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Mechanical performance, durability, qualitative and quantitative analysis of microstructure of fly ash and Metakaolin mortar at elevated temperatures

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HIGHLIGHTS

- ▶ Performance of FA and Metakaolin mortar at elevated temperatrues was evaluated.
- ▶ Using gray scale segmentation, different hydration phases were found.
- ▶ For all mixes, major strength and durability loss occurred at 400 °C.
- ▶ Major drop in hydrated paste area and rise in pore area fraction occurred at 400 °C.

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ABSTRACT

An experimental investigation was carried out to evaluate the performance of Fly Ash (FA) and Metakaolin (MK) mortar at elevated temperatures. Variables of the test program include partial replacement of cement with MK from 5% to 20%, FA from 20% to 60% and temperatures from 27 °C to 800 °C. The mechanical performance was assessed from compressive strength while the durability was assessed from chloride permeability test. Qualitative analysis of the microstructure of heated and unheated mortar was performed by Scanning Electron Microscope (SEM) while quantitative analysis was performed on SEM images using Image Pro-plus software.

Test results show that for all mixes compressive strength decreased while charge passed increased with the increase in temperature from 27 °C to 800 °C. For all mixes, major strength and durability loss occurred after 400 °C. Therefore, 400 °C can be considered as critical temperature from the standpoint of strength and durability loss. Qualitative and quantitative analysis of SEM were found to be consistent with the results of strength and durability loss. The observation of SEM images and image analysis of area fractions of hardened cement paste (hcp) of different mortar mixes indicated that with the increase in temperature the pore area fraction increased while hydrated paste area fraction decreased. These factors resulted in the degradation of microstructure and affected the strength and durability of mortar. Major drop in hydrated paste area and rise in pore area fraction occurred at 400 °C. Therefore, 400 °C could be regarded as the critical temperature for change in the properties of mortar. In general, fly ash mix (FA20) showed better performance in all aspects.

1. Introduction

Concrete can be exposed to high temperatures and pressures for considerable periods of time during their service life in industries like oil, gas, and power. Besides, in the event of fire, concrete is also exposed to elevated temperatures. Due to composite nature of concrete and different thermal characteristics of constituents, its performance is greatly affected by high temperature [1]. The issues related to the exposure of concrete to the elevated temperatures pose concerns about the serviceability and stability of the structure which, in turn, are related to human safety.

Presently, the use of pozzolan incorporated in mortar and concretes has become much common throughout the world. The application of such material is increasing day by day due to their superior structural performance, environmental friendliness and energy conservation implications [2]. The materials which are commonly used for partial replacement of cement are silica fume (SF), pulverized fuel or fly ash, ground granulated blast furnace slag (GBFS) and Metakaolin. Each of the pozzolanic materials induces various characteristics in the final product. These characteristics have been explored by the researchers throughout the world. While some areas have been covered in some detail, many other

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characteristics of such concrete are still needed to be explored. One of such areas is to characterize pozzolan incorporated in mortar after subjecting to elevated temperatures.

The research on the performance of FA in concrete at elevated temperatures started in 1969 [3] whereas Nasser and Marzouk [4] presented one of the earlier studies on the fire performance of FA concretes. Sarshar and Khoury [5] performed research on the fire performance of OPC-FA pastes. They tested OPC-FA pastes containing 30% FA by weight under a series of temperatures till 650 °C. The relative residual compressive strength was 88% at 450 °C and 73% at 600 °C, which was almost double than the residual strength shown by pure OPC pastes. This research showed that OPC-FA pastes performed better under elevated temperatures than OPC pastes. Aydin and Baradan [6] investigated the effect of high temperature on the mechanical properties of cement based mortars containing pumice and fly ash. It was found out that pumice mortar with 60% FA performed best and did not show any loss in compressive strength at all the test temperatures when the specimens were cooled in air. Yazici et al. [7] studied the effect of high temperature on the compressive strength of mortars incorporating FA, SF and pumice. In general, pumice mortars showed lowest where as silica fume mortars produced highest compressive strength values at all tested temperatures.

The investigation into the performance of Metakaolin at elevated temperature was carried by Poon et al. [8]. They investigated the residual strength of high performance concrete made with 5%, 10% and 20% Metakaolin having the unheated compressive strengths of 107, 123 and 131 MPa respectively. Temperature levels were selected as 200 °C, 400 °C, 600 °C and 800 °C. A gain of 2–8% in strength was observed when the concretes were subjected to the temperature of 200 °C. Marginal drop in the strength of the order of 5–8% was found at the temperature of 400 °C. At 600 °C a loss in strength of about 54–68% was observed while 85–90% loss in strength has been reported at 800 °C. It was specifically observed that concrete with higher replacement of MK (20%) suffered higher loss in strength at all temperatures. Poon et al. [8] also suggested severe durability loss at elevated temperatures and greater propensity of explosive spalling. This could render the concrete structure completely unserviceable. Morsy et al. [9] in their research on effect of elevated temperature on physic-mechanical properties of MK blended cement mortar found that MK improves the fire resistance capability of mortar till a replacement ratio of 20%.

This research was aimed to investigate the elevated temperature performance of mortar made with MK and FA in varying proportions. Image analysis technique has been used to scientifically determine the degradation in mechanical and durability properties of mortars.

2. Experimental investigation

2.1. Materials

The binder materials used in mixes were ordinary Portland cement (OPC), low-calcium pulverized fuel ash complying with relevant British Standards and Metakaolin. Fly ash was supplied by China Light and Power Company while Metakaolin was procured from IMERYS International. The chemical composition and physical properties of these materials are given in Table 1.

The fine aggregate used was river sand complying with the requirements of BS 882: 1992. The fine aggregate particles had specific gravity of 2.61, 24 h water absorption 0.7% and fineness modulus of 2.4. To achieve desired slump of 100 mm and above, a sulfonated naphthalene formaldehyde condensate, a dark brown liquid containing 38.6% solids was used as superplasticizer.

2.2. Mix proportions

Seven high performance mortar mixes were investigated. The details of the mix proportions are given in Table 2. PC, which is the OPC mortar mix, represents the control mix while MK and FA are pozzolans which were used as cement replacement. Based on previous research [8,10], 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 corresponds normal, medium and high replacement levels of FA in mortar.

Table 1
Composition of OPC, FA and MK.

Chemical composition (%)	OPC	FA	MK
Silicon dioxide (SiO_2)	19.6	56.8	53.2
Aluminum oxide (Al_2O_3)	7.3	28.2	43.9
Ferric oxide (Fe_2O_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_2O)	0.1	–	0.17
Potassium oxide (K_2O)	1.1	–	0.10
Sulfur trioxide (SO_3)	2.1	0.7	–
Loss on ignition	3.0	3.9	–
Physical properties			
Specific gravity	3.16	2.31	2.62
Specific surface (m^2/kg)	312	412	12,680
Compressive strength 3 days (MPa)	35		
7 days	45		
28 days	60		
Initial setting time (Min)	125		
Final setting time (Min)	240		

Table 2
Mix details (per m^3).

Mix	Cement (kg)	FA (kg)	MK (kg)	Water (kg)	Sand (kg)
PC	450	0	0	135	643
MK5	428	0	22	135	640
MK10	405	0	45	135	641
MK20	360	0	90	135	638
FA20	360	90	0	135	634
FA40	270	180	0	135	625
FA60	180	270	0	135	615

2.3. Curing conditions

The specimens were removed from the molds 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 5 months.

2.4. Heating and cooling details

The mortar specimens were heated to elevated temperatures (200, 400, 600 and 800 °C) at the age of about 180 days using automatic electric furnace. 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. The specimen temperatures were also measured with thermocouple embedded at the centre of the sample. The specimens were heated to ensure the steady state. The steady state was achieved when the temperature at the centre of specimen was either ±10 °C of furnace temperature or the rate of temperature rise at the centre of specimen was less than or equal to 5 °C per hour [11]. Accordingly, the test specimens were placed in the furnace starting from normal temperature and heated at a rate of 5 °C/min up to the peak furnace temperature. The peak temperature was kept constant up to the time when steady state was established. The variations of furnace temperature and the temperature at the centre of specimen (thermocouple temperature) with time of a typical heating cycle are illustrated in Fig. 1. It was observed that the time from the start to the setting up of steady state ranged from 3 to 4 h for all the specimens. After reaching the steady state, the specimens were allowed to cool in the furnace overnight with the door of the furnace open for easy dissipation of heat and tested after 24 h.

For SEM, samples were heated in a temperature controlled furnace at a very slow heating rate of 1 °C/min up to the desired temperatures. This slow heating rate minimized differential temperature gradients in the specimens and thus differential thermal cracking was minimized. Therefore, the application of heat was nearly uniform on the specimens. The specimens were cooled naturally in a closed chamber.

2.5. Testing procedure and methods

The compressive strengths of mortar cubes were determined by BS 1181: Part 116 1983 while chloride permeability test was carried out on selected samples of mortar specimens (PC, MK20 and FA20) according to AASHTO T277-831. As per