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The performance of Fly ash and Metakaolin concrete at elevated temperatures





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HIGHLIGHTS

• Mechanical performance and durability of Fly ash and Metakaolin concrete at elevated temperatrues was evaluated.

• Quick cooling produced greater loss in compressive strength than slow cooling.

• For all mixes, major strength and durability loss occurred after 400 °C.

• At 400 °C and above, FA20 showed better performance while MK10 and MK20 showed higher degradation in terms of durability.

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$A \hspace{0.1in} B \hspace{0.1in} S \hspace{0.1in} T \hspace{0.1in} R \hspace{0.1in} A \hspace{0.1in} C \hspace{0.1in} T$

Ordinary concrete is generally considered to have satisfactory fire resistance but when it comes to high strength concrete it shows extensive damage or even catastrophic failure at elevated temperatures. This research work was carried out to evaluate the performance of High Performance Concrete (HPC) made with Fly ash (FA) and Metakaolin (MK) at elevated temperatures. Variables of the test program include partial replacement of cement with MK from 5% to 20%, FA from 20% to 60%, temperatures from 27 °C to 800 °C and two types of cooling methods (in air and water). The mechanical performance was assessed from compressive strength while the durability was assessed from chloride permeability and water sorptivity tests. Mass loss at elevated temperatures was also determined. Moreover, quantitative analysis of the SEM images on selected concrete specimens was performed using Image Pro-plus software.

Test results showed degradation in the mechanical and durability properties of HPC at elevated temperatures. Quick cooling produced greater loss in compressive strength than slow cooling. This is because of the effect of thermal shock which was more pronounced at 400 °C. From the standpoint of durability, all mixes showed major increase in charge pass and sorptivity values in between 400 °C and 600 °C. Therefore, 400 °C could be regarded as the critical temperature for change in the properties of HPC. Quantitative analysis of the SEM images of Interfacial Transition Zone (ITZ) indicated that pore area fraction increased with the increase in temperature. This resulted in the degradation of microstructure and affected the strength and durability of concrete. In general, at temperatures (400 °C and above) FA20 showed better performance while MK mixes (MK10 and MK20) showed higher degradation in terms of durability. This gives an indication that MK mixes should be used with care especially in structures which may be subjected to temperature of 400 °C and above.

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1. Introduction

Fire remains one of the serious potential risks to most of the buildings and structures [1]. The extensive use of concrete as a structural material has led to the demand to fully understand the effect of fire on concrete. The issues related to exposure of concrete

to elevated temperatures pose concerns about the serviceability and stability of the structure which, in turn, are related to human safety. Moreover, many studies have shown extensive damage or even catastrophic failure at high temperatures, particularly to high strength concretes [2].

The development of HPC started in 1980s and thereafter it was used extensively in many countries [3,4]. Inclusion of eco-friendly materials is increasing day by day due to their superior structural performance, environmental friendliness and energy conservation implications [5]. The research on performance of FA in concrete

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at elevated temperatures started in 1969 by Lohtia [6] while Nasser and Marzouk [7] presented one of the earlier studies on the fire performance of FA concretes. Many researchers have also assessed the mechanical and durability performance of fly ash concrete [8–11] at elevated temperatures. In short, it was found that FA improved the performance of concrete at elevated temperatures and this improvement was more significant at temperatures below 600 °C [12]. Poon et al. investigated the performance of Metakaolin at elevated temperatures [13]. They found that concrete with higher replacement of MK (20%) suffered higher loss in strength at all temperatures and suggested that severe durability loss occur at elevated temperatures. This could render the concrete structure completely unserviceable. These and many other researches [10,11,13–15] used mercury intrusion porosimetry as a tool to investigate the connection between the loss in compressive strength and durability while the current research uses Image Analysis technique to scientifically determine the degradation in mechanical and durability properties. Moreover, there will be more use of Metakaolin and FA in making concrete and therefore it is important to ascertain whether the concrete made from these materials is safe in case of fire or elevated temperatures. Furthermore, many studies have been performed in the past using flat and polished surface of aggregate. However, it could promote the formation of water film at the aggregate surface, which may not correspond to real concrete situation [16]. Real concrete often includes aggregate particles which are not smooth. Therefore, there is a need to study ITZ taking into consideration the effect of rough aggregate particles. This research thus examines the microstructure of ITZ in high performance concrete made with crushed granite aggregate particles after exposure to normal and elevated temperatures by the analysis of images obtained from SEM.

In this research, seven HPC mixes made with Fly ash and Metakaolin at elevated temperatures were investigated. Variables of the test program include partial replacement of cement with MK from 5% to 20%, FA from 20% to 60%, temperatures from 27 °C to 800 °C and two types of cooling methods (in air and water). The mechanical performance was assessed from compressive strength while the durability was assessed from chloride permeability and water sorptivity tests. Mass loss at elevated temperatures was also determined. Moreover, quantitative analysis of the SEM images on selected concrete fractured specimens was performed using Image Pro-plus software.

2. Experimental investigation

2.1. Materials

The cementitious materials used in HPC mixes were ordinary Portland cement (OPC), low-calcium pulverised fuel ash complying with relevant British Standards (BS) and metakaolin. Metakaolin is a thermally activated alumino-silicate produced from kaolinite clay through calcining process. The chemical composition and physical properties of these materials are given in Tables 1 and 2.

The coarse and fine aggregates used were crushed granite and river sand respectively, complying with relevant BS. Coarse aggregates of two sizes 20 mm and 10 mm were used. The density, water absorption and fineness modulus values

Table 1

Chemical composition of OPC, FA and MK.

Chemical composition (%)	OPC	FA	MK
Silicon dioxide (SiO ₂)	19.6	56.8	53.2
Aluminum oxide (Al ₂ O ₃)	7.3	28.2	43.9
Ferric oxide (Fe ₂ O ₃)	3.3	5.3	0.38
Calcium oxide (CaO)	63.1	3.0	0.02
Magnesium oxide (MgO)	2.5	5.2	0.05
Sodium oxide (Na ₂ O)	0.1	-	0.17
Potassium oxide (K ₂ O)	1.1	-	0.10
Sulfur trioxide (SO ₃)	2.1	0.7	-
Loss on ignition	3.0	3.9	-

Table 2

Physical properties	OPC	FA	MK
Specific gravity Specific surface (m ² /kg)	3.16 312 25	2.31 412	2.62 12,680
7 days	45		
Initial setting time (Min)	125		
That setting time (will)	210		

are listed in Table 3. Moreover, a sulphonated naphthalene formaldehyde condensate was used in adequate quantities in the mixes to achieve desired slump of 100 mm and above.

2.2. Mix proportions

The details of mix proportion investigated in this research are given in Table 4. PC represents the control concrete mix while MK and FA are pozzolans which were introduced as cement replacement. Based on previous research [13,17], Metakaolin was used in replacement level of 5%, 10% and 20% (MK5, MK10 and MK20) while fly ash was introduced in replacement level of 20%, 40% and 60% (FA20, FA40 and FA60). These ranges correspond normal, medium and high replacement levels of FA in concrete.

2.3. Mixing, casting and curing details

The batching, mixing and casting of concrete was carried out in accordance with relevant BS. For curing, the specimens were removed from the moulds after 24 h of casting and placed in water tank at 27 °C for 28 days and then in a humidity (65%) and temperature (27 °C) controlled room for further five months.

2.4. Specimen preparation for SEM

The microscopic studies on selected concrete specimens (PC, MK20 and FA20) were carried out by using SEM. The concrete cubes (100 mm) were cut into two or three slices of 12 mm thickness using rotary saw. The outer 5 mm strip was discarded because of a likely inferior hydration due to escape of moisture by evaporation. From 12 mm thick slice, a strip of 12 mm imes 12 mm cross section and 100 mm length was cut. The cutting procedure is schematically shown in Fig. 1. The position of fracture at a specific place along the length of strip was selected where the fractured surface could comprise a neat interface of paste and aggregate. A V-groove was made at the selected position as a crack-inducer for fracture. The prism was then clamped and fractured by a firm knock. Fig. 2 [18] shows the process schematically. After this, the fractured surface was cut in a slice of not more than 5 mm thickness by using a precision diamond saw followed by immersing the thin fractured slices in acetone to stop further hydration in the surface zone and cleaned from free particles produced during specimen cutting and fracture operations in an ultrasonic vibrator. The specimens were then stored in clean acetone until heating in the furnace. After heating the SEM specimens, they were further treated for preparations normally performed for SEM which include vacuum drying and gold coating.

2.5. Testing procedure and methods

2.5.1. Heating details

The concrete specimens were heated to elevated temperatures at the age of about 180 days using automatic electric furnace as shown in Fig. 3. The furnace had the rating of 1280 °C with programmable microprocessor temperature controller attached to the furnace power supply and equipped with thermocouples for furnace temperature measurement. In addition, the specimen temperatures were measured with thermocouple embedded at the centre of the sample. It is worth mentioning here that the furnace had the ability to provide heat from two sides and the bottom.

The specimens were subjected to peak temperatures of 200 °C, 400 °C, 600 °C and 800 °C and they were heated to ensure the steady state [19]. Accordingly, the test specimens were placed in the furnace starting from normal temperature and

Table 3

Density, water absorption and fineness modulus of aggregates.

	Coarse aggregates		Fine aggregate
	20 mm	10 mm	River sand
Density (g/cm ³)	2.62	2.61	2.61
24 h water absorption	0.6	0.6	0.7
Fineness modulus	-	-	2.4

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