Resistance of Self Compacting Concrete to Internal Sulphates Attack

Jinan Jawad Hassan Alwash
Babylon University / College of Engineering

Abstract

The objective of this work is to study the resistance of SCC concrete to internal sulphates attack. The self compacting concrete is made by using a local filler (pigment and metakaolin). A mix of self-compacting concrete was designed according to special specifications and trials. This mix was tested by various fresh concrete workability tests as Slump-flow, T 50cm, L.box, U.box, and V.funnel tests.

Five levels of SO_3 content in (fine aggregate) sand were investigated , these levels were about [0.24% , 0.5% , 1% , 2% , and 3%]. Which yield [3.05% , 3.47% , 4.28 % , 5.9% , and 7.52%] total SO_3 content by weight of cement respectively for SCC mixes with pigment powder filler and [3.08% , 3.5% , 4.31% , 5.94% , and 7.56%] for SCC mixes with metakaolin powder .

The experimental results show that there is an optimum gypsum content in sand (SO_3 = 1.0%) by wt. of sand which gives the highest results in compressive strength, flexural strength and ultrasonic pulse velocity of SCC . As gypsum content increases beyond this limit the above mechanical properties will be decreased.

الخلاصة

الهدف من هذا العمل هو دراسة مقاومة الخرسانة ذاتية الرص لهجوم أملاح الكبريتات الداخلية. الخرسانة ذاتية الرص تحتوي على مواد مالئة متوفرة محليا (الأصباغ والميتاكاؤولين). تم تصميم خرسانة ذاتية الرص إستنادا لمواصفات خاصة مع التجريب .هذه الخلطة الخرسانية الطرية جرى فحصها بفحوصات متعددة لقابلية التشغيل مثل: إنسياب المخروط و زمن الإنسياب ، الصندوق على شكل L ، الصندوق على شكل U ، والقمع على شكل V.

محتوى الكبريتات في الرمل كان (0,24% ، 0,5 % ، 1% ، 2 % ، 3 %) التي قابلت نسبة كبريتات كلية في الخلطة من وزن السمنت تساوي (3,05 % ، 3,47 % ، 4,28 % ، 5,9 % ، 7,52 %) في الخلطات الخرسانية ذاتية الرص الحاوية على الأصباغ و (3,08 % ، 3,5 % ، 4,31 % ، 5,94 % ، 7,56 %) في الخلطات الخرسانية ذاتية الرص الحاوية على الميتاكاؤولين.

أظهرت النتائج أنه توجد نسبة مثلى لمحتوى الجبس للخرسانة ذاتية الرص مقداره (1%) من وزن الرمل والتي تعطي أعلى النتائج لمقاومة الإنضغاط ومقاومة الانثناء وسرعة الأمواج فوق الصوتية. وإذا تعدت نسبة الجبس في الرمل عن هذه النسبة فإن النتائج أظهرت إنخفاضا في الخصائص الميكانيكية أعلاه.

Introduction

Self – Compacting Concrete

Self-compacting concrete (SCC)-as the name implies-is the concrete requiring a very little or no vibration to fill the form homogeneously. SCC is defined by two primary properties: Deformability and Segregation resistance. Deformability or flow ability is the ability of SCC to flow or deform under its own weight (with or without obstructions). Segregation resistance or stability is the ability to remain homogeneous while doing so. High range water reducing admixtures are utilized to develop sufficient deformability. At the same time, segregation resistance is ensured, which is accomplished either by introducing a chemical viscosity modifying admixture or by increasing the amount of fines in the concrete **[Collepardi. M 2000, Memon. S. 2008].**

Studies to develop SCC, including a fundamental study on the workability of concrete have been carried out by Okamura and Ouchi [**Okamura 1998, Okamura and Ouchi: 1999**].

[**Su. K. et al 2002**] , studied the effect of sand ratio (S/A = fine aggregate volume to total aggregate volume) on the elastic modulus of self compacting concrete. They used various S/A ratio concretes which were cast and tested then the modulus of elasticity of SCC was compared with the modulus of elasticity of normal concrete. Slump flow test, slump test and U.box test were carried out to evaluate concrete floablity .. They found that the flowability of SCC incresses with the increase in S/A. However the modulus of elasticity is not significantly affected by S/A when total aggregate volume was kept constant

[**Drich. H. et al 2000**], studied the effect of type different sands on workability of SCC; five natural and five artificial form sand were used, each pair having identical grading curves but different particle shape and surface texture. They found that increasing the fineness of sand particles leads to increasing yield stress and plastic viscosity and increasing the ratio (fine / total) leads to increasing yield stress and plastic viscosity. The influence of changing particle shape and surface texture by replacement of the artificial sand with the natural sands is not as easy to observes the artificial and natural sands to some extent also vary with regard to fineness.

Internal Sulphate Attack

Internal Sulphate attack is considered as very important problems of concrete manufacture in Iraq and middle east countries. This problem becomes more serious with time as it is difficult to find aggregate complying with the current specifications for sulphate limits

Many researchers have studied the effect of internal sulphate attack . They believed that the formation of the calcium sulphoaluminates in an early stages, when concrete is still plastic, expansion does not cause any appreciable disruption or reduction in strength, because the formation of the sulphoaluminates is not associated with the development of the internal stresses, due to fresh concrete plasticity, but internal stresses will develop due to the formation of calcium sulphoaluminates in the hardened concrete stage.

[**Neville 1995, and lea 1976**] , stated that gypsum from different interior sources (cement, fine aggregate, coarse aggregate) reacts with some components of cement specially tricalcium aluminate (C_3A). Such reaction produces calcium sulphoaluminate (Ettringite) ($3CaO.Al_2 O_3..3 CaSO_4..31 H_2O$)

The volume of this product is 277% of the volume of reactants. The hydration process of the calcium silicate yields calcium hydroxide which react with sulphate to produce gypsum. The volume of gypsum is 124% of the volume of reactants. These volumetric changes are accompanied by interior stresses which cause strength reduction and disintegration.

[**Abdul Latif, 1997**], concluded that each type of cement has certain behavior in relation to the increase of (SO_3) percent in sand and this behavior depends on number of factors such as SO_3 % in cement, fineness, C_3A content and soundness that specifies optimum gypsum content for each case. Since this would not agree with the limits specified in IQS 45 – 1984.

Experimental Work

Materials and Mixes

Cement

Ordinary Portland cement (OPC) manufactured by united cement company, commercially known [**TASLAUTA – BAZIAN**] was used throughout this investigation. This cement complied with the Iraqi specification No.5: 1984. The chemical composition and physical properties are presented in Tables (1) and (2).

Fine Aggregate (Sand)

Natural sand from Al-Akaidur region was used. The result of physical and chemical properties of the sand are listed in Table (3). Its grading is conformed to the IQS No.45: 1984 zone 3.

Coarse Aggregate (Gravel)

The coarse aggregate was Al-Nibaee gravel with a maximum size of 14 mm. The results of physical and chemical properties of the coarse aggregate are listed in Table (4). The coarse aggregate is complying with IQS No.45: 1984.

Table (1): Chemical composition of the cement

Oxides	**% by weight**	**Limits of Iraqi specification IQS No.5 / 1984**
CaO	62.05	-------
SiO_2	21.32	-------
Al_2O_3	5.72	-------
Fe_2O_3	3.52	-------
MgO	3.17	< = 5 %
SO_3	2.44	< = 2.5 % if C_3A < 5 % < = 2.8 % if C3A > 5 %
Free lime	1.06	
L.O.I	1.21	4 %≤
Compound composition	**% by weight of cement**	**Limits of Iraqi specification IQS No.5 / 1984**
C_3S	35.84	--------
C_2S	34.08	--------
C_3A	9.20	------
C_4AF	10.71	-----
L.S.F	0.86	------

Table (2): Physical properties of cement

Physical Properties	**Test Results**	**Limits of Iraqi Specification IQS No.5 / 1984**
Fineness, Blaine, cm^2/gm	300	≥ 2300
Setting time, vicat's method Initial hrs: min Final hrs: min	 2 : 20 3 : 55	 ≥ 00 : 45 ≤ 10 : 00
Compressive strength MPa 3 days 7 days	 19.0 27.0	 ≥ 15 : 00 ≥ 23 : 00

Table (3): Properties of fine aggregate

Sive size(mm)	Passing %	Limits of Iraqi Specification IQS No.45 / 1984 for zone (3)
9.5	100	100
4.75	96	90 – 100
2.36	85	85 – 100
1.18	78	75 – 100
0.6	62	60 - 79
0.3	30	12 – 40
0.15	4	0 -10
Properties	**Test Results**	**Limits of Iraqi Specification IQS No.45 / 1984**
SO_3 %	0.24	$\leq$ 0.5
Fineness Modulus	2.45	-----
Specific gravity	2.55	------
Absorption %	1.4	-----
Fine Materials %	0.95	$\leq$ 5

Table(4) : properties of coarse aggregate

Sieve Size (mm)	Passing %	Limits of Iraqi Specification IQS No.45 / 1984
14	100	90 - 100
10	51	50- 85
5	9	0-10
2.36	0	-------
Properties	**Results**	**Limits of Iraqi Specification IQS No.45 / 1984**
Specific gravity	2.65	------
SO_3 %	0.07	$\leq$ 0.1
Absorption %	0.60	------
Fine Materials %	0.0 %	$\leq$ 3 %

Gypsum

Gypsum is added to sand to change its SO_3 content. The added gypsum is natural gypsum rock (brought from kufa cement factory) which is crushed, sifted and graded. The gradation of the gypsum added was the same as that of sand because it has been found that the gypsum has the same gradation as the sand which includes [**Al-Kadhimi 1988**].

The following equation has been used to control the SO_3 levels in the used sand :
$W = (R – P) * S / N$ **[Mater 2000, Abdul Latif, 1997**].
Where:
W = weight of ground gypsum to be added to the mix in kg.
R = percentage SO_3 required in sand %.
P = percentage SO_3 percent originally in sand %.
S = weight of sand in the mix , kg.
N = percentage SO_3 in the used gypsum.
The chemical composition is listed in Table (5).

Table (5): The chemical properties of Gypsum

Compound composition	Percent
SiO_2	8.74
R_2O_3	2.12
CaO	31.88
MgO	0.97
SO_3	41.1
I.R	7.06

Super Plasticizer

To achieve high workability needed to produce SCC, super plasticizer known as Flocrete SP33 was used. According to ASTM C494 – 92, this SP is classified as type A and F. The technical description of the super-plasticizer is given in Table (6).

Table (6): Technical Description of the Super plasticizer

color	Brown Liquid
Freezing point	-2 C° approximate
Specific graving	1.18 kg / l @25 C°
Air entrainment	Less than 2 %
Chloride content	Nil

Pigment

This material is brought from local market, and then it is used in the concrete mixes after passing sieve size 0.075(mm). The chemical composition of the pigment is shown in Table (7).

Table (7): Chemical Analysis of pigment

Oxide	%
SiO_2	4.17
Fe_2O_3	0.10
Al_2O_3	0.50
CaO	63.08
MgO	0.12
SO_3	0.63
L.O.I	31.26

Metakaolin

Metacaolin is a highly pozzolanic material by calcining china clay using an oven at temperature of (700 – 900) C° for 0.5 hour **[BSEN 2002**]. Then the sample is left to cool. The particle passing sieve size (0.075 mm). The chemical composition of kaolin and metakaolin is shown in Table (8).

Table (8): Chemical Analysis of Kaolin and Metakaolin

Oxide	Kaolin %	Metakaolin
CaO	10.00	6.14
Al_2O_3	13.83	12.74
Fe_2O_3	1.12	3.52
SiO_2	52.27	68.84
MgO	6.50	6.16
L.O.I	14.06	1.40
SO_3	1.32	0.86

Mix Design and Proportions

The Japanese mix design procedure [**cited by EFNARC 2002**] was followed to design the mix proportions of SCC. Table (9) shows the mix proportions of the SCC mix used in the present study.

Table (9): Mix Proportions of the SCC Mix

Materials	Self-Compacting Concrete Mix
Cement (kg / m^3)	450
Filler (kg / m^3)	70
Fine aggregate (kg / m^3)	730
Coarse aggregate (kg / m^3)	800
SP (Liter / 100 kg cement)	2
W / P	0.47
W / C	0.54

Mixing, Casting and Curing

To acquire SCC mix maintaining its properties during the fresh state, the procedure of mixing the ten mixes with the proportions mentioned above is as follows:

1- The total amount of fine aggregate (sand with the added gypsum)and 1/3 of the mixing water were poured in the mixer and mixed for 1 minute .

2- The amounts of cement, filler(pigment or metakaolin) and 1/3 (water + SP) were added to the mixer and mixed for 30 seconds.

3- ½ of the gravel and 1/3 (water + SP) were added and mixed for another 30 seconds.

4- The remaining ½ of the gravel and the remaining 1/3 of SP were added and mixed for a final 1 minute.

Testing of fresh concrete was conducted on the produced SCC mix. Then, from the same mix the following samples were cast for mechanical properties testing of the hardened concrete:

- (150 * 150 * 150) mm cubes were cast for compressive strength and ultrasonic pulse velocity determination. They were tested at 28, 60 and 90 days age.
- (100 * 100 * 400)mm prisms were cast for flexural strength determination. They were tested at 28, 60 and 90 days age.

Test Producers

Fresh Tests

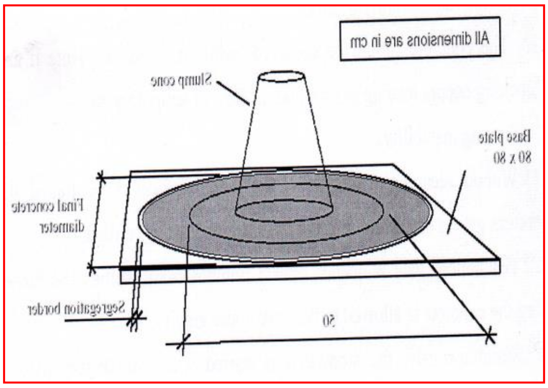
The workability of SCC can be characterized by the following properties:

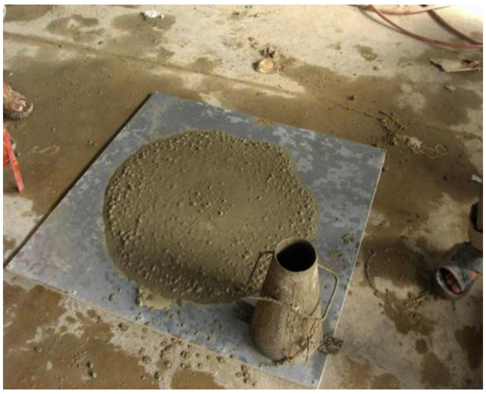
* Filling ability.
* Passing ability.
* stability .
* Segregation resistance.

In order to evaluate the filling, passing ability and segregation resistance of the fresh concrete, slump-flow, T50 cm, L.box , U box and V.funnel tests, as shown in Fig (1).**Hardened Concrete**

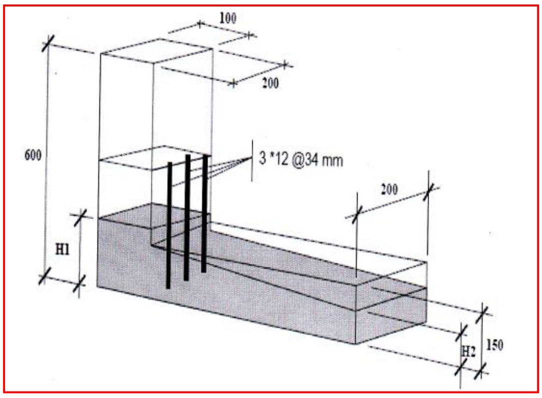
Compressive Strength

Compressive strength was carried out and tested according to B.S 1881: part 116: 1989. All specimens were cured in water until the age of testing (28, 60 and 90) days. Each result of compressive strength obtained is the average of three specimens.

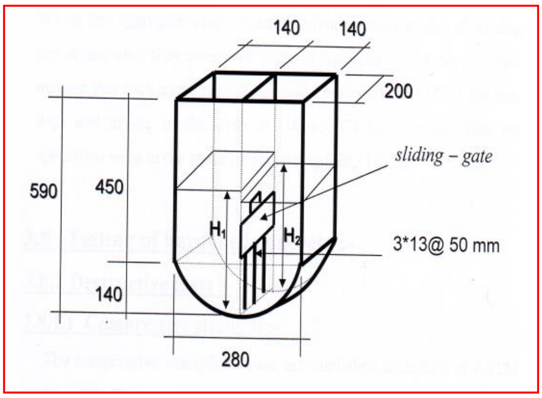




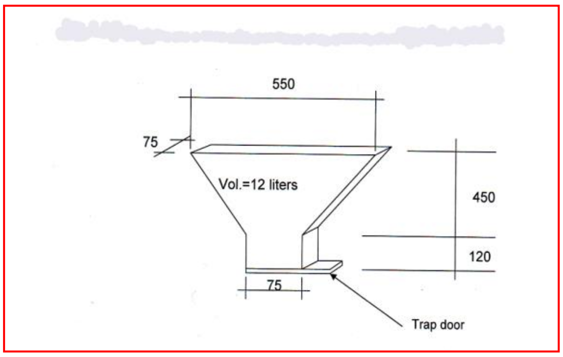
a. Slump – flow and T50



b. L – box test



c. U - box test



d. V- funnel test

Fig (1): Fresh concrete test

Flexural strength (Modulus of Rupture)

SCC prisms of dimensions (100 * 100 * 400)mm were cast and tested for flexural strength determination. This test was performed according to "BS 5328: part 4: 1990" specification. The concrete prisms were moist cured until the age of testing (28, 60 and 90) days. This test was performed using two-point load. Modulus of rupture (**R**) is calculated from the following equation.

$\mathbf{R} = PL / bd^2$

Where:

P = maximum resisted load, N

L = span length, mm

b = specimen width, mm

d = specimen depth, mm

Ultrasonic Pulse Velocity Test

The UPV method can be considered as one of the most promising methods for evaluation concrete structure. This method basically depends on the propagation of high frequency sound wave, which passes through the material by using portable equipment (PUNDIT), composed of the source / detector unit and the surface transducers, which work in the frequency range of 25 to 60 KHZ. The ultrasonic pulses depend on the density and elastic properties of material [**Lorenzi *et al* 2007].**

150 mm cubes were used to measure the propagation velocity of longitudinal stress wave pluses through concrete. The test was performed according to [**ASTM C597, 2002**], pulses of longitudinal stress wave are generated by electro-acoustical transducer that is held in contact with one surface of the concrete under test. After traversing through the concrete, the pulses are received and converted into electrical energy by a second transducer. The pulse velocity is given by the following equation:

Where:

V: pulse velocity, (km / sec)

L: Distance between center of transducer faces, (mm)

T: Transit time, (μ sec)

Results and Discussion

Fresh Concrete Test Results

Table (10) gives the experimental results obtained from slump flow, T50 cm, L-box, U-.box, and V-funnel tests that were conducted throughout the present work. It can be seen that the test results are within the limits of self-compacting concrete results established in EFNARC 2002 which means that the designed concrete mix in the present work conforms to the specifications of SCC. It can be noticed that the slum-flow of SCC mix with pigment is relatively higher than those containing metakaolin by (9.2 %) and T50 cm of SCC mix with pigment is lesser than metakaolin by (40 %). This high workability can be attributed to the dispersion of pigment powder throughout the concrete mix which can be obtained by using super plasticizer [**Neville, 1995**]. Also it can be seen that the SCC mix with metakaolin gives relatively less value of blocking ratio (H_2 / H_1) than the SCC mixes with pigment by (5.6 %). This behavior can be attributed to the relatively high viscosity of metakaolin compared to pigment, because the viscosity increased markedly with the increase of SiO_2 **[Lea, 1976**].

Table (10): Results of Workability Tests of the SCC

Workability Tests	**Unit**	**Type of Filler**		**EFNARC SCC Limits**
		*** MP**	*** Mm**	
Slump flow	mm	760	690	600 - 900
T50 cm	Sec	2.5	3.5	2 - 5
L-box	(H2 / H1)	0.95	0.90	0.8 - 1
U-box	(H1 – H2) mm	5	10	0 - 30
V-funnel	Sec	7.0	9.0	6 - 15

MP : Mixes of SCC with pigment.*
Mm : Mixes of SCC with metakaolin .*

Table (11) and Figs (2,3) present compressive strength test results for the SCC mixes with different SO_3 content in fine aggregate (sand). It can be seen that the optimum gypsum content of sand which gives highest values in compressive strength of SCC is at(SO_3 = 1) by weight of sand and the total gypsum content in concrete is (SO_3 = 4.28) by weight of cement for SCC mixes which contain pigment and at (SO_3 = 1) by weight of sand and the total gypsum content in concrete is (SO_3 = 4.31 by weigh of cement for SCC mixes which contain metakaolin .This limit of gypsum content yields maximum compressive strength at 28, 60, 90 days age. Then, increasing sulphate content in fine aggregate beyond this limit cause decrease in the compressive strength of SCC at three ages of testing. The percentage of decrease was about (5.4 %, 18.9 %) at 28 days age, (7.9 %, 23.8 %) at 60 days age and (8.4 %, 24.4 %) at 90 days age when SO_3 content in fine aggregate was increased from (0.24) to (2 % and 3 %) respectively for SCC contain pigment. and about (4.8 %, 14.6 %) at 28 days age, (5.2 %, 17.8 %) at 60 days age and (5.3 %, 20 %) at 90 days age when SO_3 content in fine aggregate was increase from (0.24) to (2 %and 3 %) respectively for SCC contain metakaolin This reflects an intensive deleterious effect of sulphate on compressive strength of SCC. It can be pointed out that at the high percentage of SO_3 in fine aggregate (2 and 3) the development of compressive strength almost vanished**.**

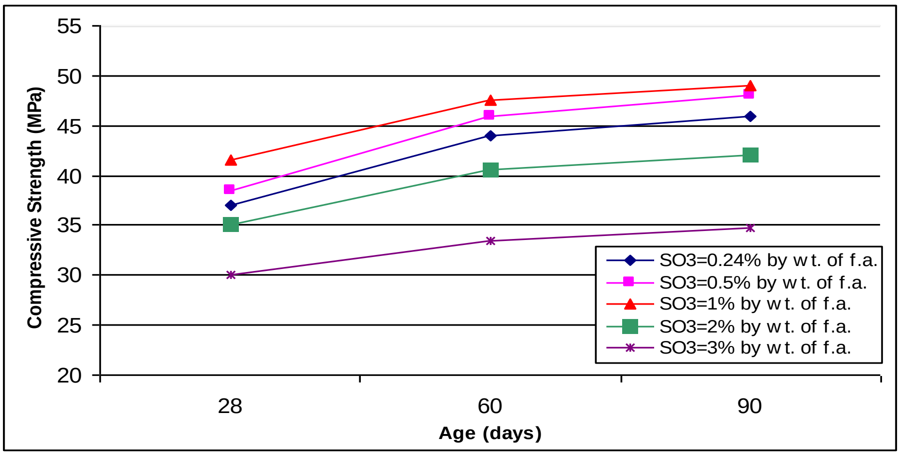
Table (12) and Figs (4,5) present the values of flexural strength at (28, 60, 90) days age for SCC mixes with different percentages of SO_3 in fine aggregate. It can be seen that with increasing the SO_3 content in fine aggregate from (0.24 to 0.5) the flexural strength increasing by about (2.06 %, 3.7 %, 2.7 %) at (28,60,90) days age respectively for SCC mixes contain pigment and by about (5.8 %, 2.6 %, 2.5 %) at (28, 60, 90) days age respectively for SCC mixes contain metakaolin. But when increasing the SO_3 content in fine aggregate from (0.24 to 1) the flexural strength increasing by about (9.5 %, 5.6 %, 6.3 %) at (28, 60, 90) days age respectively for SCC contain pigment and by about (8.8 %, 7.0 %, 5.8 %) at (28, 60, 90) days respectively for SCC mix contain metakaolin. This limit of SO_3 seems to be the same OGC in fine aggregate of SCC found from compressive strength results. Then as the gypsum content in fine aggregate increases beyond this, flexural strength decreases appreciably. It can be pointed out that the deleterious effect of sulphates in fine aggregate is more pronounced compared with their effect on compressive strength. This mechanical behavior can be explained by the intensive micro-cracking due to the delayed ettringite formation and the following progressive pronounced expansion when internal sulphate attack is acting by the continuous supplement of sulphate from sand **[Mater 2000**].

Results of the 28, 60 and 90 days ultrasonic pulse velocity of SCC with various percentages of gypsum content in sand are shown in Table (13) and Figs (6,7) for different SCC mixes. It is clear that the effect of sulphate on the ultrasonic pulse velocity is some what similar to that on compressive strength. For all mixes, there is an optimum gypsum content at which the (U.P.V) is maximum ,beyond this content the (U.P.V) has decreased.

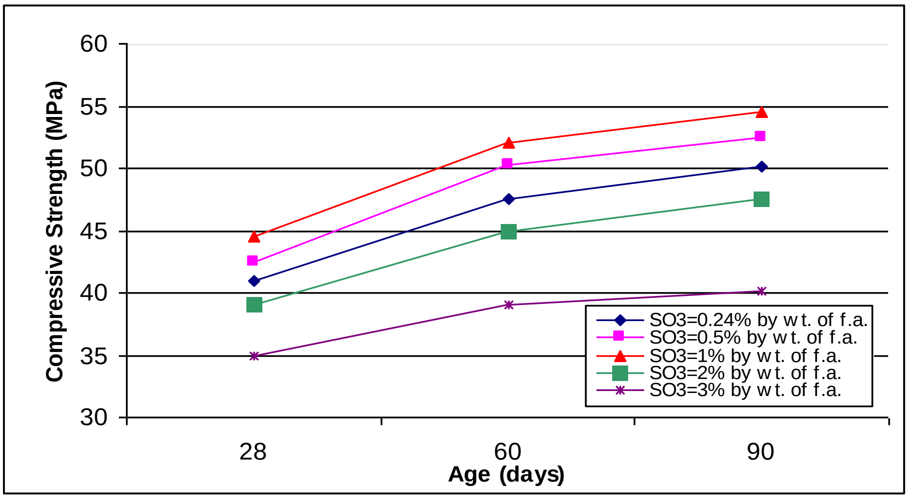
The results obtained indicate that SCC with metackaolin was founded to be effective in improving the resistance of SCC to internal sulphate attack .

Table (11): Effect of Sulphate content in fine aggregate on compressive strength of SCC.

Mix Notation	SO_3 Content in Fine Aggregate %	Total SO_3 Content in Concrete %	Compressive Strength (MPa)		
			28 (days)	60 (days)	90 (days)
MP0	0.24	3.05	37.0	44.0	45.9
MP1	0.5	3.47	38.5	46.0	48.0
MP2	1	4.28	41.5	47.5	49.0
Mp3	2	5.9	35.0	40.5	42.0
MP4	3	7.52	30.0	33.5	34.7
Mmo	0.24	3.08	41.0	47.5	50.2
Mm1	0.5	3.50	42.5	50.3	52.5
Mm2	1	4.31	44.5	52.0	54.5
Mm3	2	5.94	39.0	45.0	47.5
Mm4	3	7.56	35.0	39.0	40.2



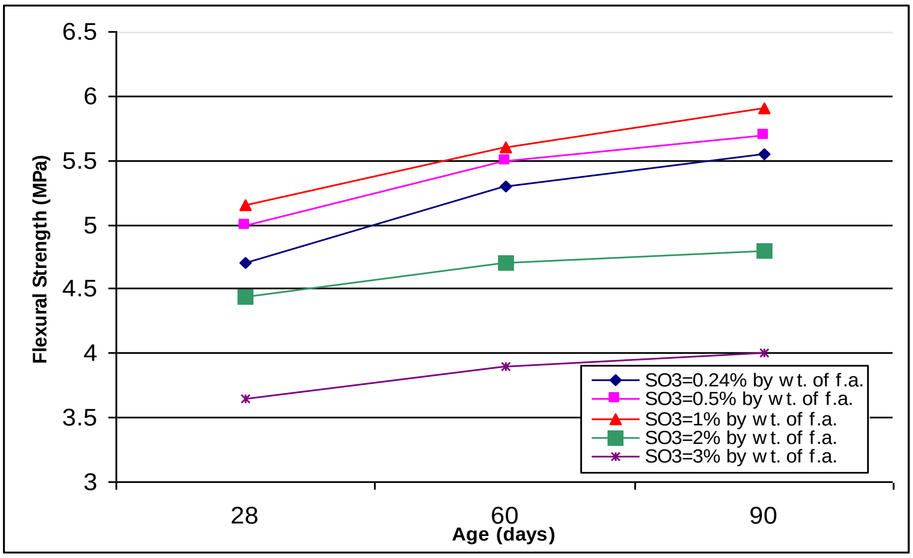
Fig(2):- Relationship between compressive strength and age of concrete at different SO_3 content in fine aggregate for pigment



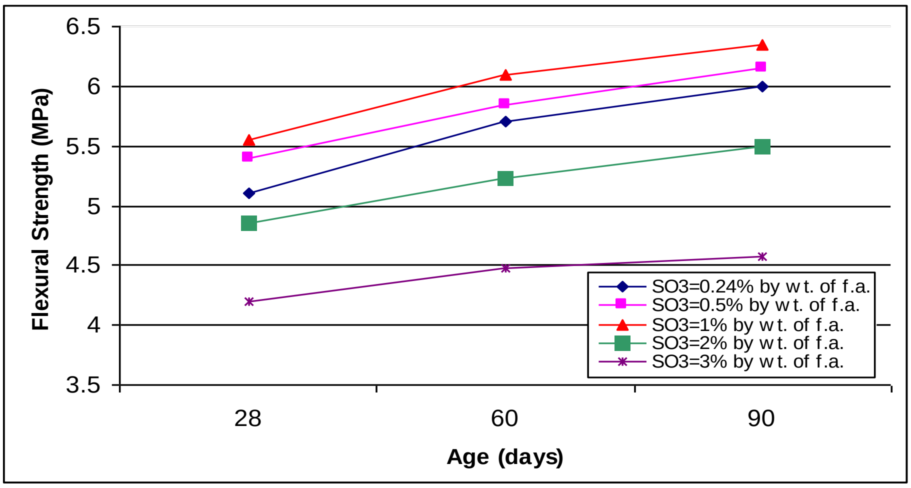
Fig(3):- Relationship between compressive strength and age of concrete at different SO_3 content in fine aggregate for metakaolin

Table (12): Effect of Sulphate content in fine aggregate on flexural strength of SCC

Mix Notation	SO_3 Content in Fine Aggregate %	Total SO_3 Content in Concrete %	Flexural Strength (MPa)		
			28 (days)	60 (days)	90 (days)
MPo	0.24	3.05	4.7	5.3	5.55
MP1	0.5	3.47	5.0	5.5	5.7
MP2	1	4.28	5.15	5.6	5.9
MP3	2	5.9	4.44	4.7	4.8
MP4	3	7.52	3.65	3.9	4.0
Mmo	0.24	3.08	5.1	5.7	6.0
Mm1	0.5	3.5	5.4	5.85	6.15
Mm2	1	4.31	5.55	6.1	6.35
Mm3	2	5.94	4.85	5.23	5.5
Mm4	3	7.56	4.2	4.47	4.58



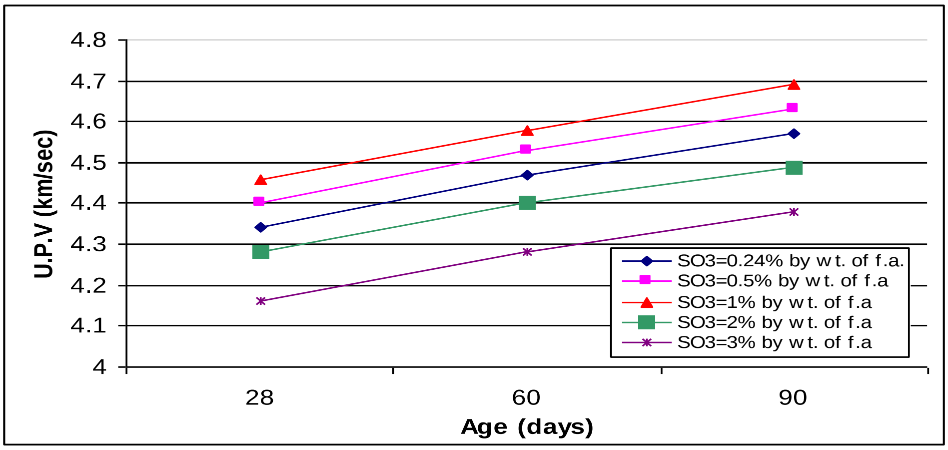
Fig(4):- Relationship between flexural strength and age of concrete at different SO_3 content in fine aggregate for pigment



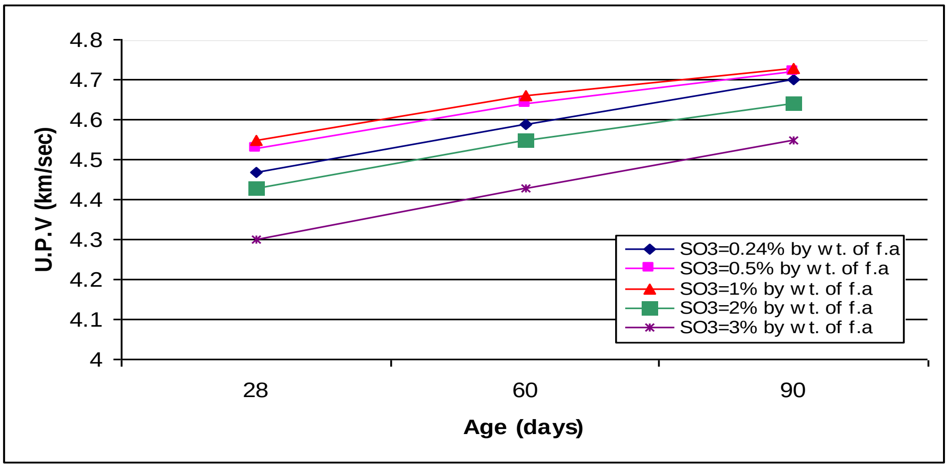
Fig(5):- Relationship between flexural strength and age of concrete at different SO_3 content in fine aggregate for metakaolin

Table (13): Effect of Sulphate content in fine aggregate on ultrasonic pulse velocity (U.P.V) of SCC

Mix Notation	SO_3 Content in Fine Aggregate %	Total SO_3 Content in Concrete %	(U.P.V) (km / sec)		
			28 (days)	60 (days)	90 (days)
MPo	0.24	3.05	4.34	4.47	4.57
MP1	0.50	3.47	4.40	4.53	4.63
MP2	1	4.28	4.46	4.58	4.69
MP3	2	5.90	4.28	4.40	4.49
MP4	3	7.52	4.16	4.28	4.38
Mmo	0.24	3.08	4.47	4.59	4.70
Mm1	0.5	3.50	4.53	4.64	4.72
Mm2	1	4.31	4.55	4.66	4.73
Mm3	2	5.94	4.43	4.55	4.64
Mm4	3	7.56	4.30	4.43	4.55



Fig(6):- Relationship between U.P.V and age of concrete at different SO_3 content in fine aggregate for pigment



Fig(7):- Relationship between U.P.V and age of concrete at different SO_3 content in fine aggregate for metakaolin

Conclusions

1- SCC can be produced with locally available material (pigment and metakaolin).

2- There is an optimum gypsum content in sand of SCC which gives highest compressive strength, flexural strength and ultrasonic pulse velocity. This limit was found to be (SO_3 = 1.0 %) by wt. of sand.

3- The presence of sulphates in sand of SCC (higher than 1.0 % SO_3 by wt. of sand) decreases compressive strength, flexural strength and ultrasonic pulse velocity.

4- The adverse effect of sulphates present in sand is more intensive on flexural strength compared with their deleterious effect on compressive strength.

5- SCC mixes with metakaolin are more resistant to internal sulphate attack than SCC mixes with pigment .

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