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Resistance of High-Volume Fly Ash Self-Compacting Concrete to Internal Sulfate Attack

Abstract- *This paper investigates the durability of high-volume Fly ash self-compacting concrete, HVFASCC that exposed to internal sulfate attack. At the present work, HVFASCC was produced with two Fly ash replacements: 50 and 60% by weight of Portland cement. The internal sulfate attack was simulated by adding natural gypsum ($CaSO_4$) that contain ion (SO_3^{-2}) to fine aggregate by two weight percentages: 1 and 2%. Limestone dust was used as filler with a content of 100 kg/m^3. The cementitious materials, cement and Fly ash, content was 400 kg/m3 and the water to powder ratio for the studied mixes was 0.34 by weight. To ensure the self-compact ability of the mixes, slump flow, T500, V-funnel and L-box tests were done. The Compressive, Splitting and Flexural strength Tests were extended to the age of 240 days. The results showed that there is no significant difference between 1 and 2% of SO_3 content on the behavior of all mixes. The presence of limestone powder in the paste solution could have a role in stabilizing ettringite and reducing paste porosity at early ages. At later age, 240 days, the harmful effect of SO_3 is diminished and that may be caused by the depletion of gypsum and the dominant product will be calcium monosulfoluminate hydrates instead of calcium sulfoaluminate hydrates.*

Keywords- *High-volume Fly ash, Limestone, HVFASCC, Strength, Sulfates.*

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1. Introduction

Concrete is the most widely used material in the world. It plays an important role in infrastructure and private buildings construction. Self-compacting concrete, SCC, is special type of concrete which can be placed and compacted under its self-weight without vibration effort, and which has in the same time cohesive enough to be placed without segregation or bleeding. This type of concrete is used to facilitate and ensure proper filling and performance of the good structural of narrow areas and members congested reinforced concrete structure [1].

SCC is used in all elements of all civil engineering construction such as substructures, infrastructure, industrial floors and Installations that are regularly subjected to aggressive environmental condition [2].

The Brundtland Report defined sustainable development as the ability of humanity to ensure that it meets the needs of the present without compromising the ability of future generations to meet their own needs [3].

Three components of sustainability are environment, economy and society. These components must be maintained healthy and balanced simultaneously to keep on the entire of planet now and in the future [4].

Today, the use of Pozzolanic materials, such as Fly ash can reduce the use of manufactured Portland cement, and make concrete more durable [5].

Fly ash reacts with calcium hydroxide, a by-product compound of cement hydration, to produce calcium silicate hydrate, which consider desirable cementations product of cement hydration. This process leads to increase strength at later ages, reduces heat of hydration, and reduces the rate of concrete free shrinkage, resulting in a decrease in thermal shrinkage cracking due to the reduction in cement content. In addition, improves resistance of concrete against sulfate and alkalis-silica reaction and permeability.

[7] Mentioned that internal sulfate attack takes place because of the reaction between sulfates found in concrete ingredients, water, cement,

sand and gravel, with calcium aluminates, C3A, and water to form calcium sulphoaluminate, which causes high tensile stresses, lead to expansion and disruption of concrete.

The more common form of internal sulfate attack is the existence of CaSO4 that interacts either with C3A or calcium sulphoaluminate, $C_6A\bar{S}_3H_{32}$, with Ca(OH)2 and water to form ettringite which has the form of hard- rod crystals and large volume lead to generate interior expansion and tensile stress [8].

If the cement was substituted by high-volume mineral admixture, such as Fly ash, the amount of produced ettringite will be reduced and effected by the insufficient portlandite $Ca(OH)_2$. The development of expansive will be limited. In additional at the late stage although that CaOH2 is available in abundant amount but the hydration of mineral admixture need to consume partial of Ca(OH)2 and the development of expansive effect would be restrained again [8].

Borosnyói [9] stated that durability of concrete is considerably improved by the incorporation of supplementary cementing materials (SCMs). Due to the pozzolanic activity and the filling effect, use of SCMs can produce high performance concrete having both enhanced mechanical characteristics and reduced permeability that leads to improved durability.

Crouch [10] pointed out that using High Volume Fly ASH (HVFA) in concrete gives a wide range of benefits, but the most attractive is durability. It was studied four mixes, two with Fly ash, 25% class C and 20% class F, respectively and two other mixes with HVFA, 50% class C and 50% class F, respectively. The mechanical and physical properties of these mixes were investigated and found that compressive strength for mix with HVFA class C ash gave in early age similar compressive strength to mix with Fly ash 25% but in late age, the strength of HVFA mix was greater. Also regarding class F ash, the mix with HVFA gave lower strength than mix with 20% class F ash in early age but the strength become similar in later age. Regarding the absorption and permeability, the mixes with HVFA had always better results than mixes with lower Fly ash contents in all ages.

This investigation deals with effect the internal sulfates that are presented in fine aggregate on properties of self- compacting concrete containing high-volume Fly ash, 50 and 60 % by weight of cement.

This investigation could be considered as an implementation of sustainability requirements. Using (HVFASCC) with more than 50% replacement of Portland cement will serve sustainability by:

1. Reducing the Portland cement content in concrete is a crucial factor in reducing the CO2 gas emissions and therefore affects the global warming.
2. Using high quantity of Fly ash, which is a by-product material from electric power plant in many of countries, will enhance the waste management systems in the globe.
3. Making use of Fly ash plays an important role in improving many of properties of concrete and also improves durability that linked to the concept of sustainability.

2. Experimental Work

Materials

I. Cement

Iraqi ordinary Portland cement with commercial name Karasta was used. The test results showed that this cement conforms to requirement of European standard EN 197- 1:2011 [11]. The physical and chemical properties of this cement are given in Tables 1 and 2 respectively.

Table 1: Physical properties of cement tested by National Center for Structural Laboratories

No	Physical properties	OPC	Limits of EN 197-1:201111]
1	Specific surface area (Blaine method) m^2/kg	376	> 230
2	Setting time, hrs: min Initial setting Final setting	 2:05 4:00	 > 45mins ≤ 10hrs
3	Soundness(autoclave method), %	0.12	<0.8
4	Compressive strength ,MPa, at age 3 days 7 days	 20 25	 ≥ 15 ≥ 23

*OPC: Ordinary Portland cement

Table 2: chemical composition of the cement

No.	Properties		Test result	Limits of IQS No.5 /1984 for (OPC) [11]
1	Oxide content %	CaO	66.11	-
		SiO_2	21.93	-
		Al_2O_3	4.98	-
		Fe_2O_3	3.1	-
		MgO	2.0	<5
		SO_3	2.25	<2.8
2	Loss on ignition, LOI		2.39	<4
3	Lime saturation factor, IR		0.93	0.66-1.02
4	Insoluble residue, LSF		1.29	<1.5
5	Compound composition (bouge's equation),%	C_3S	64.57	-
		C_2S	14.15	-
		C_3A	7.95	>3.5
		C_4AF	9.43	-

II. Fine aggregate

Al Ukhaidher sand from quarries of karbalaa region was used as fine aggregate. The gradation and sulfate content were tested and were conforming to the Iraqi Standard No.45/1984 – Zone 2 [12] as shown in Table 3.

III. Coarse aggregate

Crushed natural gravel from Nibaae region locates north of Baghdad (Iraq) with maximum size of 20 mm was used in this work. Table 4 represent list the properties of this aggregate and conformed to requirement of Iraqi slandered.

Table 3: Properties of fine aggregate

Sieve size (mm)	Percentage passing	Limits of IQS No.45 /1984 – zone 2
10	100	100
4.75	95	90-100
2.36	79	75-100
1.18	59	55-90
0.6	39	35-59
0.3	15	8-30
0.15	6	0-10
Sulfate content, %	0.103	≤ 0.5
Fine materials passing from sieve (75 µm), %	3.9	≤ 5
Specific gravity, SSD	2.65	-
Fineness modulus %	3.07	-

Table 4: grading of the coarse aggregate

Sieve size (mm)	Percentage passing %	Limits of IQS No.45 /1984 (5-20 mm) %
37.5	100	100
20	100	95-100
14	-	-
10	40	30-60
5	10	0-10
2.36	0	-
Sulfate content as a SO_3%	0.08	0.1
Specific gravity, SSD	2.7	

IV. Natural gypsum

Natural gypsum was used from Salah al-Deen region. It was got as a rock, then crushed by hummer and sieved on sieves to gradation to be filled with that of fine aggregate. The percentage of sulfate, as SO_3 in gypsum was 42%. The following equation [13] was adopted in calculating the required quantity of gypsum to be added to the fine aggregate:

$$W = (R-X)S/N \quad \ldots \quad (1)$$

Where:

W: the weight of added gypsum, kg.

R: the desired percentage of SO_3.

X: the actual percentage of SO_3 in fine aggregate (0.1 %).

S: the weight of fine aggregate in the mix, kg.

N: percentage of SO_3 in gypsum (42%).

Gypsum was used as partial replacement by weight of sand by two percentages, 1 and 2%, to simulate the sulfate contamination in natural sand.

V. Fly ash

The used Fly ash is meeting the requirements of ASTM C 618 (Class F) [14]. It has a specific surface area of 730 m^2/kg; the characteristics of Fly ash used herein are presented in Table 5 and 6.

Table 5: Physical properties of used Fly ash class F

No	Physical properties	result	Limits of ASTM C 618-03[14]
1	Specific surface area (Blaine method) m^2/kg	773	-
2	Specific gravity	2.33	-
3	Amount retained when wet-sieved on 45 µm (No. 325) sieve %	15.1	<34
4	Compressive strength activity index,MPa, at age		
	28 days 90 days	84.4 96.9	≥ 75 ≥ 85

Table 6: Chemical properties of used Fly ash class F

parameter		average	Limits of ASTM C 618-03[14]
Loss on ignition %		3.1	≤6
Sulfate(SO3) %		0.4	≤5
Compound composition %	SiO2	65.65	Total ≥ 70%
	Fe2O3	5.98	
	Al2O3	17.69	
	MgO	0.72	-
	CaO	0.96	-
	Na2O	1.35	-
	K2O	2.99	-

VI. Limestone powder

In this work, crushed limestone brought from local market is used and the fineness of the gained material is (239 m2/kg).The average particle size of the limestone powder according EFNARC requirement[15] may be less than 0.125mm to achieve its most benefits. The limestone powder passing sieve No. 0.125 mm is used in this work. The fine limestone powder (commercially named as Al-Gubra) was from of Al Anbar region.

Table 7: Chemical Analysis of the Limestone Powder

No.	test		result
1	SO3 percent		0.77
2	Specific surface area (Blaine method) m^2/kg		239
3	Compound composition %	CaO	56.1
		SiO2	1.38
		Fe2O3	0.12
		Al2O3	0.72
		MgO	0.13
		L.O.I	4.56

VII. High – range water-reducing admixture (super plasticizer)

A high –range water reducing admixture, the commercial name is GLENIUM 54 from BASF Company was used in mixes. It is a third generation of super plasticizer those based on sulphonated melamine and naphthalene.it is based on a unique carboxylic ether polymer with a long lateral chains. It has relative density of 1.07 kg/l at 20ºC according to brochure of company. The normal dosage for GLENIUM 54 is between 0.5 and 1.6 lit/100kg of cement (cementitious material) and dosage outside this range is permissible and is subjected to trail mixes. It was found that the optimum dosage is 1.1 - 1.35 lit/100kg of cement and Fly ash contents.

Experimental program

Two mixes (R50 and R60) were used as references in this work, to achieve 30MPa as characteristic compressive strength at 28 days for (100*100*100 mm) cubes depending on reference [15]. The powder content was 500 kg/m^3 and the water to powder, (W/P), ratio is 0.34 by weight (Table 8):

1. Mix R50 with 50% Fly ash replacement by weight of cement.
2. Mix R60 with 60% Fly ash replacement by weight of cement.

The R50 mix was reproduced as mixes R50S1 and R50S2 with two sulfate contents of sand, 1 and 2% respectively and the same was done with mix R60 to produce R60S1 and R60S2. Therefore, the total number of mixes was six. All these mixes were water-cured till the age of test.

Table 8: The details of the reference mixes

Type of mixes	powder material content		Aggregate			Water Kg/ m3	w/powder ratio (W/P)	Super plasticizer L / m3
	Cement Kg/m3	Fly ash Kg/m3	Limestone powder	F.A.* Kg/m3	C.A.** Kg/m3			
Reference Fly ash 50%	200	200	100	840	800	170	0.34	5.25
Reference Fly ash 60%	160	24	100	840	800	170	0.34	5.4

* F.A.: Fine Aggregate
** C.A.: Coarse Aggregate

Testing Program

Fresh state tests

To verify SCC fresh characteristics, the following tests were carried out according to the methods that are given in the Guidelines of EFNARC [16]:

1. Slump flow and T50cm.
2. V-funnel and V-funnel at T5minutes.
3. L-box.

Hardened state tests

Table 9 lists the conducted tests, the adopted standards, types and dimensions of tested specimen and age of test for the hardened HVFASCC mixes.

Table 9: Hardened state tests were tested in concrete laboratory / Technology University

No.	Test	Adopted standards	Specimen type	Dimensions, mm	Age of test, days
1	Compressive strength	BS EN 12390-3:2002 [17]	Cube	100*100*100	14, 28, 90, 180, and 240
2	Splitting tensile strength	ASTM C496-04 [18]	Cylinder	d=100, h= 200	14, 28, 90,180 and 210
3	Flexural strength	ASTM C 78-04[19].	Prism	100*100*400	14, 28, 90 and 180

4. Results and Discussion

Results of Fresh HVFASCC:

Five tests were carried out; they are slump flow, T50cm, V-funnel and L-Box for R50 and R60 only. The added gypsum did not have any detrimental effect on the properties of fresh SCC reference mixes. Therefore, Table 10 shows the results of only mixes R50 and R60. All listed test results were conforming to the requirements of the EFNARC [16].

Table 10: Fresh HVFASCC test results

mixes	Slump flow	T50cm	V-funnel	V-funnel after 5 minutes	L-box
	mm	Sec.	Sec.	Sec.	(h2/h1)
R50	750	3	11	14	0.9
R60	760	3	8	11	0.95
EFNARC Requirements 2002	600-800	2-5	6-12	+3	0.8-1

Strength Development Results of HVFASCC: Table 11 summarizes the all-types strength development for all mixes with different ingredients and at different ages.

Table 11: Strength development for the studied mixes

Mix	SO3 in fine aggregate, %	Compressive strength, MPa 14	28	90	180	240	Splitting tensile strength, MPa 14	28	90	180	210	Flexural strength, MPa 14	28	90	180
R50	0.1	39.3	46.5	56.0	57.3	63.0	3.3	3.6	4.2	4.5	5.2	3.5	4.0	4.7	5.4
R50S1	1.0	38.7	43.7	46.0	50.0	61.0	3.2	3.4	3.7	4.1	4.7	3.4	3.7	4.6	5.2
R50S2	2.0	38.5	42.6	45.2	46.9	60.7	3.15	3.3	3.7	4.0	4.6	3.4	3.7	4.5	5.1
R60	0.1	38.4	41.2	52.1	54.0	60.0	3.2	3.5	4.1	4.3	4.9	3.5	3.8	4.5	5.2
R60S1	1.0	38	41.1	45.0	47.2	59.0	3.15	3.3	3.6	3.9	4.5	3.4	3.6	4.4	5.1
R60S2	2.0	37.8	39.8	45.0	47.8	58.3	3.1	3.2	3.5	3.8	4.3	3.35	3.7	4.2	5.0

The R50 mix yielded the highest compressive strength at all ages and mix R60 was next to that. These two mixes have almost the same rate of strength gain but R60 has lower strength values at the same age due to the fact that decreasing cement content in mix R60 would decrease the production of Ca(OH)2 which is needed for the Fly ash to react with and building new C-S-H gel [6].

The effect of SO3 content in fine aggregate on compressive strength development is shown in Figures 1 and 2 for the studied mixes. It could be observed that there is no significant difference between 1 and 2% of SO3 content on the behavior of all mixes. On the other hand, increasing the SO3 from 0.1 to 1% has caused a reliable drop in the compressive strength especially at intermediate ages, 28, 90 and 180 days. At early age, 14 days, the reduction in strength is very little and that could be attributed to the long setting time of concrete that resulted from using high contents of Fly ash. The delayed setting makes the paste more flexible to accommodate expected expansions. In addition to that, the presence of limestone powder in the paste solution could have a role in stabilizing ettringite and reducing paste porosity [20].

At later age, 240 days, the harmful effect of SO3 is diminished and that may be caused by the depletion of gypsum and the dominant product will be calcium monosulfoluminate hydrates instead of calcium sulfoaluminate hydrates [21].

For the studied mixes, the development of splitting tensile and flexural strengths was similar to that of compressive strength.

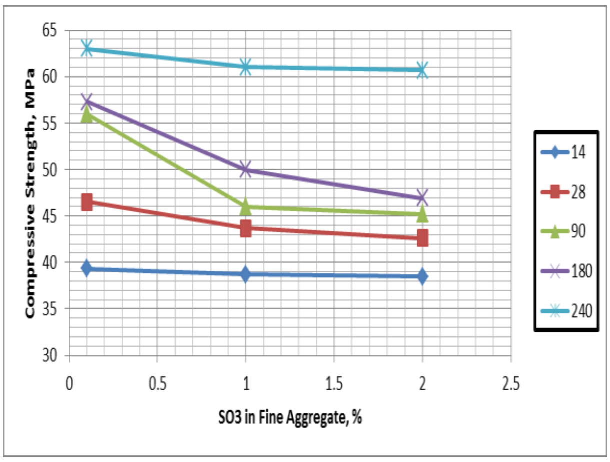

Figure 1: The effect of SO3 content in fine aggregate on the development of compressive strength through days (14, 28, 90, 180, and 240 day) for mixes R50, R50S1 and R50S2.

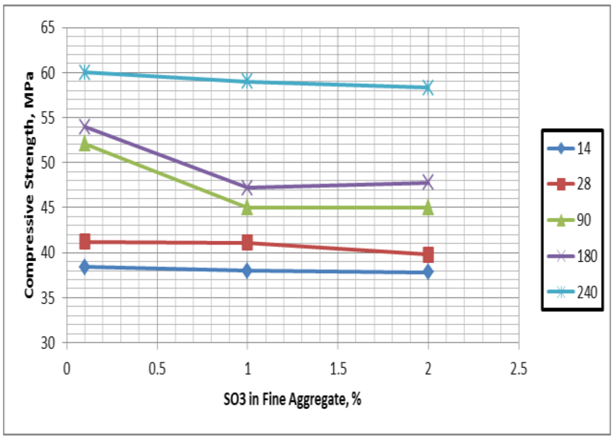

Figure 2: The effect of SO3 content in fine aggregate on the development of compressive strength through days (14, 28, 90, 180, and 240 day) for mixes R60, R60S1 and R60S2

Conclusions

The following conclusions could be drawn based on the present experimental results:

1. A self-compacting concrete could be produced by high-volume replacements of Portland cement by Fly ash. At the present work the replacements were 50 and 60% by weight of Portland cement and yet the fresh state tests of the studied HVFASCC mixes were conforming to the requirements of the international standards.
2. There is no significant difference between 1 and 2% of SO3 content on which behavior of all mixes.
3. At early age, 14 days, the reduction in strength is zero and that could be attributed to the long setting time of concrete due to replacement by high contents of Fly ash. The delayed setting makes the paste more flexible to accommodate expected expansions.
4. The presence of limestone powder in the paste solution could have a role in stabilizing ettringite and reducing paste porosity at early ages.
5. At later age, 240 days, the harmful effect of SO3 is diminished and that may be caused by the depletion of gypsum and the dominant product will be calcium monosulfoluminate hydrates instead of calcium sulfoaluminate hydrates.

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