Effect of Elevated Temperature on Mechanical Properties and Microstructure of Silica Flour Concrete

M. S. Morsy, S. H. Alsayed and M. Aqel

***Abstract*—An experimental investigation was conducted to evaluate the influence of elevated temperatures on the mechanical properties, phase composition and microstructure of silica flour concrete. The blended cement used in this investigation consists of ordinary Portland cement (OPC) and silica flour. The OPC were partially replaced by 0, 5, 10, 15 and 20% of silica flour. The blended concrete paste was prepared using the water-binder ratio of 0.5 wt% of blended cement. The fresh concrete pastes were first cured at 100% relative humidity for 24 hours and then cured in water for 28 days. The hardened concrete was thermally treated at 100, 200, 400, 600 and 800 ^{o}C for 2 hours. The compressive strength, indirect tensile strength, phase composition and microstructure of silica flour concrete were compared with those of the pure ordinary Portland concrete. The results showed that the addition of silica flour to OPC improves the performance of the produced blended concrete when exposed to elevated temperatures up to 400^{o}C.**

***Index Terms*—Concrete; Silica Flour; Strength; Phase composition; Microstructure**

I. Introduction

Concrete containing mineral admixtures is used extensively throughout the world for their good performance and for ecological and economic reason. The most common cementitious materials that are used as concrete constituents, in addition to Portland cement, are fly ash, ground granulated blast furnace slag, silica fume and rice husk ash. They save energy, conserve resources and have many technical benefits [1]. Metakaolin is a recent addition in the list of pozzolanic materials. Concrete is well known for its capacity to endure high temperatures and fires, owing to its low thermal conductivity and high specific heat [1]. However, it does not mean that fire as well as higher temperatures does not affect the concrete. Characteristics such as color, compressive strength, elasticity, concrete density and surface appearance are affected by high temperature [2-5]. Therefore, improving concrete's fire resistance is a field of interest for many researchers lately. According to their studies, it is possible to improve fire resistance of concrete in few ways. Cement replacement with pozzolanic materials is one of the very efficient methods [6-12]. The addition of polypropylene fibers in a concrete mix is also found to be useful [13-14]. However, the main attribution to thermal properties of concrete is provided by aggregates [15].

Fire resistance of concrete is highly dependent on its constituent materials, particularly the pozzolans. The effect of high temperature on concrete containing fly ash or natural pozzolans has not been investigated in detail. Researchers and investigators differ in their opinion regarding the changes in the properties of concretes, particularly in the range of 100–300 ^{o}C. Whereas for temperature above 300 ^{o}C, there is uniformity in opinion concerning a decrease in mechanical characteristics [16–20]. However, strength reductions which have been reported in the literature reveal significant quantitative differences due to the variety of high temperature condition tested, and the variety of constituent materials of concrete used. It is recognized that the behavior of concrete subjected to high temperatures is a result of many factors such as heating rate, peak temperatures, dehydration of C–S–H gel, phase transformations, and thermal incompatibility between aggregates and cement paste [21, 22]. On the other hand, quality control of concrete, by means of non-destructive methods, in structures subjected to fire or not so high temperature exposure conditions, is not particularly easy to be carried out. The correlation usually refers to the hydration age of 28 days.

Since human safety in case of fire is one of the major considerations in the design of buildings, it is extremely necessary to have a complete knowledge about the behavior of all construction materials before using them in the structural elements.

The scope of this work is to provide experimental data on the residual mechanical and physical properties of blended cement concrete subjected to heat, containing micro silica (silica flour) as pozzolanic materials. These properties are very important for a safe design of concrete and in the repair of concrete structures.

II. Experimental Work

A. materials

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The starting materials used in this study are silica flour (10 micron) supplied by Adwan Chemical Industries, Saudi Arabia and ordinary Portland cement, supplied by Yamama Cement Company, Saudi Arabia which complies with the Saudi Arabian Standard SAS 143/1979. The oxide composition of silica flour and ordinary Portland cement are summarized in Table (1). The fine and coarse aggregates were local natural river sand and crushed lime stone with maximum nominal sizes of 10 and 20 mm, respectively, which were mixed in a ratio of 1.795:2.667.

TABLE 1
CHEMICAL COMPOSITION OF MATERIAL (MASS %)

Oxide Composition	Ordinary Portland Cement	Silica Flour
CaO	63.85	0.025
SiO_2	19.83	99.410
Al_2O_3	5.29	0.200
Fe_2O_3	3.53	0.025
MgO	0.52	0.005
SO_3	2.43	---
Na_2O	0.21	0.030
K_2O	0.07	0.013
TiO_2	----	0.060
Total	95.73	99.768
Ignition loss	2.82	0.26

B. Concrete Preparation

The blended concrete mix was prepared using ordinary Portland cement that was partially substituted by 0, 5, 10, 15 and 20% silica flour as illustrated in Table (2). The concrete was prepared using water/binder ratio of 0.5 wt% of binder. The concrete was molded into 100 mm cubes for compressive strength and 100 × 200 mm cylinders for indirect tensile strength tests. The molds filled with concrete were vibrated for one minute to remove any air bubbles. The samples were kept in molds at 100% relative humidity for 24 hours, and then cured in water for 28 days. At the age of 28 days, the specimens were heated in an electric furnace at 100°C, 200°C, 400°C, 600°C, and 800°C. Each temperature was maintained for 2 hours to achieve the thermal steady state [23]. The heating rate was set at 2°C/min based on the experience of our previous research [24]. The specimens were allowed to cool naturally to room temperature. The compressive strength and indirect tensile strength tests were performed on wet and thermally treated specimens. The crushed samples resulted from compressive strength testing of wet specimen were grounded for thermal analyses and microstructure studies.

TABLE 2:
IX PROPORTIONS OF CONCRETE MIXTURES

Mix	Silica Flour (%)	Water /Binder (%)	Batched quantities (kg)						
			Water	Silica flour	Cement	Fine Aggregate		Coarse Aggregate	
						Red sand	Crushed sand	20 mm	10 mm
M0	0	0.5	10.576	0	21.153	22.780	15.178	39.594	16.814
M1	5	0.5	10.576	1.058	20.095	22.780	15.178	39.594	16.814
M2	10	0.5	10.576	2.115	19.038	22.780	15.178	39.594	16.814
M3	15	0.5	10.576	3.173	17.980	22.780	15.178	39.594	16.814
M4	20	0.5	10.576	4.231	16.922	22.780	15.178	39.594	16.814

C. Testing

Compressive Strength

The compressive strength tests were performed on 100 mm cubes using using 3000KN compressive machine (Test Plant Toni PACT II) on according ASTM C109 [25]. Three samples per batch were tested and the average strength is reported. The loading rate on the cubes was 0. 35 mm/min.

Indirect Tensile Strength

The indirect tensile tests were performed on 100 × 200 mm cylinders using Toni Tech machine as per ASTM C496 [26]. Three samples per batch tested and the average strength is reported. The loading rate on the cylinder was 0.5 mm/min. The indirect tensile strength is determined as follows:

$$\sigma_t = 2P_{max}/\pi DL$$

where σ_t is the indirect tensile strength, P_{max} is the maximum applied load, D is the
diameter and L is the length of cylinder.

Phase Decomposition

The phase decomposition study was performed using differential scanning calorimeter (DSC) analysis for which Shimadzu DSC 50 thermal analyzer was used. The thermal analysis runs were conducted using a heating rate of 20 °C/min. The samples chamber was purged with nitrogen at a flow rate of 30 cc/min.

Microstructure

The scanning electron microscope (FEI Inspect–S) was used to identify the changes occurred in the microstructure of the formed and/or decomposed phases. The resolution of SEM was 4nm.

III. RESULTS AND DISCUSSION

The compressive strength of thermally treated concrete specimens after cooling was determined. The results shown in figure (1) indicate the residual compressive strength of each specimen at different elevated temperatures. It shows a relative decrease in the compressive strength of each specimen thermally treated up to 100°C as compared to its original compressive strength before heating. From the perspective of residual compressive strength of concrete, the heating conditions can be divided into three regions as 25 – 100 °C, 100-400°C and 400 – 800 °C. Distinct patterns of strength loss followed by a gain were observed in first two regions and then subsequent sharp loss in the third region. The reduction in compressive strength at 100°C can be attributed to the driving out of free water. The silica flour concrete showed a higher increase in compressive strength in 100-400 °C range than control concrete. This increase may be due to the hydrothermal interaction of the silica flour particles as a result of temperature rise with the liberated free lime during hydration reaction. The control specimens showed a slow increase in compressive strength in 100-400°C range followed by sharp loss in strength. The observed increase in compressive strength of the control concrete may be due to

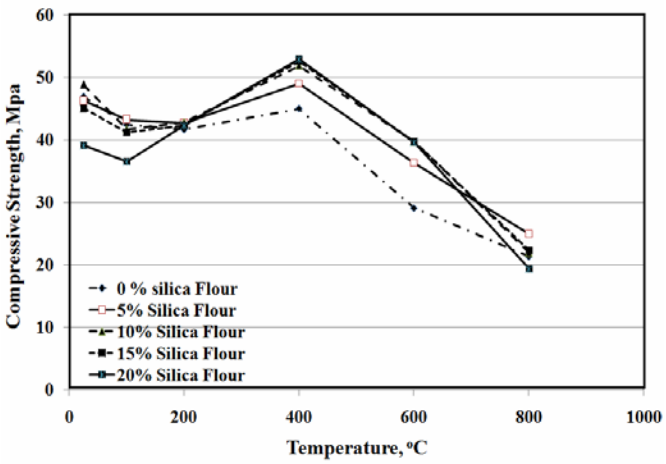


Fig. 1. Compressive strength of concrete exposed to elevated temperature after cooling

further hydration of unhyarated cement grains as a result of internal autoclaving effect. However the increase in compressive strength of blended concrete containing 5, 10, 15 and 20% silica flour, was mainly due to pozzolanic reaction; which led to the formation of additional amount of hydration products. At high temperatures, especially above 100 °C, the thermal effect might cause water migration whereas dehydration of moisture supply from outside is insufficient. Internal stress and thus micro and macrocracks are generated due to the heterogeneous volume dilatations of ingredients and the buildup of vapor in the pores. Therefore, at higher temperature, especially above 400 °C, the observed decrease in compressive strength of blended concrete containing 5, 10, 15 and 20% silica flour, may be due to internal thermal stress generated around pores which generate microcracks.

Figure (2) illustrates the indirect tensile strength of concrete with and without silica flour exposed to elevated temperature up to 800°C. Evidently, the indirect tensile strength of concrete decreases as the exposed temperature increases.

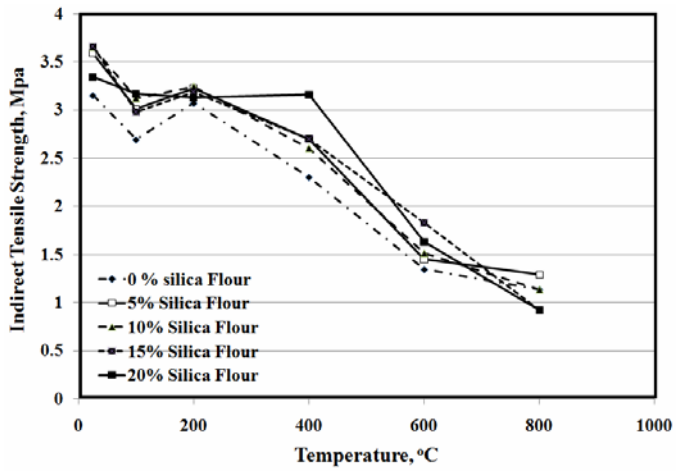


Fig. 2. Indirect tensile strength of concrete exposed to elevated temperature after cooling

The replacement of ordinary Portland cement by 20% silica flour in concrete caused a stable tensile strength up to 400°C followed by a sharp decrease. In 20% silica flour concrete, the exposure to 400 °C led to further hydrothermal reaction of unhydrated cement grains and pozzolanic reaction of silica flour with calcium hydroxide librated during the hydration process. Furthermore, the decrease in indirect tensile strength from 200°C to 800°C was due to the formation of micro-cracks. Also the reduction in tensile strength can be attributed to the driving out of free water and fraction water of hydration of concrete due to high temperatures. Dehydration of concrete causes a decrease in its strength, elastic modulus, coefficient of thermal expansion and thermal conductivity [26].

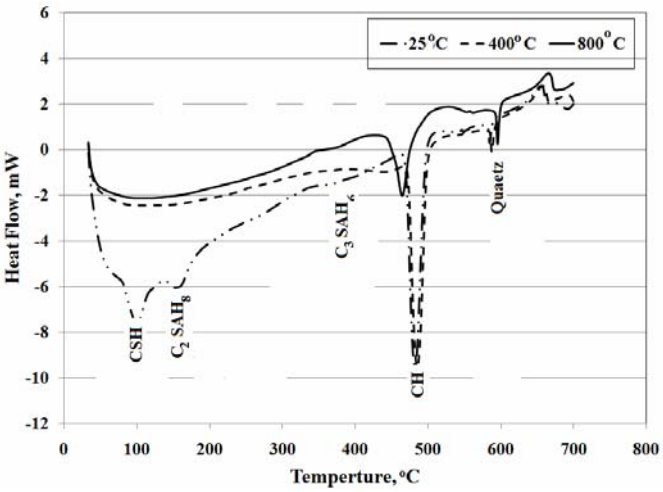


Fig.3. DSC thermograms of control concrete thermally treated at 25, 400 and 800oC

The DSC thermograms of control concrete and silica flour concrete thermally treated at 25, 400 and 800°C are shown in figures (3 & 4). Evidently, there were almost five endothermic peaks. The first peak was located at about 110-120°C, which was mainly due to the decomposition of calcium silicate hydrates (CSH). The second endothermic peak was observed at 160°C which represents the decomposition of the calcium sulpho-aluminate hydrate. The third endothermic was located at 370°C which was due to the decomposition of C_3ASH_6. The fourth endothermic peak was observed at 470°C which represents the decomposition of the calcium hydroxide. The fifth endothermic peak was observed at 580°C represents the decomposition of quartz. The endotherm obtained for control concrete after thermal treatment at 800°C, was shifted to 450°C which was lower than that required for the decomposition of well crystallized calcium hydroxide (470 – 500°C); this was mainly due to the decomposition of free calcium hydroxide (CH) located at the bulk of the hardened specimens. Actually, the free CH formed in the bulk of the specimen seems to possess an ill-crystallized or nearly amorphous character which was easily decomposed at relatively lower temperatures. However the thermograms of the silica flour concrete thermally treated at 800°C as shown in figure (4) show very weak endothermic peaks of calcium hydroxide. Evidently; this may be due to further reaction between silica flour, and CaO at high temperature (800°C). It was obvious that, the enthalpy of CH phases in control concrete decreases from 23.22 J/g to 9.23 J/g as the treatment temperature increases to 800°C. On the other hand, the

enthalpy of CH phases in the silica flour concrete decreases from 11.2J/g to 7.68J /g as the treatment temperature increases to 800°C. This may be due to the formation of ill-crystals of free lime as a result of further reaction of pozzolana at elevated temperatures and also, due to the physical filler effect of the smaller particles in the mixture. Evidently, the products of further reaction and physical filler of pozzolana deposit in the porous system cross link inside, which lowers the temperature gradient across this system. Therefore, the thermal stresses induced by heat flow may be considered a singular at the tip of cracks, which was similar to the isothermal condition.

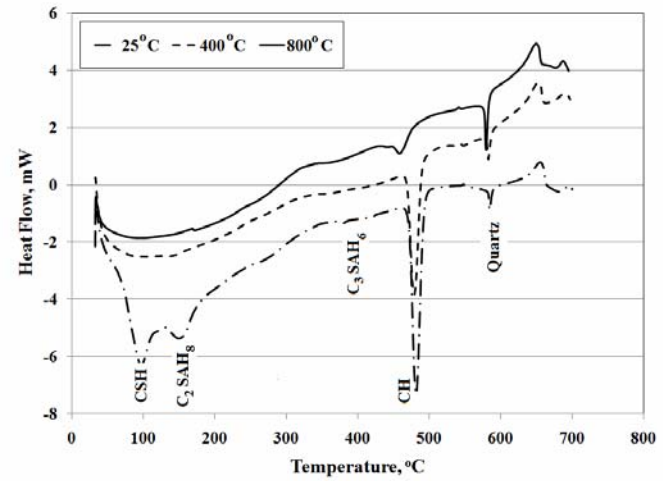
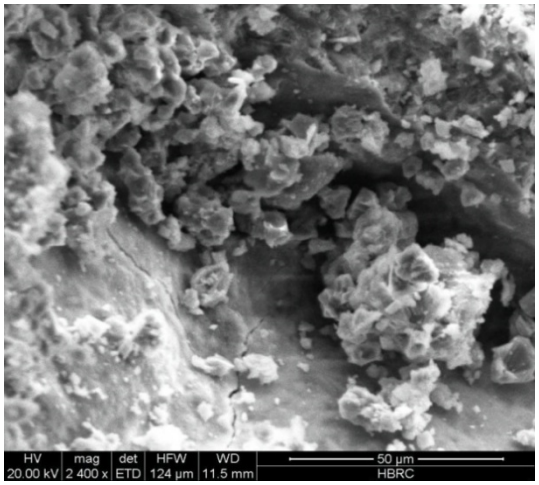


Fig. 4. DSC thermograms of silica flour concrete thermally treated at 25, 400 and 800°C

The scanning electron micrograph of control concrete (M0) and concrete containing 20% silica flour (M4) thermally reated at 25, 400 and 800°C are shown in figure (5). Evidently, the microstructure of the control concrete at 25°C displayed the existence of microcrystalline and nearly amorphous, mainly as calcium silicate hydrates (CSH); in addition to large crystals of calcium hydroxide as shown in figure (5-a).

a

Furthermore, the microstructure of control concrete thermally treated at 400oC showed the formation of dines structure of hydration products as shown in figure (5-c). For samples thermally treated at 800°C, the SEM micrograph displayed the formation of microcracks with average widths of 5 µm and also the decomposition of the hydration products as shown in figure (5-e). The SEM micrographs obtained for the pozzolanic concrete products obtained after 28 days of hydration were mainly calcium silicate hydrates and calcium hydroxide as shown in figure (5-b).



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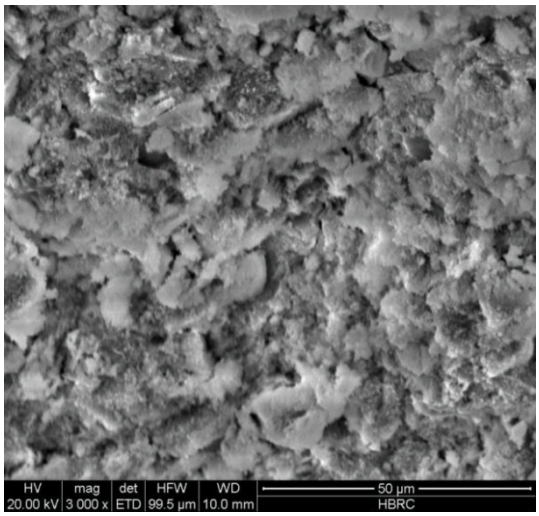
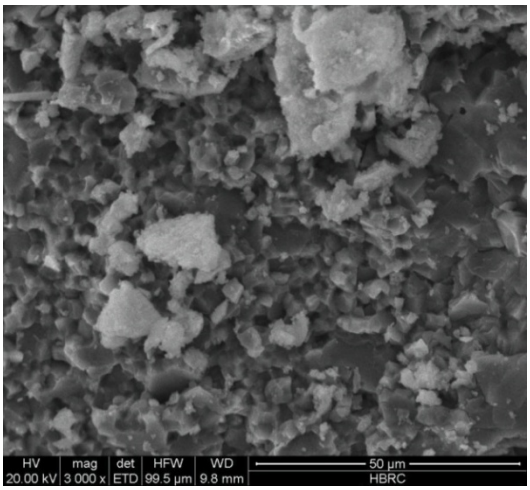


c

It was clear that, the microstructure of silica flour concrete, thermally treated at 400°C, was perfectly stable for thermal treatment and illustrates a dense structure of hydrated products as shown in figure (5-d). This can be clearly understood from the microstructure of the hardened blended cement concrete (M4) after thermal treatment at 400°C; the microstructure displayed the existence of calcium silicate hydrates (CSH) and calcium hydroxide (CH). Therefore, the replacement of

ordinary Portland cement by 20% of silica flour resulted in an improvement of the thermal stability of the hardened blended cement concrete made of mix M4 as indicated from the SEM



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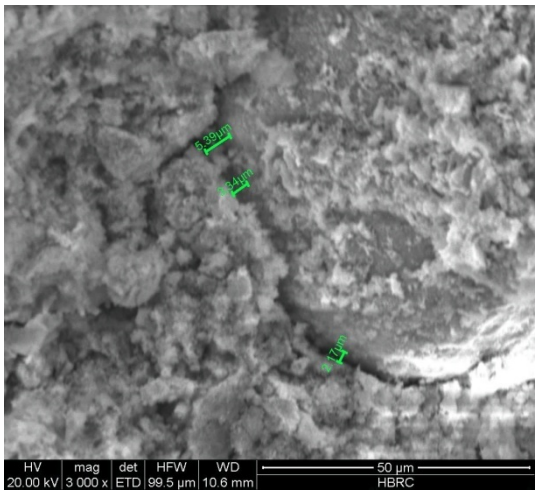

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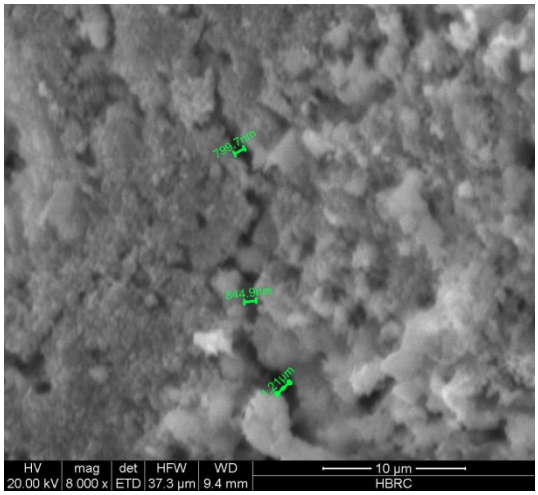

f

Fig. 5.: SEM micrograph of the control and silica flour concrete thermally treated at 25, 400 and 800°C , a) *SEM micrograph of control concrete at 25°C, b) SEM micrograph of silica flour concrete at 25°C, c) SEM micrograph of control concrete at 400°C, d)* SEM micrograph of silica flour concrete at 400°C, e) SEM micrograph of control concrete at 800oC, f) SEM micrograph of silica flour concrete at 800°C

micrographs shown in figure (5-d). Thermal treatment of mix M4 concrete at 800°C showed a decomposition of the hydration products with the formation of average microcracks of width 9nm in the structure as shown in figure (5-f).

IV. Conclusions

The following conclusions can be drawn from the present study:

- The studied silica flour has a very positive effect on the strength after thermal treatment at 400°C for 2 hours.
- The pozzolanic reaction of silica flour is accelerated between 100 and 400°C; this is accompanied by a steep decrease of Ca $(OH)_2$ content.
- Based on the mechanical and physical properties of silica flour concrete, it was observed that 20% silica flour concrete was generally more favorable than 5, 10 and 15%, and thus can be used in structural elements exposed to elevated temperature up to 400°C.

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