and the strength of BAB cement at 3-month age was 9% higher than the OPC specimens.

It is an advantage in BAB cement that it requires less water for hydration than Portland cement and thus it is easier to adjust the low water/cement ratio. Moreover, thanks to low hydration temperature, shrinkage cracks are minimized and therefore, increase of permeability by shrinkage cracks is prevented.

Ettringite Formation and Sulphate Attack on Concrete

Ettringite formation is considered to be the cause of most of the expansion and disruption of concrete structures involved in the sulphate attack. However, not necessarily any sulphate attack is

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caused by ettringite formation. Moreover, ettringite formation can be advantageously used without any sulphate attack on concrete [17]. Harmful effect of sulphate attack stems from the ettringite with increasing volume and plaster that are formed by the chemical reaction of sulphate ions with alumina (C3A) and calcium (Ca(OH)2) components in hardened concrete.

Soils containing sodium, potassium, magnesium, and calcium sulphate are the main sources of sulphate ions in groundwater. For external sulphate attack to occur the following three conditions must be fulfilled: i) High permeability of concrete ii) Sulphate-rich environment iii) Presence of water

Sulphur, is usually found in clay as the sulphate of calcium, magnesium, sodium, potassium or iron, or as iron sulphide. Generally, the proportion is small. If, however, there is carbon in the clay and insufficient time is given during burning for proper oxidation of carbon and sulphur, the latter will cause the formation of a spongy, swollen structure in the brick and the brick will be decolored by white blotches [18].

Durability of concrete is defined as; its ability to resist chemical and physical attacks that lead deterioration of concrete during its service life. These attacks are leaching and efflorescе, sulphate attack, acid attack, carbonation, alkali-aggregate reaction, freezing–thawing and abrasion. Surfaces of the concretes exposed to sulphate attack characteristically develop a whitish appearance. Corrosive effect of sulphate generally starts from sides and edges of concrete blocks. Then, this effect intensifies inwards causing the decay of concrete surfaces layer by layer as blocks [19]. In the meantime, blended cements, especially Boron modified active belite cement and blast furnace slag cements should be preferred instead of high early strength Portland cements, in the manufacture of concrete products, such as prestressed concrete parts, which are more accurate to ettringite formation.

2. Material and Method

Aggregate used in this study was supplied from the crushed aggregate produced in the sand pit of Aşkale Çimento Inc. in Erzurum province. Granulation of the aggregates comprising the concrete was determined to correspond to the ideal area according to TS 802 in terms of the biggest grain size. The biggest grain size of the aggregates used in the concrete mixes was measured as 16 mm. as a result of this arrangement, ratios of the grain classes were determined as 40% for 0–2, 25% for 2–8 and 35% for 8–16. Physical propertied of the crushed aggregate are presented in Table 1.

Table 1. Tensile Properties of CFRP and epoxy used

Name and Supplier	Type	ρ (g/cm^3)	E (GPa)	σ (MPa)	ε (%)
BASF, MasterBrace, 300/50 CBS	Thermoplastic	1.77	227	3800	1.67
BASF, MasterBrace, SAT 4500	Low viscosity epoxy	0.983	3.034	55.2	3.5

In this study, three types of cement CEM II 32.5 B-S, CEM I 42.5 R and boron active belite cement (BABC) was used. The mechanical, physical and chemical properties of the BABC used in this study are summarized in Table 2 and 3.

Table 2. Mechanical and physical properties of BAB Cement

Properties	Requirements			
Strength Class	32.5 N	32.5 R	42.5 N	42.5 R
2 days Compressive strength (MPa)	-	≥10	≥10	≥16
7 days Compressive strength (MPa)	≥16	≥20	≥20	≥30
28 days Compressive strength (MPa)	≥32.5 ≤42.5	≥32.5 ≤52.5	≥42.5 ≤52.5	≥42.5 ≤62.5
90 days Compressive strength (MPa)	≥42.5	≥52.5	≥52.5	≥62.5
Setting Times, TS EN 196-3, min	≥75		≥60	
Thermal expansion (mm)	≤10			
Heat of hydration (cal/g) (At the end of 7 days according to the TS EN 196-8, at the end of 41 hours acc. to TS EN 196-9 or measured with isothermal conduction colorimeter method at the end of 7 days)				52.5

Note: Strength at the 90. day is given for information, it is not a requirement

Ultrasound speed measurements were taken with the digital ultrasonic speedometer in our laboratory. Direct measurement method was used for the determination of ultrasound speeds. Speed tests were applied for all samples. Moreover, ultra-voice speed of the sampled exposed to sulphate effect were measured again at the end of the experiment.

New generation super plasticizer, polycarboxylic ether based GLENIUM C303, was used in concrete mixes. Produces concretes were placed into plastic cylinder molds of 10x20 cm. surfaced of the samples were covered with a piece of nylon for preventing the loss in moisture and kept in the molds for 24 h for water and air cure samples.

Table 3. Chemical composition of BAB cement

Elements	**Amount of %** (< requirement by mass %)
Silicon dioxide (SiO_2)	19.1
Aluminum dioxide (Al_2O_3)	4.68
Ferric oxide (Fe_2O_3)	3.42
Calcium oxide (CaO)	57.1
Magnesium oxide (MgO)	1.32<5
Chloride (Cl-)	% 0.001<0.1
Sulfur trioxide (SO_3)	2.68<3.5
Loss on ignition	3.82<5
Insoluble residue	0.70<5
Equivalent alkanies	0.86
Boric oxide (B_2O_3)	3.00
Clinker	86.1
Gypsum	4.85

Totally 66 cylindrical concrete samples of BAB cement were poured. Stream cure was applied to 33 of them and water cure was applied to the other 33 samples. Samples were demolded after being kept under 20 °C temperature and 90% relative humidity during 24 h, saturated for lime and then kept in a cure pool of 23±2 °C for 28 days. These specimens were immersed in water at temperatures of 23 ± 2 °C during 28 days. Samples prepared for stream cure were subjected to cure process for 36,5 h according to the characteristics in Table 4 and necessary experiments were conducted on the samples kept for 24 h. Mean values of the restitution of each sample were determined by hitting different areas of the samples for 20 times with manual test hammer. Then, each sample was subjected to fracture test by test press and their compression resistance was calculated. Afterwards, a correlation was set between the compression resistances and mean restitution values.

Table 4. Stream cure cycles and characteristics

Stream Cure Cycles	**Pre-waiting Period**	**Heating Period**	**Heat Absorption Period**	**Cooling period**	**Total Stream Cure Cycle**
Temperature (°C)	25	25-70	70	70-32	-
Time (Minutes)	240	240	1440	270	2190

Pulverulent Na_2SO_4 with 99% purity was solved with pure water in order to increase the sulphate resistance of the concretes with BAB cement. Sulphate solutions were prepared by adding Na_2SO_4 in three different ratios as 5%, 10% and 20% of the pure water weight. Concrete samples were exposed to sulphate effect for 24 weeks by inserting these solutions. Height and diameter measurements were taken every week in the first 4 weeks and every other week in the next 20 weeks. In order for these measurements to be conducted under the same conditions, weights of the samples were measured at the time of saturated dry surface. Photos of the changes in the samples after 24 weeks were taken with high resolution, their ultrasound speed measurements were performed again and compression resistances were determined after Schmidt hammer test.

3. Results and Discussion

Bulk density values of CEM I 42.5 R, CEM II 32.5 B-S and BAB concrete groups to be subjected to water cure are 2396 gr/dm^3, 2364 gr/dm^3 and 2365 gr/dm^3 respectively. Bulk density values of CEM I 42.5 R, CEM II 32.5 B-S and BAB concrete groups to be subjected to atmospheric steam cure are 2393 gr/dm^3, 2388 gr/dm^3 and 2360 gr/dm^3 respectively. At the end of atmospheric steam curing and 28 days of water curing, compression resistances of the samples crushed in press machine were determined after ultrasound speed measurement and Schmidt hammer test. Results are presented in Table 5.

Table 5. Results of the hardened concrete experiments according to cure conditions

	Cement Type	No	Ultrasound (m/s)	Schmidt	Compressive Strength (MPa)
Atmospheric steam curing (36.5 hours)	CEM I 42,5 R	1	4098	22.1	31.86
		2	4132	22.7	31.09
		3	4184	21.6	34.48
	CEM II 32,5 B-S	1	3875	17.7	17.75
		2	3898	22.1	17.34
		3	3964	22.0	17.16
	BAB	1	3880	17.2	15.08
		2	3936	18.4	18.21
		3	3946	17.9	16.91
water cure (28 day)	CEM I 42,5 R	1	4380	29.5	49.99
		2	4370	30	47.40
		3	4360	27.7	50.13
	CEM II 32,5 B-S	1	4174	21.3	38.05
		2	4080	18.8	36.20
		3	4158	19.8	35.79
	BAB	1	4301	21.2	35.28
		2	4390	20.9	35.59
		3	4329	22.4	36.05

Although samples with the lowest weight change percent during the experiment belong to CEM I 42.5 R, it is important in terms of sulphate effect that samples that are chemically the most affected also belong to CEM I 42.5 R.

According to the surface hardness of the concrete, Schmidt hammer tests were made in order to observe compressive strength. It was observed that the concrete surface hardness values at atmosphericic steam curing test, decreased if the BAB cement increased in the mixture. No significant change was observed in the height and diameter measurements of the samples subjected to sulphate effect.

3.1. Findings of the Sulphate Effect

Characteristic appearance of a concrete subjected to sulphate effect are white stains, especially expanding from the edges and sides to the whole block, cracks and flakings. Concrete subjected to sulphate effect is seen to crumble and soften easily (Baradan, 2002). In the present study, the above stated situation was not observed in CEM II 32.5 B-S and BAB concretes during the experiment period. Explicit deterioration was observed in the CEM I 42.5 R concrete samples inserted into 10% and 20% Na_2SO_4 solutions. Deterioration was not that explicit in 5% Na_2SO_4 solution. Especially the lower parts of the CEM I 42.5 R samples, which are normally excessively rigid, softened after being subjected to sulphate effect in 10% and 20% solutions, and spontaneous break-offs were observed (with the largest diameter of 2 mm). Negligible (about 1-2 grams) weight losses were observed in the water and steam cured CEM I 42.5 R samples waited in 20% Na_2SO_4 solution. Break-off or deterioration was not observed in the concrete samples including BAB and CEM II 32.5 B-S cem

In the Figure 1 and Figure 2, we have investigated the weight changes of the samples inserted into 20% H2SO4 %20 solution cured in water and stream that would cause the most intense effect, as an example for the sulphate effect on the concretes waited into 5%, 10% and 20% Na2SO4 solution prepared for this study.

Figure 1 shows the cumulative weight change percentages of the concrete samples prepared with 3 types of cement waited in 20% Na2SO4 solution and cured in water. Under these solution conditions, the biggest weight change was seen in the concretes with CEM II 32.5 B-S' and BAB cement. 1.58% weight change in the 24th week in the concretes prepared with BAB cement is not an important value under normal conditions. However, the deformation this caused would be great when long term sulphate effect is taken into consideration. It can be said that sulphate resistance of these kinds of concretes prepared with this kind of cement is low. On the other hand, it should be noted that no new cement has been supplied and cement waited for a long period has been used in this experiment, because BAB cement has no standard usage and thus no standard production.

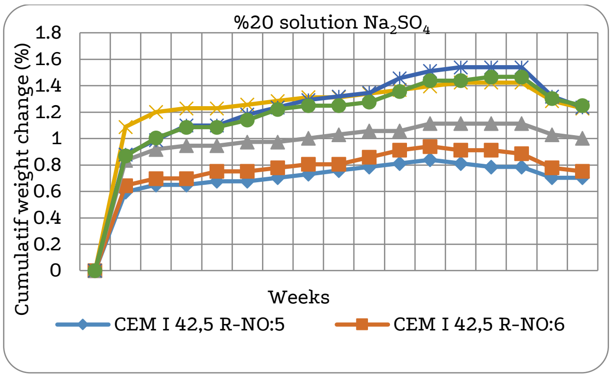

Figure 1. Weight change of the H2SO4 samples cured in water

Figure 2 shows the cumulative weight change percentages of the concrete samples prepared with 3 types of cement waited in 20% Na2SO4 solution and cured in steam. Differently in this group, weight changes of the samples belonging to the same cement type were nearly parallel. It was seen that weight loss was high in the concretes with BAB cement between the 10th and 24th weeks.

3.2 The Effect of Cure Conditions under Sulphate Effect on Compression Resistance

As can be seen in Figure 3, compression resistances of the concretes prepared with CEM I 42.5 R cement were higher than other concretes under both cure conditions. It was observed that compression resistances of the concretes prepared with BAB cement in water cure, except for 20% water cure, were higher than those values of the same samples in steam cure.

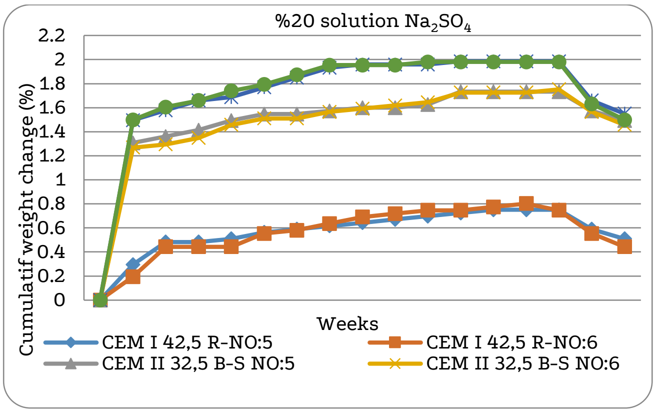

Figure 2. Weight change of the H2SO4 samples cured in steam

Photographs of the samples of BAB cement in steam and water cure taken after sulphate test are shown in Figure 4 and 5.

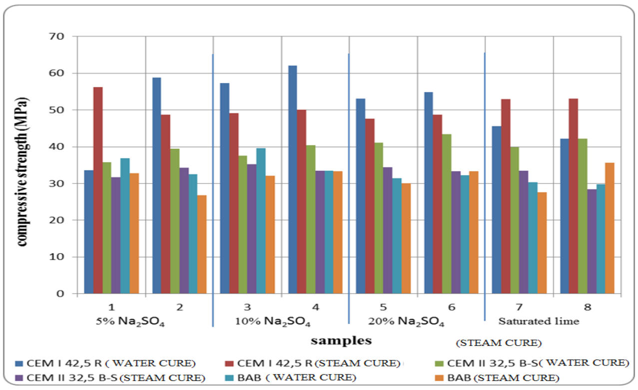

Figure 3. The effect of water and stream cure conditions under sulphate effect on the compression resistance

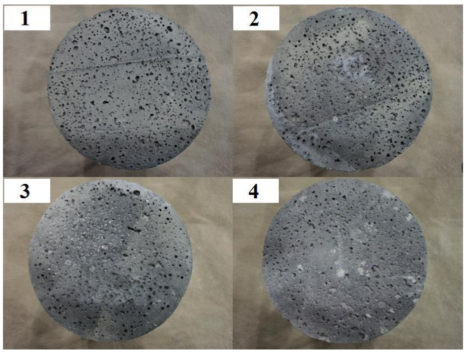

Figure 4. Photographs of the samples of BAB cement in steam cure taken after sulphate test (1: %5 Na2SO4, 2: %10 Na2SO4, 3: %20 Na2SO4, 4: Saturated Lime)

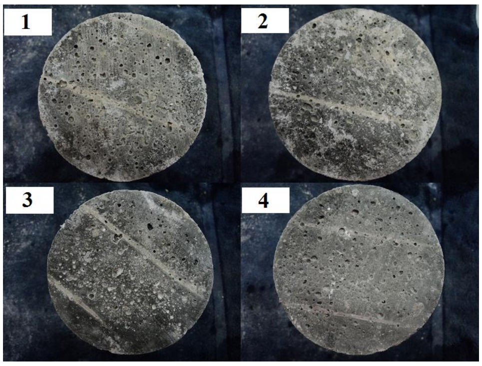

Figure 5. Photographs of the samples of BAB cement in water cure taken after sulphate test (1: %5 Na$_2$SO$_4$, 2: %10 Na$_2$SO$_4$, 3: %20 Na$_2$SO$_4$, 4: Saturated Lime)

4. Conclusions

Result of the present study, aimed at determining the sulphate resistances of the concretes prepared with heat-treated and not heat-treated CEM I 42,5 R, CEM II 32,5 B-S and BAB cements, are summarized below. It is concluded that the improper storage conditions affect the mechanical properties of BAB cement, but the deterioration in the performance of the cement is less pronounced than OPC.

- The most impermeable concrete production among the experiment samples subjected to sulphate effect for 24 weeks was observed in the concretes produced with CEM I 42,5 R cement. It is an expected situation that Na$_2$SO$_4$ salt negatively affects CEM I 42,5 R cement because of high C$_3$A and C$_3$S content of the cement.
- No explicit chemical effect was seen in the concretes poured with CEM II 32,5 B-S cement during the defined experiment period.
- While important decreases were seen in the ultrasound speed measurements of the samples belonging to water cured CEM I 42,5 R cement under sulphate effect, critical increases were observed in the samples of CEM II 32,5 B-S and BAB cements. Most serious decreases were seen in the samples of 10% Na$_2$SO$_4$ and 20% Na$_2$SO$_4$ solutions.
- Although increases were observed in the concrete samples of steam cured CEM I 42,5 R cement under sulphate effect because of the continuing setting, they were at the low levels compared to CEM II 32,5 B-S and BAB cements. This led to the idea that chemical effect was higher in the samples of CEM I 42,5 R cement. Since setting was completed 60% in steam cured samples, increases in these samples were observed at higher levels.
- No chemical effect was observed in the samples of BAB cement in both visual inspection and in measurements and experiments. The reason for this was that C$_3$A (causing increases in the volume and creating ettringite and gypsum by reacting with Na$_2$SO$_4$) was below 7% and C$_3$S was not even present. The fact that Ca(OH)$_2$ (a product of hydration) was formed 2,2 times more

than C_2S (active phase of BAB cement in C_3S) provided advantage in terms of ettringite and gypsum formation. In short, 2,2 times more reaction occur in CEM I 42,5 R cement than in BAB cement despite C_3S content.

- After all experiments were finalised, expected compression resistance values were not observed in the samples of BAB cement. The possible cause of this situation was the usage of awaited BAB cement.
- In the concretes poured with BAB cement, setting was observed, difficulties were experienced during the placement of the concrete and as a result, more porous concrete than expected was attained. It was possible that awaited concrete might cause this or there would be a mismatch with the new generation super plasticizer. Further studies to clarify this situation would be beneficial.
- Concrete of BAB cement with low permeability is thought to be safer in the structures to be subjected to sulphate effect, considering the protection from physical and chemical effects. BAB C concretes also show better resistance to sulphate attack, reduce permeability and improve water tightness.

Declaration of Conflict of Interests

The authors declare that there is no conflict of interest.

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