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The Effect of Boric Acid on Mechanical Properties and Structural Characterization of Self-Compacting Concrete

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Keywords
Self-Compacting Concrete, Boric Acid, SEM, XRD

Abstract: Within the scope of this study, it is aimed to produce boric acid doped Self-Compacting Concrete (SCC) to be used especially in other buildings that are exposed to nuclear power plants and radiation. Within this scope, a reference sample without boric acid and which fulfils the conditions of being SCC was produced, and 0.5%, 1.0%, 1.5%, 2.0%, 2.5% boric acid was added to concrete water respectively by weight in addition to the reference sample design. Freshly Mixed Concrete Flow Table Test, V-Funnel, L Shape Box, U Shape Box Tests were performed on these samples in order to observe whether there was a change in the conditions of being SCC. Compressive strength and flexural strength tests were performed in order to compare the change in mechanical properties of obtained samples. Also, SEM and XRD analyses were carried out in order to observe the structural characterization changes. As a result of the studies carried out, it has been seen that boric acid added concretes have the feature of being SCC. The compressive strength of the samples was determined to be respectively 44.28, 59.4, 52.78, 48.6 and 47.8 MPa. It has been observed that boric acid additive causes an increase in pressure resistance up to 1%, while it causes a decrease in 1.5%, 2% and 2.5%. In SEM images, it has been observed that boric acid accelerates the formation of C-S-H gels by connecting C-H to yourself over time, and with this effect, an increase in compressive strength is achieved over time. It was determined that the formation of C-S-H gel provides the structure to be more impermeable and durable.It also caused a decrease in the porous structure in boric acid material.

Borik Asidin Kendiliğinden Yerleşen Betonun Mekanik Özellikleri ve Yapısal Karakterizasyonuna Etkisi

Anahtar Kelimeler
Kendiliğinden-Yerleşen Beton, Borik Asit, SEM, XRD

Öz: Bu çalışma kapsamında, özellikle nükleer santraller ve radyasyona maruz olan diğer yapılarda kullanılabilecek borik asit katkılı Kendiliğinden Yerleşen Beton (KYB)ların üretilmesi amaçlanmıştır. Bu kapsamda, KYB olma şartlarını sağlayan ve borik asit ilavesiz bir referans numune üretilmiş olup, oluşturulan referans numune dizaynına ilave olarak beton suyuna ağırlıkça sırasıyla %0.5, %1.0, %1.5, %2.0, %2.5 borik asit ilave edilerek numuneler oluşturulmuştur. Bu numuneler üzerinde KYB olma şartlarında değişim olup olmadığını gözlemlemek için Taze Beton Deneylerinden Yayılma Tablası, V-Hunisi, L-Kutusu, U-Kutusu deneyi yapılmıştır. Elde edilen numunelerin mekanik özelliklerindeki değişimi kıyaslamak için basınç dayanımı ve eğilme dayanımı deneyleri yapılmıştır. Ayrıca yapısal karakterizasyon değişimlerini gözlemlemek içinde SEM ve XRD analizleri yapılmıştır. Yapılan çalışmalar neticesinde borik asit katkılı betonların Kendiliğinden Yerleşen Beton olma özelliği taşıdığı görülmüştür. Numunelerin basınç dayanımının sırasıyla 44,28, 59,4, 52,78, 48,6 ve 47,8 MPa olduğu tespit edilmiştir. Borik asit katkısının %1 oranına kadar basınç dayanımında artışa neden olurken, %1,5, %2 ve %2,5 oranlarında ise azalmaya neden olduğu görülmüştür. SEM görüntülerinde ise borik asidin zamanla C-H'ı kendine bağlayarak C-S-H jellerinin oluşumunu hızlandırdığı ve bunun etkisiyle zamanla basınç dayanımında artış sağlandığı görülmüştür. C-S-H jeli oluşumu yapının daha geçirimsiz ve dayanıklı olması sağladığı tespit edilmiştir. Ayrıca borik asit malzemedeki gözenekli yapının azalmasına neden olmuştur.

1. INTRODUCTION

Concrete is the bearing and protection element that is the mostly needed and used, can be adjusted for any building design, is cheap, most frequently used in the world [1-3].

It being easy to produce, long-life and cheap, as well as having high strength against loads carries the concrete in top among building materials [4]. In order to meet the sector needs and in parallel with technological progress, developments have occurred in concrete industry [5].

New concrete types have come out in order to develop concrete, eliminate the problems, and produce new properties and functional concretes [6]. One of the concretes which has come out as result of the developments in concrete industry is SCC. SCC as a type of special concrete, due to its own weight, can easily slide and flow inside different parts of the formwork and generate a remarkable consolidation within the targeted formwork [7]. It does not need any external and internal vibration and leaves no defects as the result of bleeding and segregation [8]. Recently, self-compacted concrete is used widely, due to its special properties [9]. It has been aimed to traditionalize SCC rather than its advantages, and significant findings have been obtained and important progress has been made in SCC development [10-17].

Being used as admixture for concrete, boron element has considerable reserve in Turkey and is one of the important underground sources. 803 million tons boron reserve of known boron mineral deposits in the world are situated in the west of Turkey [18,19]. Boron is used in many fields, especially in ceramic and glass industry [20]. In construction industry, it is still intensely used in cement, concrete and brick field [21-25].

This study has created a reference sample which meets the criteria of being self-compacting concrete. 6 different self-compacting concrete was obtained by adding 0.5%, 0.1%, 1.50%, 2.0%, 2.5% boric acid (H3BO3) according to the percentage by weight of water without changing W/C ratio of created reference samples Flow table test, T-50 test and V shape box, L shape box and U shape test were performed in order to examine the properties of freshly mixed concrete and compare SCC criteria on these samples. Compressive strength and 3-point loading tests were performed in order to determine compressive strength and flexural strength from hardened concrete tests. It was subjected to SEM and XRD tests in order to evaluate its structural characterization.
In the literature research, it is seen that the additives used in KYB production are mostly used as cement substitute material [26-32]. During the researches, no studies that are substitute of boron or derivatives to mix water in SCC production have been found. This aspect makes the study more specific and important.

2. MATERIAL AND METHOD

2.1. Material

CEM I 42.5 R class Portland Cement, boric acid, SAK, aggregate and water were used in this experimental study.

2.1.1. Cement

CEM I 42.5 R class Portland cement which is produced by Bartın Çimento San. A.Ş. was used in the production of SCC in experimental studies. This cement has been produced in conformity with TS EN 197-1 [33] by grinding clinker produced for Portland cement, and 5% minor addition component and gypsum together. Table 1 shows chemical analyses of cement which were used in the study.

Table 1. Chemical Analysis of Cement

Chemical Analyses %	
SiO_2	18.73
Al_2O_3	4.56
Fe_2O_3	3.07
CaO	63.91
MgO	2.08
SO_3	2.90
Ignition Loss	3.36
K_2O_3	0.62
Na2O	0.29
$(Na_2O);Na_2$+0,658*K_2O	0.70
Cl	0.0185
Non-measurable	0.46
Total	100
CaO (Free CaO)	0.56
Insoluble Residue	0.85

2.1.2. Boric acid

Boric Acid (H_3BO_3) which was used in production of Boron doped SCC in the studies was provided from Sigma Aldrich company. Physical and chemical properties are given in Table 2. Boric Acid (H_3BO_3) which is one of the boron compounds has white granular structure.

Table 2. Physical and Chemical Analysis of Cement

Physical and Chemical Analyses	
Molecular Weight (g/mol)	61.83
Degree of Purity (%)	99
Boron amount (%) (B)	17.3
Boiling point (°C)	300
Melting point (°C)	169
pH Range	5.2
Density (g/cm^3)	1.435
Resolution	Partially Soluble in Water
Physical Appearance	Crystal
Clour	White

2.1.3. Aggregate

Aggregate which was used in the production of SCC in the experimental studies was obtained from Çankırı province Ilgaz district Musaköy stone pit. Pit field constitutes permian crystallized limestone which is observed within Karakaya Formation in around of work site. “Monoliths” whose heights can vary from a few meters to over a few kilometres are called as “Limestone Member” belonging to Karakaya Melange. Crystallized limestones constituting pit area are white, grey, beige and has fissured, close-joints, solid and sturdy structure. Its cracks are filled with clay.

2.1.4. Chemical additives

Polycarboxylic ether based super plasticizer concrete admixture Master Glenium SKY608 which was obtained from BASF was used in production of SCC in the experimental studies. This admixture is being used in production of SCC and it is suggested to be used as around 0.8 – 1.5 kg for 100 kg (binder) by weight in SCC mixtures.

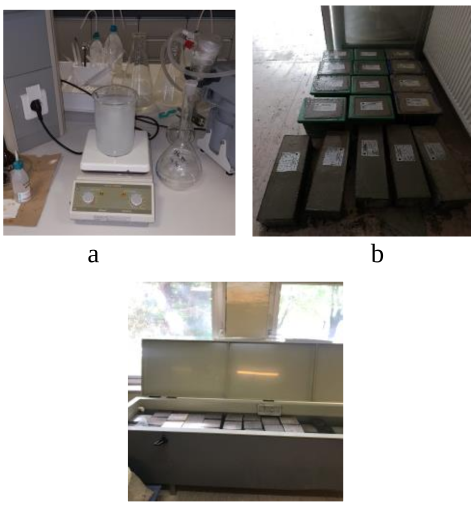
2.1.5. Water

Water which is used in production of SCC in the experimental studies was obtained from Kastamonu drinking water which has no organic substance and mineral salts.

2.2. Method

2.2.1. Production of SCC samples

Firstly, boric acid was transformed into solution at 80oC (Figure 1a). Then, aggregate, cement, water, boric acid solution and admixture which constitute the concrete were mixed through a mixer until it became homogeneous mixture. Produced concrete mixture was casted in cubic sample molds having 150x150x150 dimensions and girder sample molds having100x100x400 dimensions (Figure 1b). After being waited in the molds for 24 hours, SCC samples were removed out of mold and taken to curing pool (Figure 1c). Samples which were cured in curing pool for 28 days was taken out from curing pool, and compressive strength and flexural strength tests were applied to the samples in order to determine mechanical properties of them.



a b c

Figure 1. a) Preparation of boric acid solution, b) Pouring the mixture into the molds, c) Curing samples

Tests performed on SCC samples were carried out in the RE&DE Laboratory of the 15th Regional Directorate of Highways, and SEM and XRD measurements were carried out in Kastamonu University Central Research Laboratory.

2.2.2. Tests applied to samples

Freshly mixed (flow table test, V shape box test, U shape box test, L shape box test) and hardened concrete (compressive strength test, flexural strength test) on produced SCC samples. Also, SEM and XRD measurements were performed in order to determine structural characterization of it.

3. FINDINGS AND DISCUSSION

3.1. Results of Freshly Mixed Concrete Tests

Flow table test, V shape box test, U shape box test, L shape box tests were performed on prepared fresh concrete grout. The slump flow and T50cm is a test to evaluate the deformability, flow-ability and flow velocity of the self-consolidating concrete in the absence of obstacles [34]. Table 3 shows SCC threshold values, Table 4 shows test results of fresh concrete of SCC produced within the scope of the study. Boric Acid admixture was indicated as BAD” in Table.

Table 3. SCC threshold values

Testing Method	Property to be Measured	Unit	Threshold Value	
			Min.	Max.
Flow in Flow Table	Filling Ability	mm	650	800
Filling Box	Passing Ability	%	90	100
Time for first 50 cm in Flow Table	Filling Ability	sec	2	5
V Funnel	Filling Ability Resistance to Decomposition	sec	6	12
U Shape Box Rise Amount Measurement (H)	Passing Ability	mm	30	-

Table 4. Results of freshly mixed concrete tests

	Results of Flow Table Test (mm)	V Shape Box Test Results and T-50 Time		U-shape box Test Results (Height Diff. H2-H1)	L-shape box Test Results (Height Diff. PA= H_2/H_1)
		T-50 Time (Sec)	V Sha. Box Test Res. (Sec)		
REF	780	2< T50<4	10	6	1
%0.5 BAD	730	2< T50<4	13	7	1
%1.0 BAD	720	2< T50<4	14	7	1
%1.5 BAD	720	2< T50<4	15	9	1
%2.0 BAD	700	2< T50<4	15	9.3	1
%2.5 BAD	670	2< T50<4	17	12.5	1

When flow table test results have been examined, it has been seen that reference sample without boric acid dope is in SF3 class in terms of followability according to Efnarc.

It has been observed that flow amount of boric acid doped samples has decreased in parallel with the increase in boric acid addition. However, all test samples fulfil the SCC properties according to Efnarc [35] when flow table test results have been examined, and enter SF-2 class.

When V shape box test results have been examined, it has been seen that Reference number and all boric acid doped samples fulfil the characteristic of being SCC according to Efnarc and T-50 in terms of V shape box test results. It has been observed that V shape box flow time of boric acid doped samples has decreased in parallel with the increase in boric acid addition and T-50 time has increased generally even increase slightly in 1-2 and 2-3.

When U shape box test results; and reference sample and other boric acid doped samples have been examined, it has been seen that reference sample has the best passing ability and its passing ability has decreased in parallel with boric acid addition increase.

When L shape box test results have been examined, it has been seen that PA=(H2/H1) ratio of reference sample and all boric acid doped samples was 1, and no height difference has been occurred in L shape box test apparatus. However, time passed for closing the height difference has increased in parallel with the increase in boric acid addition. So, reference sample has completed height difference within shorter time and shown fast settlement. According to Efnarc, these test results was PA > 0.8 and sample class was PA2 because the test was performed in 3 reinforced L shape box.

3.2. Results of Hardened Concrete Tests

3.2.1. Results of compressive strength test

Compressive strengths on cubic samples in 150x150x150 (mm) was measured for 7, 28 and 90 days and recorded. The obtained data has been transformed into graphic in Figure 2.

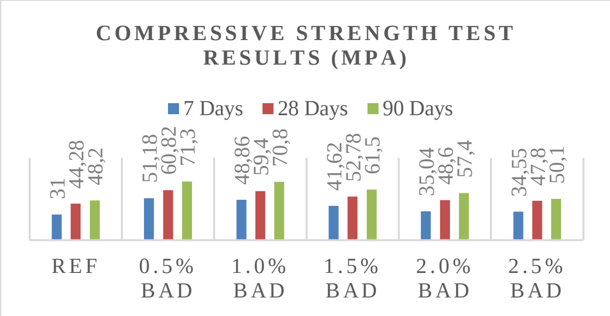


Figure 2. Results of compressive strength test

When test results have been examined, it has been seen that reference sample had the lowest compressive strength. Compressive strength values of boric acid doped samples have shown decrease inversely proportional to the increase of boric acid addition. The highest compressive strength has been observed in boric acid doped test samples with 0.5% and 1%. It has been determined that compressive strength of samples for 7, 28 and 90 days supported each other.

When all samples have been examined, it has been seen that there was slight difference between 0.5% and 1.0% boric acid doped samples and the highest values was obtained with these samples. Compressive strength in reference sample and other 1.5%, 2.0% and 2.5% boric acid doped samples was increased flagrantly.

3.2.2. Results of flexural strength test

3 point bending test was performed to samples produced in 100x100x400 (mm) dimensions, their flexural strengths were measured for 28 days and recorded, and obtained data has been shown in graphic in Figure 3.

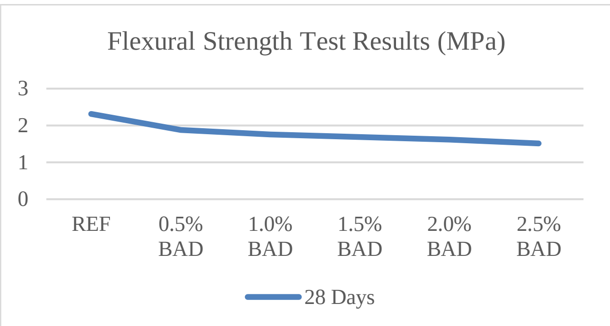


Figure 3. Results of flexural strength test

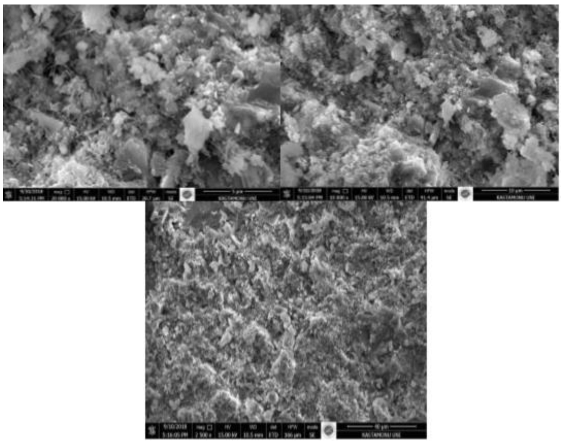
When test results have been examined, it has been seen that reference sample had the highest flexural strength. 0,5 % boric acid doped concrete had the highest flexural strength in boric acid doped samples. Flexural strength of samples has decreased in parallel with the increase in boric acid addition. The fact that reference sample having the highest flexural strength has made think that boric acid addition have a reducing effect on flexural strength of concrete.

3.3. Measurement Results Performed for Determination of Structural Characterization

3.3.1. Scanning electron microscope (SEM) results

Scanning Electron Microscope (SEM) analysis has been performed for interaction of coarse and fine aggregate and interfacial transition zone (ITZ) between cement. Shortly, all significant information on shape, texture, interface phase, pores and cracks of particles can be observed from SEM images of self-compacting concretes. SEM images have been taken from cracked samples after compressive strength. Performance of each concrete mixture can be associated with its micro structure. Different phases such as portlandite, calcite, ettringite and calcium silicate hydrate (C-S-H) which occur in concrete can be used for analysis in SEM images. Self-compacting concrete has a composite structure consisting of solid phase, pores and water. When SEM images have been examined, it has been seen that aggregate-putty interface had very intensive micro structure (Figure 4a). The main reason of this situation is linked to the behaviour of self-compacting concrete mix. It is known that interface transition areas between coarse aggregate and cement matrix has weak connection in normal vibrated concrete. It has been seen in 28-day SEM images that, C-H phases had crystallized, disappeared and gave its place to C-S-H gels.

Main factor on compressive strength is aggregate-matrix interface. Boric acid has been used up to 2.5% ratio and structure which cement linkage created around of aggregate has been examined. It has been seen that when hydration product, especially boric acid, was used in 0.5% and 1% ratio, it placed around of aggregate robustly (Figure 4b, Figure 5a). Also, it is seen that SEM image which was formed in the event of using boric acid in 0.5% and 1% ratio had tighter and voidless structure compared to control sample. Boric acid has linked C-H to itself over time and accelerated the formation of C-S-H gels and compressive strength has increased depending on time because of this situation. So, as well as a new C-S-H gel formation being obtained by adding boric acid as filling material, it has been ensured that the structure to become more impermeable and durable. Moreover, it has caused condensation in porous structure of micro structure and decrease in porosity (Figure 5b, Figure 6a). It has been seen that they also had C-S-H gel which was scattered equally and cause a compact and homogeneous structure compared to control mix. In the event of boric acid replacement in higher rates, voids and pores started to increase prominently in especially 2.5% boric acid replacement. Micro crack formation has also been observed (Figure 6b). Compressive strength increase has decreased according to control sample because of this situation.



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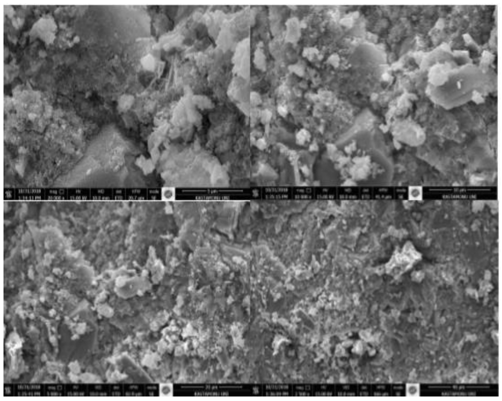
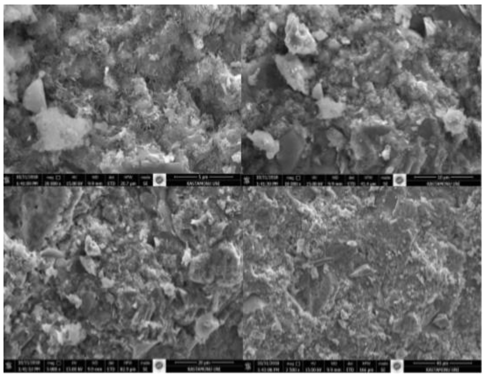


b

Figure 4. a) SEM images of reference sample, b) SEM images of 0.5% sample



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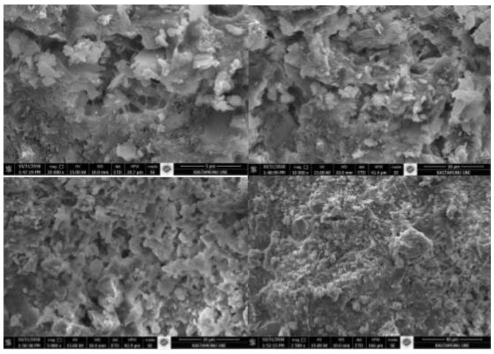
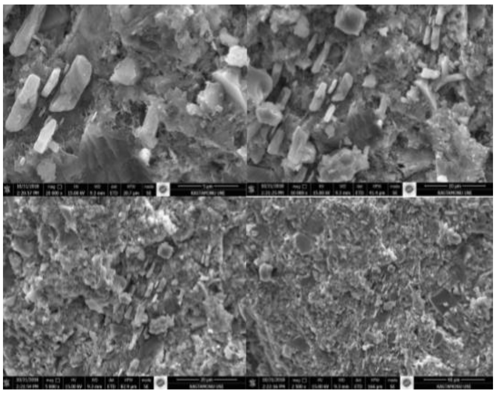


b

Figure 5. a) SEM images of 1.0% sample, b) SEM images of 1.50% sample



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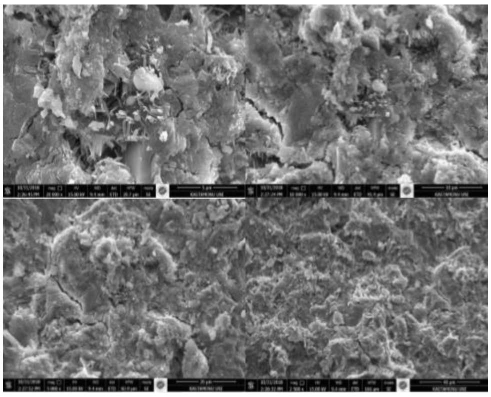

b

Figure 6. a) SEM images of 2.0% sample, b) SEM images of 2.5% sample

3.3.2. X-Ray diffraction (XRD) analyses

X-ray diffraction technique is used in order to determine crystallized structure and lattice parameters. In this technique which operates according to Bragg law, a new pattern is obtained by perceiving the reflection of sent beam over material.

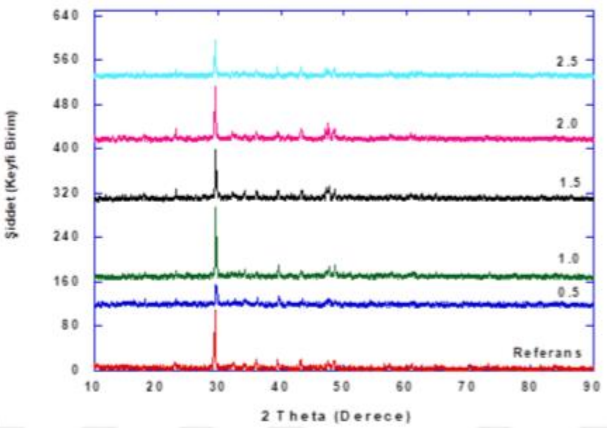

Figure 7. XRD graphic

When XRD graphic given in Figure 10 was examined, it has been seen that diffraction peak intensity has decreased in first boric acid additive (0.5%) when reference number and doped samples are compared, and intensity value has increased when boric acid amount has increased. Diffraction peaks are formed as a result of diffraction of X-ray in planes among the particles of sample. The increase and decrease of particle sizes change intensity values of these peaks as directly proportional. When XRD analyses of samples produced in this study have been examined, it has been seen that when boric acid additive has increased, dominance peaks observed in about 30o has significantly increased after especially 0.5% additive rate. However, no significant difference has been observed among diffraction peaks of 1%, 1.5%, 2% and 2.5% doped samples compared to reference sample. Addition has not changed the crystal structure. As it can be seen in Figure, diffraction peaks has occurred in the same angles. Different phase to cause a remarkable change has not been observed. This result is the indicator of that boric acid addition has penetrated into the structure to a large extent. Some additive atoms may also have been entered into particles. This is a factor which affect the porosity among particles. These results must also be supported with SEM analyses. When SEM analyses are examined, it is clear that morphological structure has not undergone an important change with the boric acid addition. When XRD peaks are examined, it is possible to associate the low peak intensity of 0.5% additive rate with the decrease in granule size. SEM image of 0.5% boric acid doped samples show that granules are smaller compared to other samples.

4. CONCLUSIONS

Results obtained from experimental study and observations are explained as items below.

– When fresh concrete results are assessed as a whole, all test samples have shown the characteristic of SCC. However, it is assessed that there has been decreases in flow together with effect of boric acid additive which was added to concrete and this would directly affect other criteria, the concrete would not meet criteria of being SCC with boric acid additive increase after a certain point.

– 0,5% boric acid doped sample has shown the highest strength in compressive strength test. Compressive strength of concrete has also decreased together with the increase of boric acid additive after 0,5%. Nevertheless, it is seen that boric acid additive has affected positively compressive strength of concrete.

– Reference sample has shown the highest strength in flexural strength test. Flexural strength of concrete has also decreased with boric acid addition. It is seen here that boric acid additive has affected flexural strength of concrete in decreasing way.

– When SEM images have been examined, boric acid has linked C-H to itself over time and accelerated the formation of C-S-H gels and compressive strength has increased in time because of this. So, a new C-S-H- gel formation has been obtained with boric acid addition and it has been ensured the structure to become more impermeable and durable. Voids and pores have started to increase prominently with 2.5% boric acid addition. Micro crack formation has also been observed. Compressive strength increase has decreased according to control sample because of this situation.

– In XRD analyses, when XRD peaks have been examined, it has been seen that when boric acid additive has increased, dominance peaks observed in about 30o has significantly increased after especially 0.5% additive rate. It is possible to associate the low peak intensity of 0.5% boric additive rate with the decrease in granule size and pores. SEM image of 0.5% boric acid doped samples shows that granules are smaller compared to other samples. SEM, XRD results supports the increase in compressive strength, especially in 0.5 boric acid doped concrete samples.

– When assessed generally, it has been seen that SCC production will be made by adding boric acid. It has been seen in SEM and XRD analyses that boric acid replacement has penetrated into concrete to a large extent.

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