Effect of Metakaolin and Fly Ash on Properties of Self Compacting Concrete through Accelerated Curing

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Abstract—Traditionally, strength of concrete in construction work is evaluated in terms of its 28 days compressive strength of cubes/ cylinders. This procedure requires 28 days of moist curing before testing as per IS: 516-1959. This time duration may be considered as a long period. Hence, needs for an accelerated curing technique has arisen, where 28 days strength of concrete can be easily predicted. The main objective of this paper is to develop mathematical model, which gives relation between accelerated curing strength and normal curing strength for 28 and 56 days compressive strength. This study presents the effect of incorporating activated metakaolin (MK) and fly ash (FA) on the mechanical properties of self compacting concrete (SCC) for a variable water/binder ratio. Hot water curing at 80±2 °C is applied to accelerate the strength gain of concrete for the early prediction of 28 days compressive strength. From the results, it was observed that 40kg/m³ replacement from MK or FA was the optimum content in terms of compressive strength. Compressive strength of 59.3MPa was achieved at 40kg/m³ replacement from MK only. Splitting tensile strength, flexural strength and elastic modulus values have also followed the same trend. This investigation has shown that the local MK and FA have the potential to produce SCC. Predicted 28-day strength of concrete from the accelerated curing test was found to be on a conservative side compared to control concrete.

Index Terms—Curing, Self Compacting Concrete, Metakaolin, Fly Ash, Non-Destructive Testing, Hot water

1 INTRODUCTION

Recent trend in engineering technology is to develop economic concrete and complete the project within time limit. To develop the economic concrete, mix design is to be developed and to complete project within time limit, the compressive strength of concrete cubes for selected mix design should be determined earlier in the laboratory. The compressive strength of hardened concrete is most common property required for the structural use. The prediction of 28 days strength at early age is needed for different purpose such as,

The fast trend of construction progress and its economic benefits attained from accelerating construction schedule.
Testing for quality control purposes
To check the suitability of concrete mixes much earlier than 28 days test.

The rate of strength gain mainly depends upon the rate of hydration and the rate of hydration depends on the surrounding temperature. The strength gain could be accelerated at early age and related to 28 days and 56 days compressive strength through calibration curves. Various techniques of accelerated curing of concrete are classified as heat water techniques, oven curing techniques, maturity methods, pressure and elevated temperature technique and expanded polystyrene molds technique IS: 516-1959 [1]. The ACI 214.1 R suggests two procedures, which can be used to provide an indication of 28 days strength of concrete only after 24 hours.

a. Warm water method : 23 to 24 hours at 35±3°C
b. Boiling water method : 23 hours at 21°C and 3.5 hours at 100°C

The ASTM C 684 recommends three different accelerated curing techniques [3].

a. Warm water method: 24 hours at 35±3°C.
b. Boiling water method: 23 hours at 21°C and 3.5 hours at 100°C.
c. Autogenous curing method: 5 hours at 150°C with external pressure.

The British standards, BS 1881, Part 112 provide three curing temperature 35, 55 and 85±2°C for accelerating the rate of gain of strength.

The IS: 9013-1978 recommends two methods of accelerated curing [5].

a. Warm-water method
b. Boiling-water method

Metakaolin (MK) or calcined kaolin, other type of pozzolan, produced by calcination has the capability to replace silica fume as an alternative material. Therefore the use of Metakaolin proves economical over that of silica fume. Previously, researchers have shown a lot of interest in MK as it has been found to possess both pozzolanic and micro filler characteristics (Poon et al. 2001) [6]. It has also been used successfully for the development of high strength self compacting concrete using mathematical modeling (Dvorkin et al. 2012) [7]. However, limited test data are available regarding the performance of the commercially available MK and Indian cements in the case of high strength concrete in the country (Basu 2003, Pal et
Metharonarat, 2004 [10] evaluated the effect of accelerated curing by using high temperature rises curing in water of fly ash concrete. Three water to binder ratio (0.32, 0.42 and 0.52) and three replacement percentage of cement by fly ash (15, 18 and 21) were used. From the test results showed that, the ratio of average compressive strength by accelerated curing method to the 28 days normal curing method (0.983) show the high effectiveness of this method. As well as, concluded the higher replacement percentage by fly ash may cause the rate of hydration reaction to be lower than the lower replacement percentage by fly ash.

Steam curing increased the 1-day flexural strength. However, as oppose to the compressive strengths, the 28 and 90-day flexural strengths of steam cured concrete were comparable to or higher than those of water cured concrete (Kou et al, 2007) [11]. They also concluded that, the flexural strength of concrete significantly decreased as the fly ash content increased. However, at longer curing ages, the beneficial effects of fly ash became evident for moist and steam curing.

The effect of the curing temperature on the static modulus is for different SCC mixes essentially the same as for the compressive strength. The curing temperature affects hardly on the mixes with low w/c ratio. The static modulus of elasticity decreases with increasing curing temperature for the mixes with high w/c ratio compared to storage at 20 °C, with no difference between heat treatment at 60 °C and 80 °C. (Natt et al, 2009) [12].

The accelerated curing by using hot water method had been used in this study. This method is the advantageous method compared to the other accelerated curing methods. First, this accelerated curing method can allow the specimens to be submerged into the water; therefore, the specimens are kept with the same saturated condition as normal curing method. Second, since an increase in the curing temperature of concrete increases its rate of strength development, the provided heating rate can satisfy this condition.

2 EXPERIMENTAL INVESTIGATION

An experimental program was designed to produce a high strength concrete by adding several combinations of FA and HRM. The materials used and the experimental procedures are described in the following sections.

2.1 Materials

The following materials were employed:

A. Cement

Ordinary Portland cement, conforming to the IQS 5/1984 was used for the experimental investigation manufactured by united cement company commercially known (TASLUJA-BAZIAN). Test results indicate that the adopted cement conforms to Iraqi specifications (IQS No.5/ 1984) [13] and given in Table 1.

B. Fly Ash (FA)

Fly ash comprise of the non-combustible mineral portion of coal. Fly ash particles are glassy spherical shaped, ball bearings, finer than cement particles, which helps to reduce amount of water and improve workability. It also reduces heat of hydration and improves durability; the chemical compositions of fly ash are given in Table 1, and physical properties are given in Table 2.

C. High Reactivity Metakaolin (HRM)

It is highly pozzolanic material. Throughout this experimental work Iraqi kaolin clay brought from Dwekhla region was used. It is obtained by calcinations of Algerian kaolin at 700°C for 4 hour, and then the metakaolin was cooled to room temperature for 24 hrs. This procedure of clinking is based on the work of many researches especially (Al-Hadithi, 2003) [14] and (Jawad, 2009) [15]. The silica and alumina contained in the metakaolin are active and react with free lime to form C-S-H and alumina-silicates which greatly improve the strength. The chemical compositions of metakaolin are given in Table 1. The HRM used in this work conforms to the requirements of (ASTM C618, 2002) [16] Class N pozzolan as listed in Table 3.

D. Fine Aggregate

The fine aggregate from Al-Ekadir region confirming to zone 3 as per Limits of Iraqi specification No.45/1984 [17] was used. Physical properties of fine aggregate are presented in Table 4.

E. Coarse Aggregate

Crushed gravel of 10 mm maximum size from Al-Nebai quarry confirming to Limits of Iraqi specification No.45/1984 [17] was used in all mixes. Physical properties of fine aggregate are presented in Table 4.
Superplasticizer (SP) is an essential component for SCC production. It is used to achieve high workability and stability for SCC mixtures. A superplasticizer (SP) of sulphonated melamine and naphthalene formaldehyde condensates, known as (GLENIUM-51) is used in this investigation as a high range water reducing superplasticizer. The typical properties of Glenium 51 are shown in Table 5.

2.2 Test Procedure
To get the early age strength through accelerated curing (AC), hot water method was adopted. In this investigation, two curing procedures were applied. The first procedure (standard water curing) included demoulding of the specimens 24 h after casting and their subsequent storage in the normal curing tank filled with water. The second procedure (hot water curing) in this procedure, after the casting of samples, specimens were cured at normal temperature for 4 hours as a “preset period”; and then put in an accelerated tank with temperature 80 ± 2°C for 4 hours. The rate of temperature rise is 20°C/hour which represents a moderate rate of temperature rise. The period of curing at the maximum temperature is about 12 hours. At the end of curing time, the heaters are switched off manually. For the cooling regime, unheated water is poured from the top of the curing tank with discharge (3.1796 × 10-5) m³/sec and the hot water is drained from the bottom of the tank with the same discharge until the temperature of curing tank reaches to laboratory temperature with cooling rate nearly equal to 20°C/hour. After this period of accelerated curing, specimens are removed from the curing tank and some of these tested for obtained accelerated strength (1 day), and another demolded and stored in normal curing (NC) tank till the age of test (7, 28 and 56) days. This cycle includes the delay period (preset period), the temperature rise period, the period of the curing at the maximum curing temperature and the cooling period. It is designed according to (ACI C517, 1992) [18].

The mix proportioning for SCC used in the present work was designed according to (EFNARC, 2005) [19]. Different constituent materials proportions of the various concrete mixes used throughout this investigation are presented in Table 6.

2.3 Mixing Procedures
All the materials were mixed using a pan mixer with a maximum capacity of 80 l. The mixing procedure involves the following steps:
1. After starting mixer, coarse and fine aggregate were added into drum and homogenized for 30 seconds.
2. Cement and mineral admixtures (FA or MK) were then added within 10 seconds of stopping period and then mixing continued for additional 120 seconds and then stopped.
3. 60% of water mixed with super plasticizer was then distributed all over the mix and mixing continued for 30 seconds.
4. Remaining 40% of water mixed with super plasticizer was added and mixed for 60 seconds.
5. Stop and rest for 30 seconds.
6. Starting again the mixer and dispersion by hand gradually over concrete mix within 60 seconds.
7. Mixing continued for additional 120 seconds and then stopped.

2.4 Placing process
The placing process of the concrete mix is the most critical moment. For fresh SCC mixes which require no compaction work. The fresh concrete was placed into the tight steel
molds until it's fully filled without any compaction. All steel molds were prepared for mixing by placing oil along the interior surfaces of the mold in order to prevent adhesion with concrete after hardening. Finally, surface finishing was done carefully to obtain a uniform smooth surface.

2.5 Tests on Concrete

A. Fresh Concrete

The fresh properties of plain SCC were tested by the procedures of (European Guidelines for self compacting concrete). In this work three tests were used slump flow test, L-box test and sieve segregation resistance were used for assessment of fresh properties of SCC in this study.

B. Hardened Concrete

The engineering properties studied were compressive strength, splitting tensile strength, flexural strength and static modulus of elasticity of concrete. All the specimens were tested, on saturated surface dry condition at the age of 1, 7, 28 and 90 days on 3000 kN machine. Samples were divided into three groups, (i) standard cubes of dimensions 150×150×150 mm for measuring the compressive strength, (ii) cylinders of 100 mm diameter and 200 mm height for measuring the indirect tensile strength and the modulus of elasticity and (iii) prisms of 100×100×500 mm to measure the flexural strength.

2.6 Curing Tank

Curing tank shall be constructed from any material of suitable strength that will resist the effect of corrosion. Internal dimension should be adequate to accommodate the required number and size of test specimen. The tank shall contain sufficient water and be controlled so that temperature of water around the specimen immersed in the tank is maintained at the desired level. For this work the curing tank is fabricated in the form of a cylinder with a well insulated circumference and bottom by glass wool 25mm thick to minimize the loss of temperature from water tank. The internal dimension of the water tank is (750mm in height and 1000mm in diameter) and done by steel plate thick 4mm with two story. The first story is done by steel mesh at 125mm from the base of the tank; while the second story is done by three steel angles on the side of the water tank at 400mm from the base; steel tire place over these angle. Base of the tank is raised 200mm from the ground level by steel angle. Five electric heaters are used; three of them are fixed at 100mm from the base of the water tank and the others at 375mm from the base. The cover of the tank contain five relief valves to minimize the pressure inside curing tank; one of them used for cooling stage to input tap water to the curing tank. Fig. 1 depicts the curing water tank.

<table>
<thead>
<tr>
<th>Materials</th>
<th>SCC-1</th>
<th>SCC-2</th>
<th>SCC-3</th>
<th>SCC-4</th>
<th>SCC-5</th>
<th>SCC-6</th>
<th>SCC-7</th>
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<tbody>
<tr>
<td>Cement Kg/m³</td>
<td>500</td>
<td>460</td>
<td>420</td>
<td>460</td>
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<td>340</td>
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<tr>
<td>FA Kg/m³</td>
<td>0</td>
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<td>0</td>
<td>40</td>
<td>80</td>
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<td>HRM Kg/m³</td>
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<td>Gravel Kg/m³</td>
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<td>Water l/m³</td>
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<td>SP l/m³</td>
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<td>Water-cementitious material ratio</td>
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<td>0.33</td>
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</table>
3 RESULTS AND DISCUSSION

3.1 Compressive Strength
The results of compressive strength for the SCC mixes are shown in Table 7 and plotted against mixes in the Fig. 2 for the different ages, and various curing conditions. It is clear from this Fig. that as the curing temperature increases, the compressive strength increases at different ratios. At age of 7, 28 days and 56 days after accelerated curing condition, it is obvious from above the Table and their corresponding Fig., that significant reduction in the percentage of increment in strength of concrete when the age increase from 7 to 56 days when compared with normal moist curing. For compressive strength test results are correlated with 28 days compressive strength of standard water curing. The Mathematical models developed to show the relation between accelerated curing compressive strength and (28, 56 days) normal curing compressive strength for SCC is derived from Fig. 3.

The rise in curing temperature speeds up the chemical reaction of hydration and, thus, affects the early strength of concrete. The rapid initial rate of hydration at higher temperature retards the subsequent hydration and produces a non-uniform distribution of the products of hydration within the paste. This is due to the fact that at the high initial rate of hydration, there is insufficient time available for the products of hydration away from the cement particle and for a uniform precipitation of the products of hydration space. As a result, a high concentration of the products of hydration is built in the vicinity of the hydration particles, and this retards the subsequent hydration and adversely affects long-term strength (Hawra, 2003) [20].

The reduction in compressive strength for (SCC-3, 5 and 7) compared to SCC-2, 4 and 6) is explained as the result of a clinker dilution effect. The dilution effect is a result of replacing a part of cement by the equivalent quantity of MK and FA. In MK and FA concretes, the filler effect, pozzolanic reaction of MK and FA with calcium hydroxide and compounding effect (synergetic effect of mineral admixture) react opposite of the dilution effects (Parande et al. 2008; Ding et al. 1999) [21, 22]. For this very reason, there was an optimum MK and FA replacement for MK and FA SCC. With time, the compressive strength differences between the MK, FA mixtures and reference SCC concrete becomes smaller. This might be due to the fact that all cementitious materials reactions were close to completion, or had stopped; mainly because the reactions between MK, FA and OPC mixtures were slowed down with time (Wild and Khatib 1997) [23].

3.2 Splitting Tensile Strength
The tensile strength results of reference, MK and FA concretes with varying amounts of MK and FA are shown in Table 7. The average values of the 28-day tensile strength for the concretes made was about 4.85 and 4.85 MPa, which corresponds to 5.15 and 5.15 % of NC and AC respectively of the compressive strength for the same concretes. Table 7 shows that the average ratio between the tensile strength (fsp) to cube compressive strength (fc) of concrete at 28 days was lower than the range (of about 9-10 %) for medium strength concrete reported earlier (Neville 1997; Rasiah 1983; Haque and Kayali 1998) [24, 25, 26]. This indicates that as the compressive strength increases lower would be the ratio, which is consistent with the results published by other investigators earlier (Rasiah 1983; Haque and Kayali 1998; Yogendran et al. 1987) [25, 26, 27]. From the results it can be seen that similar to compressive strength the splitting tensile strength also exhibited the highest strength at SCC-2 mixture. Fig. 5 presents the relation between compressive strength and splitting tensile strength of all the mixtures at 28 days. It can be observed that as the compressive strength increases, the tensile strength also increases.

3.3 Flexural Strength
As shown in Fig. 6, the presence of MK and FA influenced the flexural strength of the studied mixes. The results showed an increase of the flexural strength from (5.10 to 8.00) MPa and (5.89 to 8.36) MPa after replacement of 40kg/m3 from MK in the case NC and AC respectively. The increment that obtained from the using MK and FA mixes.
This behavior is due to the pozzolanic activity of MK or FA on hydration of cement, where MK or FA reacts with Ca(OH)2 and this reaction leads to augmentation in the densification of transition zone and thus increases the bonding strength at the interface zone and the formation of micro cracking is decreased. Hence, the micro cracking initiation occurs at a higher stress level (Aulia and Deutschmann, 1999) [28].

Fig. 2. Variation of compressive strength with respect to pozzolanic materials percentage.

Fig. 3. Relation between accelerated (1 day) and normal compressive strength at (28 & 56) days.

Fig. 4. Comparison of splitting strengths for different methods of curing.

Fig. 5. Variation of compressive strength with respect to splitting tensile strength.
3.4 Elastic Modulus

The secant modules for all the mixes are experimentally determined for each testing age as the average of two cylinders for NC and AC and the results are shown in Table 7. These static modules are plotted against SCC mixes, as shown in Fig.7. The modulus of elasticity is mainly related to the compressive strength of concrete. However, due to the existence of non-linear relationship between them (Neville 1997; Mehta and Monteiro 1999) [24, 29], the increase in the modulus of elasticity is not in proportion to the increase in compressive strength as noted in Table 7. The modulus values presented in Table 7 indicate that the rate of increase in the modulus is lower than the rate of increase in the compressive strength. The elastic modulus (E) values with respect to the MK contents that the similar trend is to that obtained for compressive strength; here the optimum MK percentage that gives maximum E is at 40kg/m³.

From preliminary observation for the previous Table and its corresponding Figures mentioned, it is noticed that the values are higher for metakaolin mixes than those for reference mixes. Moreover, mixes contain MK records values slightly higher than those for mixes contain FA. The strength (Fcu) is correlated with E as shown in Fig. 8. A direct linear, power and an exponential relationship were attempted and it was found that the power relationship in the form given below fitted the data best.

In addition, the predicted values according to the American Concrete Institute (ACI) model \( E = 4.73\sqrt{F_{cu}} \) and BIS model \( E = 5.0\sqrt{F_{cu}} \) are also plotted in the same Fig. 8. The Fig. 8 shows that the data points of MK mixtures lie slightly above the predicted modulus of ACI model but the BIS model overestimates the values obtained by actual testing.
3.5 Non-Destructive Testing of Concrete

a) Surface hardness results

Surface hardness of the concrete cubes was assessed by the "Schmidt rebound hammer" (RN). The Schmidt rebound number could be used as an indication of the degree of degradation of the concrete surface. Table 8 shows the results of the rebound number for concrete specimens of the two curing methods. Fig. 9 depicts the variation of rebound number, at different ages, for specimens cured by conventional wet curing and accelerated curing respectively. The average rebound numbers of SCC mixes have been more or less the same and ranged from 26-40 for specimens of normal curing and accelerated curing.
b) Ultrasonic pulse velocity test results (U.P.V)

Fig. 10 depicts the variation of UPV in km/sec, at different ages, for specimens of three series of concrete mixes cured by conventional wet curing and membrane curing respectively. The measured pulse velocity in concrete can be affected by many factors, including smoothness of concrete surface, temperature of specimen, moisture conditions, age of the specimen, and presence of steel reinforcement. The relationship between the compressive strength and pulse velocity is limited by the following strict limitations (Relis, 1988) [30]:

1. The pulse velocity depends on the modulus of elasticity of the aggregate.
2. The pulse velocity depends also on the aggregate content in the mix.
3. The type of the aggregate also affects the velocity of the waves.
4. Mix proportions.
5. Water/cement ratio.

The pulse velocity values of samples presented in Table 8, shows that the velocity criterion for concrete quality grading as per IS: 13311-1992 (Part-1) [31], which have been evolved for characterizing the quality of concrete in structures in terms of the ultrasonic pulse velocity. The highest ultrasonic pulse velocity value of 5.22 km/sec was obtained in the case of the accelerated curing SCC concretes incorporating metakaolin and was measured in mix SCC-2 containing 40kg/m³ Silica Fume (SF). The 28-day UPV values of the normal and accelerated curing of SCC ranged from 4.02 to 4.96 km/sec. In general, the UPV values of all SC concretes tested indicate the concrete of good and excellent quality.

4 CONCLUSIONS

Based on the results of the experimental work carried out in this study to verify of types of curing and mix proportion, the following conclusions are deduced:

a) The incorporation of MK and FA as a partial replacement 40kg/m³ of weight cement of mixes improves significantly all mechanical properties of SCC at all ages as compared with mixes without materials (MK or FA) for two curing regime.

b) It was found that the test results show that there is a significant increase in compressive strength for SCC specimens cured at 80°C with respect to those cured at 25°C, for all mixes.

c) At all ages, the compressive strength of SCC with 40kg/m³ metakaolin is found to be greater than that of all SCC and for two curing methods.

<table>
<thead>
<tr>
<th>TABLE 8 NON-DESTRUCTIVE TEST RESULTS.</th>
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<tr>
<td>Mix</td>
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<td>SCC-1</td>
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<td>SCC-3</td>
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<td>SCC-6</td>
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<td>SCC-7</td>
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d) The achieved results of elasticity at later ages are not affected by the curing temperature as it is noticed at early ages. The condition of curing regime does not play an important role at later ages.

e) The optimum replacement level of OPC by MK was 40 kg/m³ of metakaolin, which gave the highest compressive strength in comparison to that of other replacement levels; this was due to the dilution effect of partial cement replacement. These SC concretes also exhibited a 28-day splitting tensile strength of the order of 5.15 % of their compressive strength and showed relatively high values of modulus of elasticity. Splitting tensile strengths and elastic modulus results have also followed the same trend to that of compressive strength results showing the highest values at 40 kg/m³ metakaolin replacement.

f) The Rebound numbers of SC concretes have been more or less the same. They are in the range 26-37 for specimens of conventional wet curing and 29-38 for specimens cured with accelerated curing respectively.

g) The ultrasonic pulse velocity values of various concretes, cured by different techniques, correspond to excellent and good concrete quality grading.

REFERENCES

[17] Iraqi Specification, No.45/1984, "Aggregate from Natural Sources for Concrete and Construction".


