

Microstructural Changes in High Density Concretes Exposed to High Temperatures

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Abstract

Reinforced concrete structures, when submitted to fire or exposed to high temperatures, might suffer important microstructural changes, which in turn affect their macroproperties, such as compressive strength and porosity. These changes are both chemical and physical in nature, involving loss of water, thermal expansion/contraction and the modification of the crystalline arrangements of some of its constituents. Their combined action might significantly reduce the resistance of the structural member, inclusive to the point of collapse. Researches in the field of concrete fire resistance are usually focused on the monitoring of the external signs of degradation that are visually noticeable, such as the micro-cracks, expansions and spalling. The chemical and physical changes in the microstructure are less examined, although they are the primary reasons behind the degradation. The research discussed in this paper was designed to look into the determination of the residual properties of mortar and cement pastes after heating exposure at up to 800oC. The changes in the microstructure were monitored with XRD, SEM, DTA and dilatometry tests. The results obtained confirmed that high density concretes are more vulnerable to spalling and that the degree of deterioration can be correlated to changes in the x-ray difratograms.

Keywords: high density concrete; high temperatures; residual properties; spalling.

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1 Introduction

During a fire, the users of a building are subject to a high life risk, and the building can suffer considerable structural damages. Researches on the exposure of concrete elements to high temperatures, as well as the analysis of the resulting structural behavior, indicate that, under certain conditions, the effects of heating can be quite deleterious and intense, inspiring concerns about the dynamics and consequences of the material degradation process and raising doubts about the performance of some types of concrete exposed to fires.

The behavior of concrete in high temperatures is determined by several factors that interact in different ways, being very difficult to make a proper analysis of the degradation process, which prevents the formation of generic concepts regarding the matter [1]. It is known that the microstructure of the concrete is highly heterogeneous and complex, making difficult the establishment of exact models to estimate the behavior of this material during heating [2]. Both the cement paste and the aggregate are made up of components that might decompose, in different degrees, when exposed to high temperatures. The concrete permeability, the size of the structural member, and the rate of temperature growth are all influent factors, since they rule how internal pressures build up during the process.

However, the concrete structure is basically made up by cement paste, aggregates and the transition zone among them. One can have a general idea of the resulting behavior analyzing the properties of each constituent and the relationships among them. Macroscopically, a sample of concrete can be seen as being made up by aggregate particles with varied shapes and sizes, involved in a matrix constituted by a continuous mass of hardened paste, that is to say, a double-face material made up by aggregate particles scattered in a cement matrix [2].

Microscopically, the concrete structure is more complex, since the two phases of the structure are not homogeneously distributed with reference to each other, neither are themselves homogeneous. There can be situations where the paste is extremely dense, compared to the aggregates, and others when the paste is largely interrupted by voids and pores. Moreover, several aspects of the behavior of the concrete under loading can only be understood and explained when the transition zone, i.e., the interface between cement paste and aggregates is treated as a third phase in the concrete structure.

In the last few years the manipulation of the concrete microstructure allowed the production of high-density concretes, with very high compressive strengths and excellent durability. Nonetheless, some researchers have reported that there are some important differences in the performance of high-density concretes (HDC) at elevated temperatures, when compared with normal strength concrete (NSC). When HDC is exposed to heating, the build-up of internal water pressure under steep temperature gradients generates high local stresses, which may end up causing explosively spalling [3,4].

It has been theorized that HDC is more susceptible to spalling, in great part, because of its low permeability. The extremely high water vapor pressure generated during exposure to fire cannot escape the high-density matrix of HDC and often reaches the saturation vapor pressure [4], generating tensile stresses in the material.

The spalling originates from two simultaneous processes. The first one is a thermo-mechanical process that is related to the thermal dilation gradients taking place in the structure; the second one is a thermo-hydro process that is associated with the transfer of mass in the porous network (air, vapor, liquid water), which results in the build-up of high pore pressures and pore pressure gradient [5]. The phenomenon can, therefore, be seen as a result of a thermo-hydro-mechanical coupled process.

Spalling results in the loss of concrete during a fire and has the effect of exposing deeper layers of concrete to fire temperatures, thereby increasing the access of heat to the inner layers of the structure [4].

Although there are still a limited number of experimental programs in this area, some researchers have reported that the addition of polypropylene fibers to HDC is a suitable way to avoid explosive spalling under fire conditions. This change in behavior is derived from the fact that polypropylene fibers melt in high temperatures, leaving more space to the heated gas to occupy and, eventually, creating a pathway for the gas to escape the concrete matrix.

These mechanisms would reduce pore pressure and allow the outward migration of water vapor, resulting in the reduction of internal stresses [6,7,8,9,10].

At 160°C, the reduction in the volume of polypropylene fibers due to the melting starts to happen. As the heat increases, the fibers will degrade, beginning to ignite at temperatures close to 360°C. At this point, the fibers regress to their constituent materials, and all that remains of individual filaments is soot, which occupies approximately 5% of the original size. The voids that remain create the additional space and the routes that let the water vapor escape. So, the vapor pressure is reduced or released and the explosive spalling is avoided, at least for a while [11].

Due to this mechanism, the melting of polypropylene fibers turns HDC into a more porous material, which will behave more like NSC when exposed to high temperatures. Nonetheless, although spalling is avoided, it is still necessary to establish the residual strength after fire, since the loss of the fibers, the creation of pores and the effects of the heat will affect the strength of the material [9]. The deterioration may be so intense that it will call for the strengthening of elements.

The analysis of the alterations suffered by the concrete due to exposure to high temperatures must be made as much in the macro structural level, which corresponds to the ones caused to the structural stability of the building, as in the micro structural level, which corresponds to the damages caused to the properties of the materials [12].

Researches in the field of concrete fire resistance are usually focused on the monitoring of the external signs of degradation that are visually noticeable, such as the microcracks, expansions and spalling. The chemical and physical changes in the microstructure are less examined, although they are the primary reasons for the whole concrete degradation.

The research discussed in this paper was designed to look into the determination of the residual properties of mortar and cement pastes after heating exposure at up to 800°C.

To begin, a brief revision of the micro structural alterations suffered by the cement paste and by the aggregate because of the heating will be made, since they will greatly influence the residual properties of the concrete.

1.1 Cement Paste

The hydrated Portland cement paste is basically made up of calcium silicate, calcium hydroxide and calcium sulfoaluminate. The paste normally has a great amount of free water and capillary water, besides the physically combined and the adsorved water. From the view of the prism of protection against fire, the concrete temperature will not rise until the whole evaporable water has been removed, which requires a considerable amount of heat for the conversion of water into vapor.

The presence of evaporable water in the concrete causes damages to the material, which takes the form of superficial delaminations usually known as spalling. The same occurs when the vapor pressure inside the material increases in a rate higher than the pressure relief caused by the liberation to the vapor to the atmosphere [2].

The free and capillary water present in the cement paste starts to evaporate when the temperature reaches 100°C, delaying the heating of the concrete mass. The total capillary water evaporation occurs between 200°C and 300°C, but the alterations in the structure of the hydrated cement are still not significant, and do not impact its compressive strength. From 300°C to 400°C, there is a progressive reduction in the gel water, resulting in a considerable strength loss and in the development of superficial cracks in the concrete [13].

1.2 Aggregates

The aggregates present in the concrete do not have the same coefficient of thermal dilatation of the cement paste, leading to the appearance of internal stresses caused by the differential material expansions. Many times these expansions are increased by structural transformations of the internal structure of certain aggregates. Some aggregates rich in crystalline silica, such as natural quartz river sand, granite and sandstones, might suffer a sudden volume change in temperatures close to 570°C. Their volume can increase more than 85%, creating tension cracks. This occurs because of the changes of the crystalline array of the quartz from a to β .

Carbonaceous rocks might also suffer damages in temperatures above 700°C, originated from dissolution reactions. Calcareous and light aggregates are the less affected by heat than those mentioned above. This favorable performance derives from the low coefficients of thermal dilatation and the endothermic reactions that occur when the temperature increases, which lead to the creation

of a superficial film of \mbox{CO}_2 that acts as a thermal insulator $\mbox{[13]}.$

2 Experimental Investigation

An experimental program was design to investigate the residual properties and the microstructure changes in mortar and cement pastes after exposure to temperatures up to 800°C. The work was developed in pastes and mortars representative of typical HDC that might be used today. The basic idea was to isolate the effects in each one of the components, gradually increasing the complexity of the material to check the resulting interactions. In this work the cement paste and fine aggregate are investigated. The next step is to make investigations in concrete samples, analyzing the effects of introducing the coarse aggregate.

Changes in the microstructure were monitored with X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM), DTA (Differential Thermal Analysis), and Dilatometry tests.

2.1 Materials

For molding the specimens, a high-early strength cement (ASTM Type V) was used. Natural river sand, with a characteristic diameter of 4.8 mm, was used as the fine aggregate. The polypropylene fibers had a length of 12 mm and a diameter of 18 microns. The proportion of fibers used was fixed in 3 kg per cubic meter of concrete.

2.2 Experimental Variables and Mix Proportions

The experimental matrix designed contained, as variables, the potential compression strength of a concrete made with the cement paste or mortar tested, the maximum temperature of exposure, and the type and characteristics of fiber addition. The levels adopted for each one of them are summarized in table 1.

Table 1 - Experimental matrix

Specimen Type	Level of Resistence [Mpa]	Exposure Temperature [°C]				
		23°C	400°C	800°C		
Past	20	Without Fiber				
	50	Without or 0,007 kg/m ³				
Mortar	20	Without Fiber				
	50	Without or 0,007 kg/m ³				

The level of resistance of the HDC was limited to 50 MPa to represent the requirements that are being used today to produce durable concrete in current projects in Brazil. To allow the combination of all these variables in their various levels, three mix proportions were established. These are summarized in table 2.

Group		Cement Paste			Mortar	
Group	NSC	HDC	HDC-PF	NSC	HDC	HDC-PF
Cement (kg/m ³)	10	1	1	0.5	0.5	0.5
Water (kg/m ³)	0.57	0.22	0.22	0.35	0.15	0.15
Natural Quartz Sand (kg/m ³)	***	***	***	1690	800	800
Superplasticizer (kg/m ³)	***	0.01	0.01	***	0.005	0.005
Polypropylene Fiber (kg/m ³)	***	****	0.007	***	***	0.007

Table 2 - Mix proportions of the specimens.

NSC: normal density concrete HDC: high density concrete HDC-PF: with polypropylene fiber.

2.3 Test Method

Figure 1 shows the $10 \times 10 \times 60$ mm molds used to make the cement paste and mortar samples for the XRD, SEM, DTA, and dilatometry tests.

All specimens were cured during 7 days in a controlled environment set at 23°C \pm 2 °C and 98% relative humidity. After this initial period, they were stored in a controlled environment set at 23°C \pm 2°C and 70% R.U. for 21 days, allowing them to achieve moisture equilibrium with the environment, in order to reduce their humidity content. This care was taken because, otherwise, the specimens would be more susceptible than normal concrete elements to the deleterious effects of heating, given the high moisture content. At 28 days, the specimens were prepared for the heat exposure in ovens.



Figure 1 - Molding of the cement paste and mortar samples.

After the exposure, the tests were conducted. In the next sections each one of these tests is briefly described, highlighting the expected contributions that they might provide to detect and quantify the alterations that occur in the microstructure of concretes subject to sudden temperature elevations, which will in turn affect the macroproperties and the structural behavior.

2.4 X-Ray Diffraction (XRD)

XRD tests lead to the identification of the crystals present in the structure of the material, allowing the monitoring of the crystalline changes that occur when the samples are submitted to different levels of temperature.

This type of test allows a qualitative analysis of the material and is based on the physical phenomenon of diffraction, which occurs due to the phase difference between two or more waves that go through different optical ways. The incoming x-ray photons, when reaching a crystal, are diffracted by the crystalline structure and, subsequently, picked up and processed by electronic circuits, being the resulting signal plotted in a graphic, called difratogram, where the vertical axis shows the radiation intensity, and horizontal axis the angle of diffraction (2 θ). Each crystalline arrangement produces peaks in different angles, allowing the identification of the crystal present in the material. The equipment used in the test was a Phillips X´Pert MPD, which operates with a copper target. The data analysis was performed using the Phillips X´Pert Graphics Identify and the Profit Profile softwares.

This method has the advantage of not being destructive and requiring only a little amount of material. To perform it, the samples must be grinded and sifted in a sieve 200 mesh, with the passing particles being used in the analysis.

2.5 Scanning Electron Microscopy (SEM)

The Scanning Electron Microscopy (SEM) is one of the most versatile and widely used investigative tools for microstructural analysis, since it allows the study of both the morphology and the composition of biological and physical materials.

By scanning an electron probe across a specimen, high resolution images of the morphology or topography of the sample, with great depth of field, at very low or very high magnifications can be obtained. Compositional analysis of a material may also be made by monitoring secondary X-rays produced by the electron-specimen interaction. Thus detailed maps of elemental distribution can be produced from multi-phase materials or complex materials. Characterization of fine particulate matter in terms of size, shape, and distribution as well as statistical analyses of these parameters, may also be performed.

2.6 Differential Thermal Analysis (DTA)

Differential thermal analysis (DTA) involves heating a material at a controlled rate to a pre-determined temperature and comparing any emissions of heat (exothermic) or absorptions of heat (endothermic) from the material in comparison with a passive material such as alumina. It is useful for determining the precise temperature at which chemical or physical transformations have occurred, such as melting, sublimation and glass transitions or identifying a phase change or crystal

transitions. The samples are ground and drizzled in sieve 120 mesh, being the obtained powder heated up to 800° C, at a rate of 10° C/min.

2.7 Dilatometry

This is thermo-analytical method for measuring the thermal behavior of materials over a controlled temperature regime, since the resulting expansion or shrinkage is dependent on the characteristics of the material. The test was conducted until the temperature reached 800°C, using a constant heating rate of 10°C/min, with a plateau of 30 min at the final temperature. The used equipment was a Harrop dilatometer. The specimens tested were small bars with 10x10x60 mm.

2.8 Exposure Technique

The choice of exposing the specimen in ovens was made because the development and control of a real fire is a complex operation and the temperature patterns are difficult to predict and model, since the number of variables involved is very great and their behavior is not easily determined. The simulation of the effects of the exposure to high temperatures using high capacity furnaces was therefore considered as a reasonable strategy for the research purposes. Therefore, electrical ovens were used for heating the test specimens.

The heating rate employed was 8°C/min, as adopted by other researchers [10] [9]. Each batch was kept at the maximum temperature level (400°C or 800°C) for 30 minutes. After this interval, the oven was turned off and left to cool until it was possible to open the doors. The specimens were then taken out of the furnace and slow cooled in air until reaching room temperature. After 24 hours of the exposure, both the control specimens and the fire exposed ones were prepared for the tests.

3 Data Analysis

3.1 X-Ray Diffraction (XRD)

A comparative analysis of the difratograms obtained at ambient temperature, 400°C e 800°C leads to the identification of certain milestones that can be used to check how far the heating process affected the concrete microstructure.

At ambient temperature, the major crystalline phases identified in the cement paste samples were traced back to ettringite [Ca₆Al₂(SO₄)₃(OH)₁₂.26H₂O], calcium hydroxide [Ca(OH)₂], hydrated calcium silicates [C-S-H], calcite [CaCO₃] e larnite [Ca₂SiO₄]. On the samples exposed to 400°C, the ettringite phase was not noticeable anymore, and the main peak of calcite was less strong. The exposure to 800°C produced a great reduction in the peaks of calcium hydroxide and calcium silicates, which can be associated with the dehydration process. The degradation of C-S-H could be linked to a progressive increase in the peak of larnite. Meanwhile, the dehydration of the calcium hydroxide was related to the increase in the amount of calcium oxide [CaO]. This provides evidence of the loss of gel and chemically combined water at this temperature. These changes greatly affect the compressive strengths of the material, as reported by [14], which can be reduced in up to 85%.

It is believed that the relationship between crystalline changes and strength loss can be used to determine the mechanical damage in concrete structures exposed to extreme temperatures, as during a fire. The collection and analysis of powder samples by XRD can provide valuable information about the temperature of exposure and degree of damage. The calcium hydroxide is one of the most important markers in this case, since it degrades at around 400°C and has completely disappeared at temperature close to 500°C, temperatures around what the strength losses start to become more significant.

3.2 Scanning Electron Microscopy (SEM)

Figure 2 presents the internal portions of some concrete samples not affected by fire or heat exposure. It is possible to verify that the aspect of the samples are typical of good quality, high density concretes, marked by a high quantity of ettringite and calcium hydroxide dispersed among a sound C-S-H matrix.



Figure 2 - Microstructural aspect of concrete samples exposed to 800°C. (a) sample from the experimental program (b) sample studied by carvalho [15].

For comparison, figure 3 shows some aspects of the microstructure of concrete paste samples affected by heat exposure over 800°C. It was possible to identify the lack of cohesion and the friable, porous appearance of the C-S-H matrix and to identify some altered portions of the samples.

3.3 DTA

The graph in figure 4 shows the observed behavior of concrete pastes during the DTA tests. For temperatures between 40 and 105° C, it can be noticed the effects of the decomposition of the ettringite (which occurs circa 50° C) and of the removal of the gel and capillary water.

At 105°C, the dehydration of the calcium silicate hydrates (C-S-H) starts, which is accompanied by a gradual loss of mass, phenomenon that is extended until temperatures above 800°C. At 490°C, an endothermic reaction takes place, causing the appearance of a large drop in the curve of mass loss. This reaction is attributed to the dehydration of calcium hydroxide crystals (Ca(OH)₂), as confirmed by the X-ray diffraction results. At temperatures near 800°C, another endothermic pick was registered, representing the loss of carbon dioxide of the calcium carbonate formed in the dehydration of calcium hydroxide. The bottom part of the figure shows an exothermic pick at 310°C, originated by the pyrolysis of the polypropylene fibers.



Figure 3 - Microstructural aspect of concrete samples not exposed to high temperatures.(a) matrix sample from the experimental program (b) sample studied by Carvalho [15] (c) detail of altered portion of $Ca(OH)_2$ crystal.



Figure 4 - Thermograms of concrete pastes (a) normal strength concrete, (b) high density concrete high and (c) high density concrete with polypropylene fiber.

3.4 Dilatometry

As can be seen in the dilatogram obtained in the fig. 5(a), in normal strength concrete (NSC) paste samples, an initial expansion was observed until approximately 110°C, during which the capillary water in the smaller pores begins to evaporate, causing shrinkage.

The effect continues until the maximum temperature reached in the test, 800°C, due to the gradual loss of adsorbed and interlamellar water and, finally, from the loss of the chemically combined water, mainly from the calcium hydroxide.

In samples of high density concrete (fig. 5b and 5c), it can be noticed that the specimen shrinks up to 570°C, where it begins to expand again. Between 500°C and 570°C there is a large drop in the curve, due to dehydration of the calcium hydroxide. After 570°C, most of the CH seems to have already decomposed, and the loss of the little water left is not able to compensate the dilation of the sample.

In all cases, during the cooling period, the sample retracts, as expected. However, the coefficient of thermal retraction is smaller than the one observed during heating, due to slow and partial re-hydration of some phases like C-S-H and calcium hydroxide.

Figure 5c shows the results for a concrete sample with polypropylene fibers. It follows a similar pattern as the sample of high density concrete, presenting a slightly higher retraction due to larger density of the fiber in relation to the water.

Figure 6 shows dilatograms of mortar specimens. In all graphics, we noticed the evaporation of the free water, provoking a small change in the curve of expansion of the samples at temperatures near to 100°C. There is no shrinkage, as observed in the paste samples, due to the presence of the sand, which avoids the decrease of the volume.

At temperatures close to 550°C, a large drop is observed in the expansion curve, due to change of the quartz phase (from a to β). After 700°C, the volume of the sample stays constant in relation to the temperature, due to an equilibrium between the loss of carbon dioxide by the calcium carbonate and the specimen natural expansion.

In the cooling phase the β -quartz transforms back into aquartz and a similar curve is registered. Again, during cooling, there seems to be a partial re-hydration of some compounds.

4 Discussion

The results of the experimental program indicate that, as expected, normal strength concretes have an acceptable behavior in face of heat exposure, while more dense concretes show a greater susceptibility to heat damage. This tendency emphasizes the importance of undertaking research about the subject, aiming to determine how to predict and control heat deterioration, since the strength loss can be quite significant.



Figure 5 - Dilatograms of concrete pastes (a) normal strength concrete, (b) high density concrete high and (c) high density concrete with polypropylene fiber.

The analysis of the data collected point out that, for normal strength concretes (NSC), where the porosity is already great, the additional space created by the melting of polypropylene fibers at high temperatures is not necessary to reduce pore pressure, and the presence of the fibers just reduces the strength after exposure, although it increases the resistance of the specimens without exposure. For high performance concretes (HPC), the addition of polypropylene fibers reduces the heat damage of specimens exposed at 400°C and 800°C, indicating that the melting of the fibers creates extra space and reduces the pore pressure.

5 Conclusions

In summary, the preliminary data collected in this research indicates that the use of polypropylene fibers can be potentially useful to control the deterioration due to heat exposure of concretes with dense microstructures and that microstructural analysis can be a useful tool to accompany the degradation or estimate the amount of damage suffered by concrete when exposed to fire.



Figure 6 - Dilatograms of the concrete paste samples from: (a) normal strength concrete, (b) high density concrete and (c) high density concrete with polypropylene fiber.

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