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Effects of re-curing on microstructure of concrete after high temperature exposure

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HIGHLIGHTS

- High temperature exposure and re-curing cause morphological changes in concrete.
- Hydrates disappeared after heating, resulting in a decrease in mechanical properties.
- Rehydration of CaO caused further deterioration of concrete after air re-curing.
- Regeneration of hydrates resulted in mechanical recovery in water re-cured concrete.

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ABSTRACT

Concrete contains many phases such as ettringite, portlandite, C-S-H etc. which undergo phase changes by forming some active products such as lime and calcium silicates during heating. These new active phases give reactions with water and carbon dioxide in air and affect the residual properties of concrete after subsequent days of cooling. In order to understand better these phase changes and their effects on residual properties, 3 different types of concrete were produced depending on mineral admixture type. Concrete specimens were heated to 1000 °C and one face heating procedure was applied in the furnace. Then concrete specimens were subjected to air and water re-curing processes. Microstructural investigations XRD, TGA and SEM/EDX were conducted on the samples obtained from concrete specimens.

1. Introduction

Due to excessive temperature increases in concrete hydration products which determine the strength and durability performance of concrete start to alter by losing their water content due to dehydration and also by releasing carbon dioxide due to decarbonation [1–3]. Firstly, ettringite, C-S-H and other hydrates which have more than one water molecule in their compositions deteriorate due to dehydration [4,5]. Then, dehydration of $Ca(OH)_2$ takes place around 450 °C and decarbonation of carbonates begins after 600 °C [4,5]. After 800 °C most of the constituents in concrete become decomposed and weight loss of concrete slows down [4]. These new compounds are similar to compounds of unhydrated cement and they include calcium silicates, calcium aluminates and calcium oxides etc. which are chemically active [5]. Therefore, chemical activity continues at the post-heating stage, causing changes in physical and mechanical properties of concrete [3,6,7]. Water vapor and carbon dioxide in air can react with some oxides and calcium silicate phases after cooling and resulting products determine the final state of concrete [3,7,8]. For example, disintegration and further deterioration of concrete can be observed due to CaO rehydration. Because of the fact that, $Ca(OH)_2$ is produced at the end of this rehydration and its volume is 44% higher than CaO [7–9]. Due to expansive behavior of this rehydration, size and numbers of existing cracks can increase and mechanical performance of concrete may decrease after cooling [9,10]. In literature it was reported that using mineral admixtures in concrete could eliminate or reduce the negative effect of CaO rehydration since slag consumed $Ca(OH)_2$ after rehydration [8].

On the other hand, when concrete come in contact with water after cooling, healing and recovery of concrete can be possible in the case of regeneration of C-S-H and carbonates phases in concrete [3,9,11]. However, water re-curing is a debated application since some of researches inferred that water re-curing after cooling

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could be beneficial for the recovery of concrete and many other researchers found water re-curing application detrimental for concrete [4,5,11–13]. Unfortunately, there are very limited numbers of study about water re-curing after heat exposure. Moreover, some of the curing techniques proposed in these researches are not easily applicable on full scale structural members.

Consequently, a comprehensive research project was designed in order to understand disintegration and recovery processes at micro level and the effects of microstructural changes on residual mechanical properties of concrete. Hence, concrete groups were subjected to air and water re-curing applications after heating to 1000 °C. In this project, one face heating conditions were applied and also only the heated faces of concrete specimens were subjected to water during water re-curing since these methods can be considered more realistic and applicable. Ground granulated blast furnace slag (GGBFS) and pulverized fly ash (PFA) as mineral admixtures were added into concrete mixes in order to evaluate their possible effects on deterioration and recovery of concrete at the post-heating stage. PP fiber reinforced and air entrained concrete groups were produced in order to reduce the spalling risk of concrete by forming a more permeable network in concrete during heating [10].

2. Experimental study

2.1. Materials and specimens

CEM I type Portland cement (PC), ground granulated blast furnace slag (GGBFS) and F type pulverized fly ash (PFA) were used in concrete groups as cementitious materials. PC represents Portland cement concrete group, SC represents concrete incorporating GGBFS and FC represents concrete incorporating PFA. Cement replacement ratios of GGBFS and PFA were 40% and 30%, respectively since these ratios were found to be optimum in previous researches [14,15]. Total amount of cementitious materials in 1 m^3 of concrete was 450 kg in every concrete group and all concrete groups had a water to binder ratio of 0.45. Water contents were determined according to EN 206-1 and TS 13515 since cement equivalence factors for GGBFS and PFA are given to be 0.8 and 0.4, respectively. Table 1 shows mix proportions of concrete groups. As chemical admixtures, oil alcohol and ammonium salt based air entraining admixture (AEA) and modified polycarboxylate based superplasticizer were used in concrete mixes. AEA content was the same and 0.3 kg (0.7% of total weight of binder) in all concrete groups. Super plasticizer amount changed in order to obtain slump levels in S4 limits given in EN 206-1. PP fibers were used as low melting point fibers to reinforce concrete groups and PP fibers were used 0.2% of volume of concrete. River sand and siliceous gravel were used in all concrete groups as aggregates.

15 cubic specimens with dimensions of 15 × 15 × 15 cm were produced for each concrete group. One day after production, specimens were demolded and placed in curing water for 27 days. Then they were conditioned in laboratory environment for additional 2 months before tests [14,16,17]. Three cubic specimens were used to determine initial properties of concrete before heating. Remaining 12 cubic specimens were heated to 1000 °C. Three of them were used to monitor temperatures in concrete cubes during heating, 3 of them were tested to determine properties after cooling and remaining 6 cubic specimens were subjected to air and water re-curing. Therefore, in tables and graphs 0, X, Z and W represent tests before heating, after cooling, after air re-curing and after water re-curing, respectively.

Table 1
Mix proportions.

Materials	PC (kg/m^3)	SC (kg/m^3)	FC (kg/m^3)
Portland Cement	450	270	315
GGBFS	–	180	–
PFA	–	–	135
Water	203	186	167
Aggregate No1	484	493	496
Aggregate No2	484	493	496
Crushed Sand	564	574	577
River Sand	225	229	230
Super Plasticizer	6.0	7.8	9.6
Air Entraining Admixture	0.3	0.3	0.3
PP Fiber	1.8	1.8	1.8
Density	2285	2280	2296

2.2. Heating procedure

An electrical furnace which has a 1250 °C maximum operation temperature was used in this study. Two stages of heating were determined and during the first stage specimens were heated to 1000 °C and during the second stage specimens were kept at this temperature until the end of the total heating time (200 min). Fig. 1 represents the heating procedure and also shows thermocouple positions in a cubic concrete specimen. Electrical furnace was operated at full power and temperature inside the furnace reached 1000 °C in approximately 120 min. Since furnace heating capacity was limited, heating rate was 20 °C/min at the beginning but the rate decreased to 5 °C/min at the end of the first stage of heating. After the second stage completed, hot concrete specimens were not taken out until the furnace cooled down to 100 °C. This heating rates applied in this study can be considered as too high for material characterization due to the fact that higher heating rates cause thermal stresses which may result in further damage in concrete during heating [18]. However, the aim of this study was to mimic real fire conditions as close as can be done with the available facilities. Four concrete cubes were placed together in the electrical furnace and in order to simulate one face heating conditions they were insulated with aerated concrete blocks as shown in Fig. 1. For temperature monitoring K-Type thermocouples were placed inside a concrete specimen during heating.

2.3. Re-curing processes

After heating and cooling processes 3 specimens were subjected to air re-curing and 3 specimens were subjected to water re-curing for 28 days. Only the heated faces of water re-cured specimens were subjected to tap water. Air re-cured specimens were kept in laboratory environment which has a relative humidity of 65 ± 10% and temperature of 20 ± 2 °C during re-curing period.

2.4. Macroscopic observation

Visual changes on heated or fire exposed concrete give information about maximum temperature experienced and amount of deterioration of concrete [1,10,19]. Heated surfaces of each concrete group were monitored after re-curing processes.

2.5. Microscopic observations

Microstructural investigations were conducted on samples which were obtained from a specimen before heating and from specimens after cooling, after air re-curing and after water re-curing, in order to determine phase changes such as $CaO/Ca(OH)_2$ and $CaO/CaCO_3$ conversions. These samples were taken from the center of heated surface of concrete specimens and from the centroid of concrete specimens by using a percussion drill in order not to use cooling water during cutting process (the maximum length on 1 dimension was 1 cm).