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Mechanical Behavior of High-Performance Concrete under Thermal Effect

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Abstract

Several studies on the behavior of concrete at high temperatures are the subject of recent concerns, following the latest fires in various European tunnels. In these extreme conditions, significant degradations of concrete structures can be observed (peeling, cracking, breaking of the structure). A priori prediction of concrete behavior during this type of stress is therefore essential and is not possible without a good understanding of the different mechanisms of concrete damage at high temperatures. These mechanisms are often considered as the main causes of cracking and peeling of concrete subjected to high temperatures. Therefore, a fire can strongly modify the behavior of concrete and jeopardize the stability of concrete. In case of fires, it is necessary to know the instantaneous and residual behavior of concrete subjected to temperatures up to 1000°C. In this work we present a study of the mechanical performance of high-temperature high-performance concrete (HPC) exposed to four maximum temperatures, 200, 400, 600, and 900°C. The results obtained show that the mechanical strength at 28 days increases with the degree of temperature compared with that measured at 20°C. On the contrary, a clear decrease is observed between 600 and 900°C.

Keywords: HPC, high temperatures, mechanical strength, porosity, mineral additions

1. Introduction

High-performance concrete (HPC) is a concrete mixture, which possesses high durability and high strength when compared to conventional concrete [1]. This concrete contains one or more of the cementitious materials such as fly ash, silica fume, or ground granulated blast-furnace slag (GGBFS) and usually a super plasticizer. High strength of concrete is achieved by reducing porosity, inhomogeneity, and micro-cracks in the hydrated cement paste and the transition zone. Consequently, there is a reduction of the thickness of the interfacial transition zone in high-strength concrete. The densification of the interfacial transition zone allows for efficient load transfer between the cement mortar and the coarse aggregate, contributing to the strength of the concrete. For very high-strength concrete where the matrix is extremely dense, a weak aggregate may become the weak link in concrete strength [2].

The importance in the behavior of concrete at a high temperature mainly results from the many cases of fires taking place in buildings, high-rise buildings, tunnels, and drilling platform structures [3]. During a fire, the temperature may reach up to

1100°C in buildings and even up to 1350°C in tunnels, leading to severe damage in a concrete structure. When concrete is heated under conditions of fire, the increase in temperature in the deeper layers of the material is progressive, but because this process is slow, significant temperature gradients are produced between the concrete member's surface and core inducing additional damage to the element. Fundamental issues related to the impact of high temperature on concrete involve identification of the complex changes that take place in concrete while heated. This concerns both the physical and chemical changes taking place in the cement matrix, as well as the phenomena involved in mass movement (gases and liquids) [4].

The analysis is complicated due to the fact that cement concrete is a composite consisting of two substantially different constituents: cement paste and aggregates. The effects of the various changes taking place in heated concrete are the alterations of its physical, thermal, and mechanical properties [5].

Many research have demonstrated that changes in the strength of concrete as a function of temperature are related to, inter alia, concrete composition, the type of aggregate used, the water/cement ratio, the presence of pozzolana additives, etc. Important factors are also the rate of heating and the time of concrete exposure to high temperature.

2. References

2.1 Effect of curing temperature on cement hydration

Portland cement hydration is affected by many variables, including chemical composition, the water/cement ratio, the presence of mineral additions, and fineness. Yet another variable, however, is regarded to play a key role, bearing on early hydration kinetics and the properties of the hardened cement paste: that variable is temperature [6].

Parry-Jones et al. studied hydration in cement pastes cured for up to 31 days at temperatures ranging from 20 to 80°C. These authors calculated the degree of hydration with ²⁹Si MAS NMR. For pastes hydrated from 20 to 55°C, strength and degree of hydration were linearly correlated, but for a given degree of hydration, the pastes hydrated at 80°C had perceptibly lower strength than the pastes cured at lower temperatures [7]. Curing temperature affects both the inner and outer calcium silicate hydrate (C-S-H) gel structure. Regourd and Gautier reported that the outer C-S-H formed at 80°C was much more fibrous, exhibiting morphology reminiscent of pastes hydrated with calcium chloride accelerators [8].

Kjellsen et al. reported thicker inner C-S-H rims than pastes hydrated at low temperatures. Such brightness is associated with several developments: an increase in the average atomic number, a decrease in the water content or both, or high-sulfate concentration in C-S-H gels formed at high temperatures [9, 10].

Temperature affects the interaction between additions and cement compounds. Alite hydration was found to rise sharply from a very early age in the presence of slag and volcanic ash but much less abruptly when fly ash was added. Belite hydration was delayed in the presence of fly ash at 40 and 60°C but was somewhat enhanced at lower temperatures [11–13].

Escalante et al. noted that for all blended cement pastes, an increase in curing temperature led to greater porosity, with the most prominent differences appearing between 10 and 60°C. The same authors confirmed that in fly ash-based pozzolanic cement pastes, Ca(OH)₂ was almost absent in pastes cured at 60°C, whereas at 10°C, clusters of Ca(OH)₂ were visible in the microstructure [12].

2.2 Effects of high temperature on the residual performance of concretes

The increase in temperature results in water evaporation, C-S-H gel dehydration, calcium hydroxide and calcium aluminate decomposition, etc. Along with the increase in temperature, changes in the aggregate take place. Due to those changes, concrete strength and modulus of elasticity gradually decrease, and when the temperature exceeds ca. 300°C, the decline in strength becomes more rapid. When a 500°C threshold is passed, the compressive strength of concrete usually drops by 50–60%, and the concrete is considered fully damaged [14].

Many researches showed a heating of cement paste results in drying. Water gradually evaporates from the material. The order in which water is removed from heated concrete depends on the energy that binds the water and the solid. Thus, free water evaporates first, followed by capillary water and finally by physically bound water. The process of removing water that is chemically bound with cement hydrates is the last to be initiated. The mechanical properties of cement paste are strongly affected by chemical bonds and cohesion forces between sheets of calcium silicate hydrate (C-S-H) gel. It is assumed that approximately 50% of cement paste strength comes from cohesion forces (important C-S-H gel sheet area); therefore, the evaporation of water between C-S-H gel sheets strongly affects the mechanical properties of the cement paste [15, 16].

According to the work of Verbeck et al. [17], in the process of simultaneously exposing the material to high pressures and temperature, it may activate the changes in the microstructure of hydrates and often increases cement paste strength. The nature of the phase changes will depend upon the mineralogical composition of the cement, its C/S ratio (mol of lime per mol of silica; CaO/SiO_2), the amount of fine particles (quartz or silica fume), and the temperature and pressure levels that have been reached. Heating the cement paste with a C/S ratio around 1.5 to temperature above 100°C produces several forms of calcium silicates, in general highly porous and weak. When the C/S ratio is close to 1.0 and the temperature is above 150°C, a 1.5–1.0 to be more gel can form. At temperature between 180 and 200°C, other silicates such as xonotlite and hillebrandite may be formed [17].

During heating, ettringite decomposes first, even before the temperature reaches 100°C. C-S-H gel dehydration is progressive and takes place from the very beginning of material heating. In this state the structure of the cement paste is partially damaged due to dehydration at a temperature of 105°C, which is standard for the drying of materials. As soon as cement paste is heated to temperature of 500–550°C, the portlandite content rapidly decreases, as it decomposes according to the following reaction:

At 550°C, the peak corresponding to the decomposition of the free limestone $\text{Ca(OH)}_2\text{CaO}$ [18].

Hager noticed that the CaO created in this reaction makes the elements made of the portland cement practically redundant after cooling. The dehydration process of the C-S-H gel reduces its volume, which in turn increases the porosity of the cement matrix. Moreover, during heating, the cement paste experiences a slight expansion up to temperature of approximately 200°C although the intense shrinkage begins once this temperature is exceeded. This significantly contributes to the porosity evolution of the cement paste. Due to heating total pore volume increases, as does the average pore size [15].

Reinforced concrete structures exposed to the environment require durable concretes to provide long-lasting performance with minimal maintenance. Low

permeability is an important characteristic of durable concretes and may be obtained by lowering the water/cementitious material ratio (W/C) and using pozzolans (fly ash and silica fume) or slag as a portion of the cementitious material [15, 18].

2.3 Effect of temperature on durability of slag concrete, fly ash concrete, and silica fume concrete

During the production of cement, a significant quantity of CO₂ is released into the atmosphere. It is estimated that the production of each ton of portland cement releases approximately 1 ton of CO₂ gas. The world production of clinker accounts for about 7% of the total CO₂ emissions. The use of these additions as cement replacement decreases the amount of clinker required, resulting in a limiting gas emissions of CO₂ and dust in the atmosphere. The cement replacement does not only reduce the production costs but also address some environmental issues providing the enhanced concrete performances (better workability, lower hydration heat, and good durability) [19].

High-performance concrete (HPC) has been used more widely in recent years due to the increasing demand for durable concrete, thus to extend service life and reduce maintenance fee of concrete structures. HPC is known as a concrete which has a compressive strength over 60 MPa [20]. The pozzolana addition influences positively the compressive strength that could be easily increased up to 150 MPa. Pozzolanic materials are very important in the production of HPC. Further, HPC may contain materials such as silica fume, fly ash, ground granulated blast-furnace slag, natural pozzolana, chemical admixtures, and other materials, individually or in various combinations.

In slag cements, clinker is the principal activator of the binder. However, the first produced hydrates will be those of the clinker; C-S-H and Ca(OH)₂ uniformly cover the grains with the slag and the clinker; there after the lime excess activates the hydration of the slag with a texture of C-S-H similar to that of cements; it results in calcium silicate hydrates and hydrated tetra-calcium aluminates. Other research undertaken on the subject reported that the more fine the slag is, the better is its performance [21].

Tebbal's research indicated that the compressive strength is higher, when the cement is replaced by 5% silica fumes mainly, the combined mixture of 5% of silica fume and 10% of slag, at a maximum value of 170 MPa [22].

During the hydration reaction between portland cement and water, a cementitious gel and lime are formed. Pozzolanic materials react with this lime in the presence of moisture to form additional cementitious gel. Slags also submit to this pozzolanic reaction. This reaction leads to a reduction in the permeability of the concrete and an increase in its strength [23].

Traditionally, slag, silica fume, and fly ash were used in concrete individually. Nowadays, due to the improved access to these materials, concrete producers can combine two or more of such materials to optimize concrete properties at fresh state (workability) and hardened state (strength and durability); a reduction in the rate of penetration of chloride ion concrete reduced the potential of chloride-induced corrosion [20].

Ravindrarajah et al. stated that concrete consists of discrete and interconnected pores of a variety of sizes and shapes and their distribution depends on the binder material type. The refinement of pore size as well as grain size of concrete is attained by the use of fly ash, slag, and silica fume due to its fineness, pozzolanic, and cementitious property. Free water in the gel causes capillary cavities, and combined water in hardened cement paste improves the hydration process. Combined water can be dehydrated at 1000°C only due to its stability [24].

According to Bingöl et al., there are no significant losses in the compressive strength of lightweight aggregate concrete observed between 150 and 300°C. The initial strength loss is important for all mix groups at 750°C. The heating duration does not affect the strength loss significantly, but high temperature is a significant parameter of strength loss. Stephen S. Szoke stated the degradation of pavement color resembles the degradation of cement paste properties [25].

The effect of thermal cycles on the compressive strength of high-volume fly ash concrete has been studied by Srinivasa Rao et al. They are confirmed that the structural elements when exposed to solar radiation, the thermal gradients in the elements are influenced by the degree of humidity. In that way the elements of structures undergo one thermal cycle per day and also are exposed to peak value of heating period and cooling period [26].

Noumowé mentioned that thermal gradients were very significant and generating high compressive stresses at the specimen surface during the heating tests at 210 and 310°C. This thermal loading causes stresses which are accompanied by the degradations of cement paste due to abrupt changes in volume further leading to a damage of the concrete. Contrasting conditions observed while cooling the temperature in the center of specimen are more in the surface. This condition causes compressive stresses at the center and tensile stresses on the surface. Compressive strength is decreased with the increase in temperature. However, losses were very less between 20 and 110°C. There was an appreciable reduction in the strength above 210°C, and loss in compressive strength was 8% [27].

Falade concluded that the compressive strength of concrete is reduced with increase in water/cement ratio and increase in temperature but increased with increase in curing period. The bond between the concrete within matrix decreases as the temperature increases. The loss in strength of specimens is in between 24 and 40% at a maximum temperature of 800°C/h., which is influenced by the mix proportion and curing age. Lightweight concrete consists of periwinkle shells which are the only appropriate material for structures that will be exposed to temperature lower than 300°C [28].

Siddique et al. concluded that concrete with GGBFS can be used in constructions exposed to elevated temperatures. The degradation of mechanical properties of concrete is less between 27 and 100°C. The values of compressive strength, split tensile strength, and modulus of elasticity are reduced lower than 40% after exposing to a temperature more than 350°C. The loss in mass is not very important at temperatures between 200 and 350°C. GGBFS may contribute to some extent to the residual compressive strength of concrete at elevated temperatures. Similar findings were observed by Siddique and Kaur [29].

Pathan et al. stated that in practice, at a temperature of 250°C, calcium hydroxide starts to dehydrate generating more amount of water vapor. Further, significant reduction in their compressive strength was observed at temperatures in the range of 300 and 600°C [30].

Seshagiri Rao et al. stated that a considerable change in physical structure and chemical composition appears when concrete is exposed to high temperature. Above 100°C, dehydration of water in C-S-H gel is important. This is added to thermal expansion of aggregates which causes the increase in internal stresses at 300°C, and further micro-cracks are developed. $\text{Ca}(\text{OH})_2$, the product of hydration of cement paste, separates into CaO and H_2O at 400–600°C subsequently; shrinkage of concrete occurs [31].

Chowdhury stated that at high temperatures, the loss in compressive strength and tensile strength was observed for all three concrete mixes of 100, 80, and 40 MPa. The loss in strength for 80 MPa mix was about 44% when exposed to 400°C for 12 h. At 60°C, the loss in tensile strength for 80 MPa mix was about 18% for 72 h of exposure. A nonlinear relationship was observed between weight loss and maximum temperature, but loss was least in the case of the highest strength

mix for every temperature and duration of high temperature exposure. An easy way to comply with the conference paper formatting requirements is to use this document as a template and simply type your text into it [32].

According to Santosh Kumar et al. [33], the materials like pozzalonas may be natural and artificial like industrial wastes or by-products which require less energy to make fine particles. These materials exhibit cementitious properties and combine with calcium hydroxide producing cementitious material [34].

Gowri et al. presents the results of experimental studies conducted on performance of High Volumes of Slag Concrete (HVSC) exposed to elevated temperatures up to 600°C. In HVSC, 50% of cement is replaced with Ground Granulated Blast Furnace Slag (GGBS). In this experimental studies, HVSC of 100 mm cubes are cast and tested for various water/binder ratios ranging from 0.55 to 0.27. The specimens are exposed to elevated temperatures of 200°C, 400°C and 600°C for 4, 8, 12 hours. Result of compressive strengths and weights of cubes after expose to high temperature are estimated. Percentage loss in compressive strengths and weights are also evaluated. The results illustrate that the loss in compressive strength and weights are more for higher temperatures for longer duration for higher water/binder ratios [35].

According to Jawed et al., percentage loss of compressive strength is higher with an increase amount of fly ash in concrete samples, i.e., for 20% fly ash concrete. This is due to high impermeability and moisture gained in longer curing period resulting in high pore pressure but low initial strength gain [36].

In 2004, Yüzer et al. carried out a study on the effects of fire, and extinguishing on the properties of concrete, mortars with and without silica fume were exposed to different temperatures, such as 100, 200, 300, 600, 900, and 1200°C and cooled slowly in the air and fast in water in two groups. Flexural and compressive strength tests were performed on the samples which were cooled up to room temperature, and changes in compressive strength in color were determined by Munsell color system. High temperature has caused damages to decrease in mechanical strengths at 600°C. Researchers observed that the changes in color hue component and the compressive strength have similarities. Test results show that residual color changes in mortar can give an idea about the effect of high temperatures on mechanical properties of mortar during a fire [37].

Ahmad's research includes an experimental investigation to study the effect of high temperatures on the mechanical properties of concrete containing admixtures. A comparative study was conducted on concrete mixes, reference mix without an additive, and that with an admixture. Concrete was exposed to three levels of high temperatures (200, 400, 600°C), for duration of 1 h, without any imposed load during the heating. Super plasticizer, plasticizer, retarder, water-reducing admixture, an accelerator, and an air entraining admixture, five types of admixtures, were used. Mechanical properties of concrete were studied at different high temperatures, including compressive strength, splitting tensile strength, modulus of elasticity, and ultimate strain. Test results showed a reduction in the studied properties by different rates for different additives, and for each temperature, the decrease was very limited at a temperature up to 200°C but was clear at 400–600°C [38].

3. Tebbal et al.'s research on the effect of temperature on high-performance concrete

This study examines the effect of the additions of silica fume and super plasticizer on the mechanical performance of high-performance concretes at high temperatures. The tested concretes are formulated with 5% silica fume and two dosages of super plasticizers in the ratio of (2%, 2.5%) the weight of cement after having been exposed to four maximum temperatures, 200, 400, 600, and 900°C, without any imposed

load during the heating. The results obtained show that the mechanical resistance at 28 days increases with the degree of temperature compared to that measured at 20°C. On the contrary, a clear decrease is observed between 600 and 900°C. However, material composition seems to have great influence on the mechanical strength.

In the present study, three factors were studied at high temperatures:

- The influence of super plasticizer on the mechanical strength
- The additions of silica fume on the behavior of HPC
- The internal structure, X-ray diffraction (XRD) patterns and gravimetric and differential thermal analyses (ATG and ATD) of concrete subjected at high temperature

3.1 Materials and methods

The portland cement-type CEM II/A 42.5 from Hammam Dalâa local factory was used in this experimental study. The used cement type has an absolute density, consistency, and fineness values of 3.1 g/cm³, 28%, and 4000 cm²/g, respectively. The chemical composition of the cement is shown in **Table 1**.

The silica fume is obtained from GRANITEX in Algeria region. It results from melting the silicon and ferrosilicon. The reduction of high-purity quartz to silicon at temperatures up to 2000°C produces SiO₂ vapors, which oxidize and condense in the low-temperature zone to tiny particles consisting of noncrystalline silica [39]. The physical properties and particle size by laser granulometer (Mastersizer 2000) of silica fume are shown in **Table 1** and **Figure 1**.

The natural fine aggregates used were dune sand with particles ranging from 0.08 to 5 mm in size, with a fineness modulus, Mf, of 2.44. This natural sand was

Cement (%)				Silica fume (%)		
SiO ₂	20.7			>85		
Al ₂ O ₃	04.75			—		
Fe ₂ O ₃	03.75			—		
CaO	62.92			—		
MgO	01.90			—		
SO ₃	1.98			<2.5		
CL	—			<0.2		
Mineralogical composition of cement						
Item content	C ₃ S	C ₂ S	C ₃ A	C ₄ AF		
	59	14	6	10		
Physical properties of silica fume						
Particle size	Density		Specific surface		Moisture by storing at 105°C	
<0.1 microns	<0.5		>15 m ² /g		<1%	
Characteristics of the chemical admixture						
Super plasticizer	Form	Color	PH	Density	Chlorine content	Dry extract
Medaplast SP40	Liquid	Brown	8.2	1.2 ± 0.01	<1 g/l	40%

Table 1.
The chemical and physical properties of cement and silica fume [18].

Figure 1.
Particle size distributions of silica fume [18].

taken from the region of Bou Sâada (250 km east of Algiers). The sieve analysis is performed according to the European standard NF EN 933-1. The mineralogical composition determined by X-ray diffraction shows that the siliceous sand is more than 95% of quartz and calcite traces.

The coarse fraction of aggregate is gravel (G1) of size 3/8 mm and gravel (G2) of size 8/15 mm. The adjuvant used is a super plasticizer which is highly water reducing (Medaplast SP40). It is a solution of pH = 8.2 and a density of 1.22, with 40% of solids. Its normal use scale is fixed by the manufacturer’s recommendation which is between 0.6 and 2.5% of the cement weight. The tap water used all through the study from mixing was taken from the laboratory of civil engineering.

3.2 Mixture design

Fresh concrete mixes were prepared in a modified laboratory mixer; the mixing procedure is explained in **Table 2**. The concrete specimens were preserved in their molds in a wet place at a temperature of 20°C and 95% relative humidity (RH) during 24 h. After demolding, they were immersed in water at 20°C until the age of testing. The physical and mechanical characteristics of the concretes with and without the addition of silica fume have been compared. The silica fume is added at a dosage of 5% cement weight and the super plasticizer at 2 and 2.5%, respectively. The final compositions of high-performance concrete (HPC), after optimization, are reported in **Table 3** [19, 22].

The following acronyms will be used henceforth:

CR_{2.5}: Concrete without silica fume and 2.5% (by weight of cement) of the chemical admixture.

HPC_{2.5}: Concrete dosed at 5% of silica fume and 2.5% of the chemical admixture.

HPC₂: Concrete dosed at 5% of silica fume and 2% of the chemical admixture.

Time,s	Mixing procedure
60	Mixing of aggregates, silica fume, cement
30	Addition 100% of water and a third of the volume of super plasticizer
180	Mixing
30	Addition of the remaining super plasticizer
60	Mixing

Table 2.
Mixing procedure [18].

Mix (kg/m ³)	Cement	Sand	Gravel (3/8, 8/15)	Water	Additions	
					Silica fume (%)	Super plasticizer (%)
CR _{2.5}	400	662	1090	220	—	2.5
HPC _{2.5}	444	645	1042	119	5	2.5
HPC ₂	444	662	1042	122	5	2

Table 3.
Compositions of concrete with and without silica fume [18].

After 28 days, specimens with dimensions (100 mm × 100 mm × 100 mm³) are dried in an oven (at 100 °C), until stabilization of their mass. All specimens are subjected to high temperatures, 200, 400, 600, and 900°C, according to the time-temperature schedule of ASTM E 119-00 [39]. After cooling, they were subjected to compression tests.

The slump values were obtained for all three mixtures according to the NF EN 12390-4 and EN 12390-5 [40, 41]. The axial compressive strength was tested at 28 days according to NF EN 12390-4 [40] for the concrete at 20°C that was not subjected to high temperatures.

The protocol of porosity accessible to water conforms the recommendations of AFREM group [42]. The open porosity allows us to appreciate the evolution of hydration and structuration of hydrated products; this is a key for identification of the most sustainable concrete [43]. The test pieces for testing of water porosity are dried in an oven at a temperature of 100°C to constant weight and then returned to room temperature in a desiccator.

The porosity test is carried out on test pieces of dimensions 10 × 10 × 10 cm³, by applying the following steps:

- Dry in an oven at 105°C the sample for at least 24 h until obtaining a constant mass. This mass is denoted as “A.”
- Immerse the sample in water for 24 h.
- Heat to boil for 5 h, and then weigh the sample in air (i.e., the weight is denoted as “C”).
- Then hydrostatic weigh (the weight is denoted as “D”).

The porosity was calculated by the formula:

$$P \text{ (%) } = \frac{C - A}{C - D} \times 100 \tag{1}$$

Phase compositions of these concretes were investigated on the fine powders using X-ray diffraction method. The powder samples of concrete heat-treated aggregates at 20, 600, and 900°C were collected after abrasion. X-ray diffraction analysis was performed on an X-ray diffractometer (X’Pert) coupled to a computer system. The essential purpose of this analysis is to identify the different phases of crystal present in a sample.

Gravimetric and differential thermal analyses (ATG and ATD) make it possible to quantify portlandite; these techniques are used to characterize degradations.

3.3 Results and discussion

3.3.1 High-temperature effects on surface properties of concrete

As seen in **Figure 2**, some color changes happen on the surface of concrete samples due to high temperature effects. As a result of these color changes the range of temperature values can be certainly appraised. It can be seen that cracking, rupture, and color changes happened much more in the concrete samples exposed to 900°C than the ones with 400 and 600°C.

3.3.2 Loss of concrete's mass related to temperature

Figure 3 shows the evolution of mass loss during the heating cycle of the studied specimens. Before 100°C, the mass's exchange is very low. The mass loss in this temperature range corresponds mainly to the water's escape from concrete's pore, because the water chemically bound to the silicates becomes free. The chemical bonds in the C-S-H break, and the gel buildup water is released and can be evacuated.

Between 100 and 600°C, a strong mass loss for all concrete specimens was tested. Each concrete lost from 3 to 12% of its original mass. Most of the water in each concrete specimen evaporated during heating between 100 and 400°C. The increase is almost linear up to a temperature of 600°C. This is due to the evaporation of water and the progressive dehydration of C-S-H gel. Several authors, Noumowé et al., confirm that beyond 600°C, there is no more water in the concrete specimen [43, 44].

3.3.3 Result of the crushing of concrete after cooling

The compressive strength of all concrete mixtures at ambient temperature and after heating at 200, 400, 600, and 900°C is illustrated in **Figure 4**.

Figure 2.
Concrete samples exposed to high temperature [18].

Figure 3.
Evolution of mass loss related to temperature [18].

Figure 4.
Compressive strength after cooling relative the initial strength [18].

This figure clearly shows that the compressive strength of all concrete mixtures decreases at elevated temperature. According to the results obtained from the present investigation, the strength of concrete HPC₂ after heated at 200, 600, and 900°C decreases by 3, 11, and 78% of its unheated strength, respectively.

The concrete HPC_{2.5} exhibited the greatest loss in strength, about 4, 15.38, and 100% of its unheated strength when heated at 200, 600, and 900°C, respectively. We observe a stress peak at 400°C indicating a maximum value of the compressive strength of the concretes. The decomposition temperature of Ca(OH)₂ varies between 400 and 600°C. If static temperature conditions are maintained, Ca(OH)₂ will decompose at 400°C. This temperature seems to be the critical temperature for a portland cement concrete [45].

Kalifa et al.'s work has shown the role of water on the performance of HPC temperature. Pressure values observed in HPC of 100–4 MPa were only 3 cm deep from the heated side to the vicinity of 250°C [46]. This further explains the prestressing force caused by the water trough described by Burlion et al. [47].

After the evaporation of physically and chemically bound water, a pressure is built up which leads to an extensive inner cracking. This inner cracking is the main reason for the reduction in strength of all concrete mixtures. Apart from it, the cement paste contracts, and aggregate expands due to loss of water at higher temperature which leads to loss of the bond between paste and aggregates.

3.3.3.1 Influence of super plasticizer on the mechanical strength

At 900°C, we notice a low resistance for concrete with 2% adjuvant and a bursting of the concrete to those dosed of 2.5% super plasticizer (Figure 4). This can be explained by:

- The concretes with superplasticizer have high resistance. The role of the super plasticizer in the distribution of cement particles in improving the compactness of concrete is highlighted. The concrete mixes containing superplasticizer are affected by high temperatures especially at 600°C and above compared to those with less super plasticizer dosage.
- The decrease of resistance estimated at 78% for HPC with 2% super plasticizer exposed to a temperature of 900°C, on the other hand, is zero for ordinary concrete. During these processes, some cracks occur, and concrete is crumbled and becomes a porous material [48]. Aggregate's effect on concrete at high temperatures is related to their mineral structures. This process results in volume increase and damage [18, 19].

3.3.3.2 Influence of silica fume on the mechanical strength

In this part of the study, we want to highlight the influence of silica fume on the mechanical behavior of HPC. After the passage in the oven, the concrete specimens were cooled for 24 h in the laboratory, at a temperature of $20 \pm 5^\circ\text{C}$ before submitting to compressive strength test. The specimens of HPC exposed at high temperature are schematized as shown in **Figure 5**.

As expected, the replacement of cement by 5% of silica fume increased the compressive strength approximately 30% at 28 days. This is due to the reaction of the silica fume with calcium hydroxide formed during the hydration of cement that caused the formation of calcium silicate hydrate (C-S-H) as well as filler role of very fine particles of silica fume. In general, it can be concluded that concretes containing silica fume had significantly higher strength than that of CR concretes at room temperature. After exposure to 200°C, significant reductions occurred in the compressive strength of concretes without SF. Results showed the strength recovery of 18% for the concretes HPC_{2.5} after heating to 400°C when compared to 200°C. The compressive strength gains at 400°C are attributed to the increase in the forces between gel particles (van der Waals forces) due to the removal of water content [48].

In the range of 400–600°C, severe strength losses occurred in two concretes, HPC_{2.5} and CR_{2.5}. During exposure to high temperatures, cement paste contracts, whereas aggregates expand. Thus, the transition zone and bonding between aggregates and paste are weakened. After heating to 600°C, the compressive strengths of CR were lower than those of the concretes HPC_{2.5}. This is attributed to the presence and amount of silica fume in concretes that produced very denser transition zone between aggregates and paste due to its ultrafine particles as filler and its pozzolanic reactions.

Figure 5. Compressive strength of (HPC_{2.5}) and (CR_{2.5}) with temperatures [18].

At 600°C, the quick losses in compressive strength for HPC_{2.5} concretes are attributed to the dense microstructure in this type of concretes, which caused the buildup of higher internal pressure due to the water vapor transition of the inter-layer water. As a result, this process as well as chemical decomposition of hydration products causes severe deteriorations and strength losses in concrete after subjecting to high temperatures. It seems that the dosage of silica fume has no significant effect on the relative residual compressive strength at 200°C. However, between 200 and 400°C, the amount of 5% of silica fume has significant effects on the residual compressive strength. The greatest relative residual strength losses of concrete HPC_{2.5} and without silica fume were observed at 600°C, which were 24 and 2%.

Beyond 600°C, the concrete may lose the majority of these properties, i.e., there are properties that can cancel out, and one can say that the concrete has become weak.

When temperature increases beyond 400°C, the concrete strength decreases more rapidly due to the degradation of calcium silicate hydrate (C-S-H). The second phase of the C-S-H decomposes in the temperature range from 600 to 800°C forming β -C2S [16]. At a temperature of 900°C, the C-S-H breaks down completely. Therefore, the critical temperature for concrete ranges from approximately 400–900°C. In this range concrete loses most of its strength.

3.3.3.3 Porosity

The results of the porosity for the HPC after various heat treatments ranging from 20 to 900°C are shown in **Figure 6**.

- For all high-performance concretes, a monotonic and fairly regular increase in porosity with temperature is observed.
- For CR_{2.5}, the porosity values are higher than that of the HPC for all the temperatures.

Between 20 and 200°C, the porosity increases very little. The HPC shows a decrease of 0.26%. Kalifa explains that the decrease in the porosity of HPC is associated with the densification due to the complementary hydration of HSC.

Figure 6.
Porosity as a function of temperature [18].

Between 200 and 400°C, the porosity increases by 4.5% for the HPC. This growth is associated with the discharge of water, whether present in the water network or chemically bonded. Kalifa explains that the decrease in porosity of CR between 200 and 300°C compared to HPC is associated with densification due to complementary hydration and carbonation of portlandite under internal autoclaving conditions, that is to say, under a pressure higher than atmospheric pressure. On the other hand, this densification is not observable in the HPC which contains very little portlandite, thanks to the presence of silica fume.

At 600°C, the porosity value has substantially increased by 6.1% compared with that of 20°C. For the concrete CR_{2.5} and is almost 6.33% for the HPC. The evaluation of the porosity at 900°C is practically impossible as the test tubes that have undergone severe damage and have disintegrated.

Concerning the porosity of cement paste at high temperature, Piasta has shown that the porosity increases in a parabolic manner according to the temperature [49]. This increase, also noted in other works by Bazant et al., is accompanied by an increase in the average pore size and total pore volume. This is due in part to the internal fracture of the C-S-H gel structure during the dehydration process [49].

3.4 Internal structure, ATG, and ATD of concrete

Dehydration of the cement paste, thermal expansion and cracking, crystal processing, and mineral decomposition of aggregates are important reasons for the deterioration of concrete at high temperature.

Internal structure XRD patterns, ATG, and ATD of concrete subjected at high temperature are shown in **Figures 7** and **8**.

The analysis by X-ray diffraction is carried out in the physics laboratory of the University of M'sila by an X-ray diffractometer (X'Pert) coupled to a computer system. The essential purpose of this analysis is to identify the different crystalline phases present in a sample.

The analysis of the spectrum in **Figure 7** is used to report the following findings:

The diffractogram of a heated concrete (20°C) reveals the presence of portlandite, calcite, or α -quartz.

To a more severe heat treatment (400 and 600°C), all the peaks relative to the portlandite disappear (dehydroxylation between 400 and 600°C). This transformation was observed on ATD and ATG. Also, it does not detect the allotropic transformations of α -quartz in β -quartz between the diffractograms of 400 and 600°C since it is about a reversible transformation.

At higher temperatures (900°C), crystalline transformation of aggregates occurs, e.g., α - to β -quartz transformation in siliceous aggregates. Decarbonation of carbonates plays a prominent part if the concrete contains limestone aggregates.

Figure 7C shows that the peak intensity at the position that 2θ is 55°C increases. Therefore, it can be concluded that a new production was formed.

The results of the ATG and ATD powder of HPC at 20, 600, and 900°C are shown in **Figure 8**. A test sample of 200 mg of the concrete was analyzed according to linear heating from ambient temperature to 1400°C, with a speed of 10°C/min.

Six endothermic peaks were observed: 110–130, 180, 400, 450–550, 573, and 800°C. These thermal flux peaks are essentially related to the phase exchange temperatures of the different hydrates of the cement paste.

At a double peak of 110 and 130°C, the free water starts evaporating rapidly. In the temperature range from 80 to 150°C, dehydration of ettringite takes place followed by the decomposition of gypsum between 150 and 170°C [50].

In contrast, a small endothermic peak is observed at a temperature of 180°C; this peak indicates the dehydration of the calcium monocarboaluminate hydrate [51].

Figure 7. XRD patterns of powder of HPC at (A) 20°C, (B) 600°C, and (C) 900°C (notes: C, calcite; Q, quartz; and Br, brucite) [18].

Between 200 and 300°C, a so-called water plug develops in concrete pores, and there are slight variations in flux to the continuous dehydration of C-S-H [51].

At 400°C, a small peak was observed which we could not identify clearly the phase. A similar transformation was observed by Sha et al., on cement pastes [52].

These authors attribute this change of crystalline state or dehydration of a solid solution of Fe_2O_3 . But other sources [53] attribute this peak to the decomposition of brucite $[\text{Mg}(\text{OH})_2]$.

Between 450 and 550°C, the peak corresponds to the decomposition of the free limestone $\text{Ca}(\text{OH})_2 \rightarrow \text{CaO}$ [54].

At 573°C, the allotropic transformation of the quartz- α and quartz- β is accompanied by a phenomenon of expansion (cracking of the siliceous aggregates) [54]. Between 600 and 700°C, C-S-H decomposes and transforms into a new form of

Figure 8. ATG and ATD powder of concrete HPC at (a) 20°C, (b) 600°C, and (c) 900°C [18].

hydrates less rich in water and donation without it being formed of anhydrous compounds. These are mainly di-calcium silicates (β -C2S) and β -wollastonite (β -CS) [53].

The last peak coincides with the temperature of 800°C. It is well defined that in the temperature range from 700–900°C, the limestone decomposes, so this peak indicates the decomposition of calcium carbonates (CaCO_3), also known as “cal-cite,” by releasing lime accompanied by a release of CO_2 [5] according to the highly endothermic reaction which is as follows:

A quasi-linear decrease is observed up to 800°C, and the concrete exhibits a severe decrease in the density above 800°C. This decrease in density is related to two phenomena, i.e., complete dehydration and anhydrous formation, which take place only at temperatures in the region of 900°C.

4. Conclusions

From the study, one can conclude that, when using HPC specimen, the speed of temperature rising influences the drop in strength between 400 and 600°C.

- The critical temperature, which causes maximum attenuation of different properties (compressive strength, mass loss), is between 400 and 600°C.
- Beyond 600°C the concrete may lose the majority of these properties, i.e., there are properties that can cancel out; one can say that the concrete has become weak.
- The concretes with super plasticizer are affected by high temperatures especially at 600°C and above compared with less super plasticizer dosage.
- For a more resistant concrete, the addition of silica fume leads to lower resistance (24%) in the temperature range tested, between 400 and 600°C.
- The HPC specimens containing silica fume have high compressive stress compared to HPC specimen without silica fume (CR).
- Color changes were observed on concrete under the effect of high temperature.

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