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J.-C. Mindeguia, Izabela Hager, Pierre Pimienta, K. Mróz, Hélène Carré

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## Concrete properties at high temperature – complete characterization of a HPC concrete: M100

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## Concrete properties at high temperature – complete characterization of a HPC concrete: M100

Mindeguia J.C., Hager I., Pimienta P., Mróz K., Carré H.

### Abstract

The report presents concrete properties at high temperature of a High Performance concrete (HPC) M100. These properties have been studied extensively in the frame of the National French Project HPC 2000. The very detailed characterization at high temperature was performed by Izabela Hager - Gaweska during her PhD thesis in CSTB.

The report provides the composition of the concrete and its constituents, mass transport properties (porosity, permeability ...), thermal properties, mechanical properties (strength, thermal strain, transient thermal strain, fracture energy...). In addition, the report includes the results from pore pressure and mass loss measurements (PTM) at high temperature.

Finally, several tests have been carried out on fire exposed large-scale specimens, beams, columns and walls. The references of the publications are given at the end of the report.

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## 1. INTRODUCTION

The drafting of this report was initiated as part of the work of the RILEM Technical Committee 256-SPF Spalling of concrete due to fire: testing and modelling. More specifically, it was initiated in the dedicated to the modelling work on 2 concretes M100 and B40.

Both concretes have been characterized extensively. The report presents concretes properties at high temperature of a High Performance concrete (HPC) M100.

This concrete has been studied extensively in the frame of the National French Project HPC 2000. The Project, which brought together a large number of French laboratories as well as many other construction companies in France, carried out a wide-ranging study of high-performance concretes (mechanical properties, durability, steel reinforcement and concrete bond, etc.). A large part of the work focused on the concrete properties at high temperature and the concrete behaviour under fire [1], [2], [3], [4], [5] and [6]. Detailed characterization of the high-temperature properties was by Izabela Hager during her PhD thesis [7] whose work has given rise to numerous publications [8] to [17].

The report provides the composition of the concrete and its constituents, mass transport properties (porosity, permeability ...), thermal properties, mechanical properties (strength, thermal strain, transient thermal strain, fracture energy...). In addition, the report includes the results from pore pressure and mass loss measurements (PTM) at high temperature.

Finally, several tests have been carried out on fire exposed large-scale specimens, beams, columns and walls. The references of the publications are given at the end of the report.

The characterization of the 2 concretes B40 and B60 at high temperature has also been carried out and is summarized in a second report: Concrete properties at high temperature – complete characterization of 2 concretes B40 and B60 by Mindeguia J.C., I, Pimienta P., Mróz K., Carré H. and La Borderie C..

This report is intended to evolve and be updated as new data are acquired. Readers are invited to contact the authors if they detect any corrections or additions to be made.

## 2. MATERIALS AND MIXTURES

### 2.1. Concrete compositions

The mixture, as well as the main characteristics of the concrete M100C at 20 °C, are presented in Table 1.

For 1m <sup>3</sup> of M100C	Unity	M100C
CPA CEM I 52,5 PM CP2 Cement	kg	377
Silica fume	kg	37.8
0/5 Sand	kg	439
0/4 sand	kg	432
5/12,5 calcareous gravel	kg	488
12,5/20 calcareous gravel	kg	561
Water (W)	kg	124
Superplasticizer	kg	10.7
W/(Cement + SF) ratio	/	0.30
28 days compressive strength	MPa	112 – 123 *
28 days modulus of elasticity	GPa	50.8

*\* depending on the type of test facility, age of the sample and date of the batch*

Table1: mixes of the studied concrete M100C.

### 2.2. Binders properties

The cement used in M100C formula comes from Le Havre town in France (it is composed by 97 % of clinker and 3 % of limestone). Its main characteristics are presented in Table 2.

The silica fume for M100C comes from the Anglefort plant in France. It is composed by 90 % of amorphous SiO<sub>2</sub>, metal oxides and < 1 % of crystalline SiO<sub>2</sub>. Its density is 2.23 g/cm<sup>3</sup> and specific surface is between 10 and 30 m<sup>2</sup>/g. The characteristic dimension of the grains is 0.5 µm.

<b>Physical characteristics</b>	<b>M100C</b>
Shrinkage at 28 days ( $\mu\text{m}/\text{m}$ )	590
Hardening (min)	175
Density ( $\text{g}/\text{cm}^3$ )	3.17
Hydration heat ( $\text{J}/\text{g}$ )	(at 12 h ) 170
<b>Composition (%)</b>	<b>M100C</b>
Loss on ignition	0.18
<b>SiO<sub>2</sub></b>	<b>22.90</b>
Al <sub>2</sub> O <sub>3</sub>	2.97
Fe <sub>2</sub> O <sub>3</sub>	1.85
TiO <sub>2</sub>	
MnO	
<b>CaO</b>	<b>67.38</b>
MgO	0.91
SO <sub>3</sub>	2.23
K <sub>2</sub> O	0.18
Na <sub>2</sub> O	0.16
P <sub>2</sub> O <sub>5</sub>	
S--	< 0.01
Cl-	0.01

Table 2: properties of the cement used for M100C.

### 2.3. Aggregates properties

M100C has been fabricated with crushed calcareous gravels and sand, coming from the Boulonnais quarry in France. Their nature is: 99.5 % CaCO<sub>3</sub>, 2 % MgCO<sub>3</sub>, 1 % SiO<sub>2</sub>, 0.5 % Fe<sub>2</sub>O<sub>3</sub>, 0.6 % Al<sub>2</sub>O<sub>3</sub> and less than 0.06 % of sulfur, with a density of 2.7 g/cm<sup>3</sup> and a compressive strength between 140 and 180 MPa. The granulometry curves for the sand and aggregates are given in Figure 1.

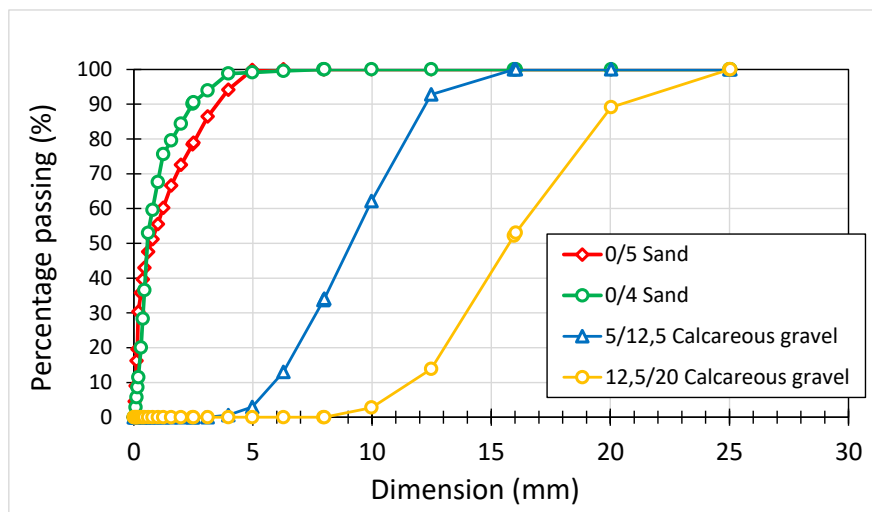


Figure 1: granulometry curve of M100C aggregates.

### 2.4. Storing conditions

The storing conditions of the samples prior the mechanical tests were the following. After casting, the samples were kept near the mixer (inner hall of the laboratory) in their moulds for 24 hours. The moulds were covered with plastic caps in order to avoid excessive evaporation of water. The samples were then removed from the moulds and placed in plastic bags at 20 °C during 6 days. Then their were removed from the pastic bags ans stored in a room at 20 °C and a 50 % RH until the day of the test. A minimum of 90 days of conservation was respected before testing.

## 3. EVOLUTION OF THE MAIN PROPERTIES OF CONCRETE VS TEMPERATURE

### 3.1. Water porosity

The concrete porosity was measured by a technique of water intrusion. The operation of such a test is based on the recommendations of the AFREM [18] and is based on measuring the mass of a specimen of concrete (quarter-cylinder) in various states of saturation:

- Mass in the dry state  $m_{dry}$  : the mass of the specimen is measured after oven drying at 80 °C,
- Mass in the immersed saturated state  $m_{sat}^{imm}$  : the specimens are immersed in a bell vacuum (pressure of 25 mbar) for 24 hours. During this period, the porous network is assumed to be fully saturated with liquid water. The mass of the immersed specimen is then measured by hydrostatic weighing,
- Mass in the saturated state  $m_{sat}$  : the saturated specimen is taken out from the water and its mass is measured in the ambient air.

Water porosity of concrete (in %) is then calculated according to the expression (eq.1).

$$\Phi = \frac{m_{sat} - m_{dry}}{m_{sat} - m_{sat}^{imm}} \times 100 \quad (\text{eq.1})$$

The water porosity of concrete is measured on specimens that have been thermally pre-treated at set temperature allowing to estimate the evolution of porosity with temperature. The heating rate and set values of temperatures for different concretes are summarized in Table 3.

Before cooling and measurement of the porosity, the maximal temperature of the cycle is maintained during several hours in such a way that specimens are assumed to be in a homogeneous thermo-hygral state. The stabilization time must be long enough to ensure that most of the physico-chemical transformations and that the departure of water from the specimen are achieved. Hence, the stabilization time is longer at lower temperatures. Table 4 summarizes the results obtained by Kalifa [5] and Hager [7] for M100C. Figure 2 presents the evolution of the water porosity with temperature for MC100.

Concrete	Temperatures (°C)	Heating rate (°C/min)
<b>M100C</b> (by Kalifa) [5]	105, 200, 300, 400	0.2
<b>M100C</b> (by Hager) [7]	180, 600	1.0

Table 3: heating rate and set values of temperatures for different concrete

M100C by Kalifa [5]		
T (°C)	Mean value (%)	Dispersion (%)
<b>105</b>	9.37	± 0.2
<b>200</b>	9.39	± 0.23
<b>300</b>	9.14	± 0.17
<b>400</b>	10.25	± 0.38

M100C by Hager [7]			
T (°C)	N°1	N°2	Mean value (%)
<b>20</b>	5.3	5.22	5.26
<b>180</b>	10.04	10.67	10.355
<b>600</b>	14.62	14.6	14.61

Table 4: water porosity after different thermal exposure (residual measurements) for M100C

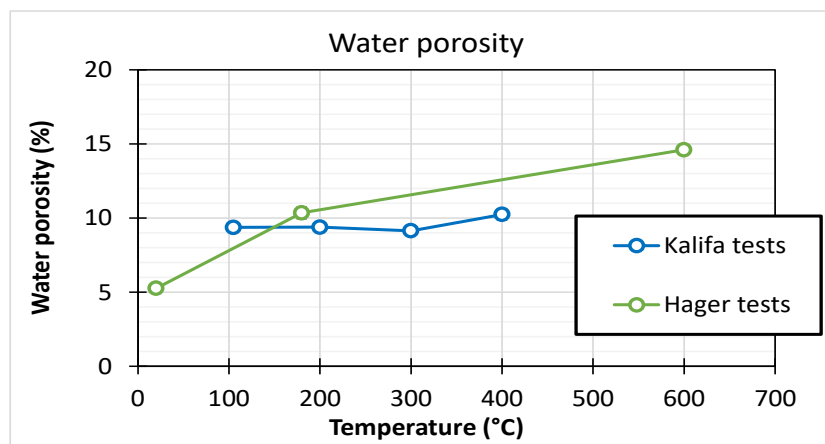


Figure 2: water porosity vs. temperature (mean values) for M100C by [5] and [7]



### 3.2. Density

The apparent density for M100C and its change with temperature is measured. The evolution of the M100C density with temperature is given in Figure 3.

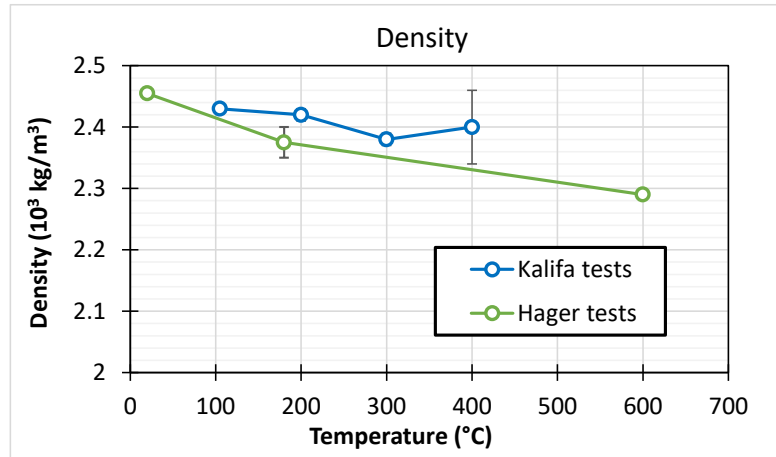


Figure 3: density vs. temperature (mean values) for M100C by [5] and [7]

### 3.3. Mass loss

Setup for experimental measurement of the loss of mass in transient heating is presented in Figure 4. It consists of a cylindrical specimen ( $\varnothing 104$  mm, H60 mm) placed inside an electric furnace with radiating elements. During the test, the specimen is placed on a perforated metal plate, suspended by an electronic balance. Balance is fixed on a rigid steel frame to minimize measurement noise caused by laboratory vibration and is connected to a computer during the test to measure continuous mass loss.

After measuring the initial mass, the specimen is exposed to heating rate of 1°C/min. The heating continues up to 600 °C. The mass loss and mass loss rate is monitored and presented in Figure 5.



Figure 4: Setup for experimental measurement of the loss of mass in transient heating.

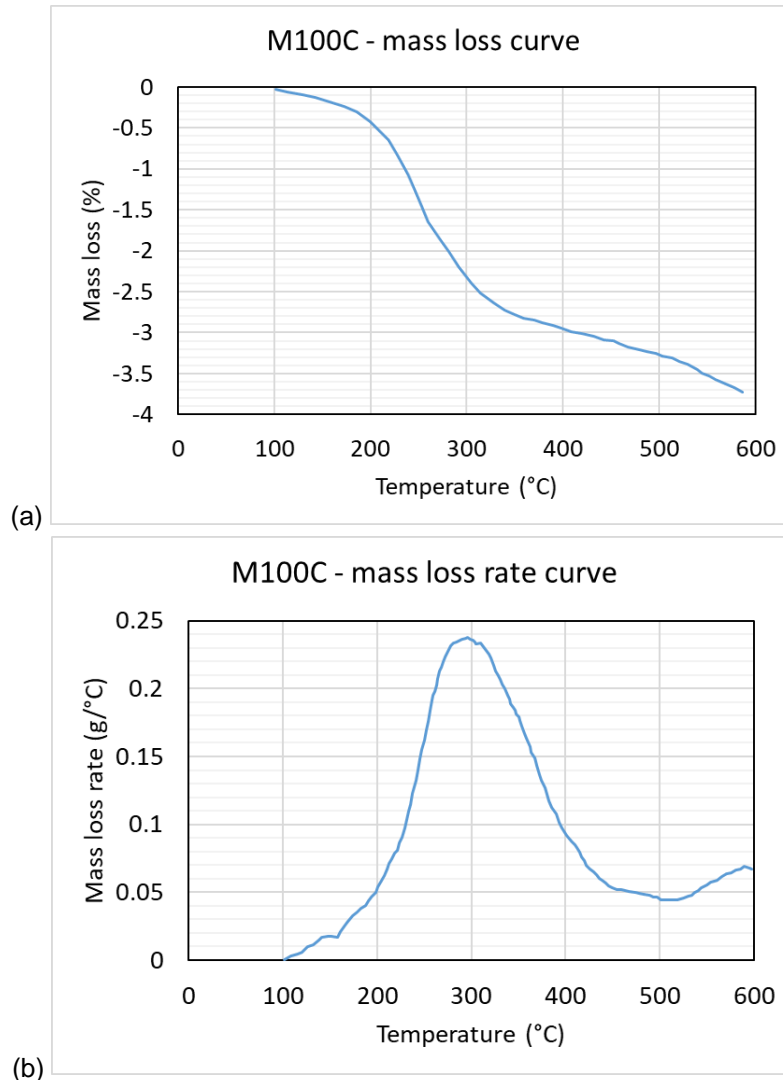


Figure 5: Results of: (a) mass loss and (b) mass loss rate vs. temperature for M100C.

### 3.4. Gas permeability

The gas permeability of concrete is measured through a constant pressure permeameter (“CEMBUREAU” device). The procedure is based on the recommendations of the AFREM [18] and consists in measuring the gas flow (di-nitrogen) percolating through a concrete disk (150 mm in diameter and 50 mm thick). For each measure, two levels of gas pressure are applied in order to determine the intrinsic permeability of the material according to the Klinkenberg's approach [19].

The evolution of the concrete gas permeability with temperature is estimated by applying the same thermal cycles as for water porosity measurement (see § 3.1).

The concrete M100C is tested for 20 °C, 105 °C, 200 °C, 300 °C and 400 °C.

Table 5 presents the gas permeability of M100C depending on the temperature. Figure 6 presents the evolution of the gas permeability with temperature for M100C.

	T (°C)	Mean value (10 <sup>-16</sup> m <sup>2</sup> )	Dispersion (10 <sup>-16</sup> m <sup>2</sup> )
<b>M100C</b>	<b>20</b>	0.21	± 0.12
	<b>105</b>	9.8	± 7.3
	<b>200</b>	3.9	± 2
	<b>300</b>	5.5	± 2.5
	<b>400</b>	29	± 10

Table 5: gas permeability after different thermal exposure (residual measurements) for M100C.

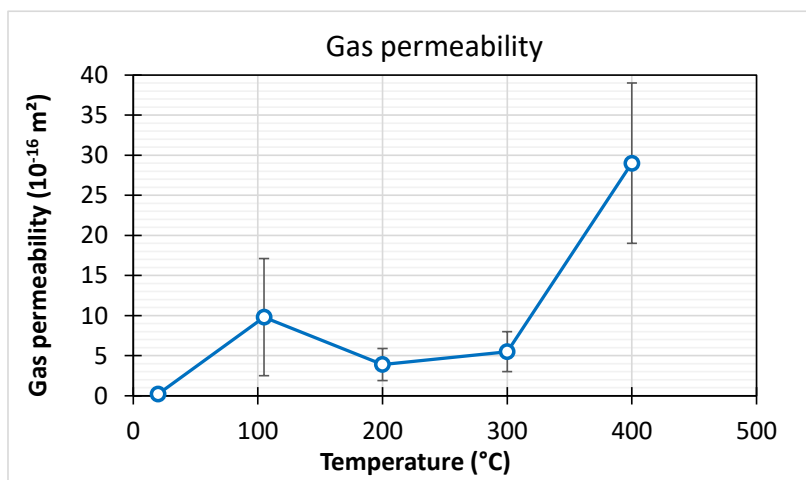


Figure 6: gas permeability vs. temperature (mean values) for M100C.

### 3.5. Thermal properties

The thermal properties of concrete (thermal conductivity and specific heat) were measured by mean of the so-called "TPS" (Transient Plane Source) method, developed by [20]. Table 6 gives the thermal properties measured at ambient temperature for all materials. To estimate the evolution of the thermal conductivity with temperature, the specimens and the TPS resistivity sensor can be placed in an oven. Figure 7 presents these changes measured for M100C. Table 7 present the results for thermal conductivity after different thermal exposure for M100C, and Table 8 presents the results for specific heat of M100C. Figure 8 presents the changes of specific heat with temperature for M100C.

	Thermal conductivity (W/mK)	Specific Heat (J/kgK)	Density (kg/m <sup>3</sup> )	Thermal diffusivity (mm <sup>2</sup> /s)
<b>M100C</b>	2.17	1011	2451	/

Table 6: thermal properties at 20 °C (TPS measurement).

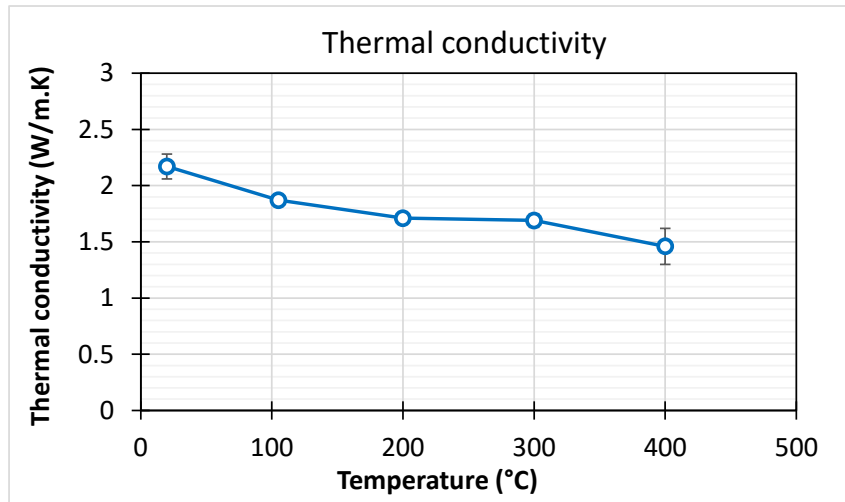


Figure 7: thermal conductivity vs. temperature (mean values). Results from Kalifa [5]

	T (°C)	Mean value (W/mK)	Dispersion (W/mK)
<b>M100C</b>	20	2.17	± 0.11
	105	1.87	± 0.02
	200	1.71	± 0.01
	300	1.69	± 0.06
	400	1.45	± 0.16

Table 7: thermal conductivity after different thermal exposure (residual measurements) for M100C.

	T (°C)	Mean value (J/kgK)	Dispersion (J/kgK)
<b>M100C</b>	20	1011	± 55
	105	843	± 5
	200	902	± 36
	300	870	± 58
	400	844	± 18

Table 8: specific heat after different thermal exposure (residual measurements) for M100C.

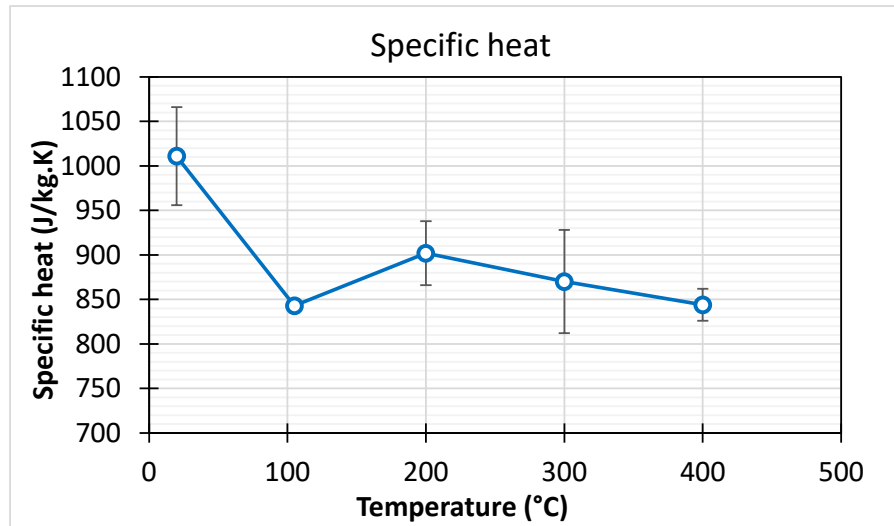


Figure 8: specific heat vs. temperature (mean values). Results from Kalifa [5]

### 3.6. Thermal expansion and Transient Thermal Strain

The mechanical properties at high temperatures were measured on cylindrical specimens (diameter of 104 mm, 300 mm height) [7] and [9] to [17]. These specimens are placed in a cylindrical oven. A system for measuring strains in longitudinal direction of the specimen is coupled with the furnace. The entire device can be placed between the plates of a hydraulic press, which allows measurements of mechanical properties at high temperature.

More details on the experimental device are presented in [9] and [21].

Samples (diameter 104 mm and height 300 mm) are heated at 1 °C/min until 600 °C. During the heating phase, different strains can be measured:

- **Free thermal strain (FS):** this is the strain of the sample in the longitudinal direction, without any mechanical loading
- **Total strain under load (TS):** here the sample is mechanically loaded in compression before heating. The studied loading levels are 20 % and 40 % of the 20 °C compressive strength of the M100C. The mechanical load is kept constant during the heating phase of the sample. TS is the strain measured during the heating phase
- **Transient thermal strain (TTS):** this strain component is defined as the difference between the total strain under load and the free thermal strain. It is then defined as  $TTS = TS - FS$

Figure 9 presents the results of FS, TS and TTS for M100C.

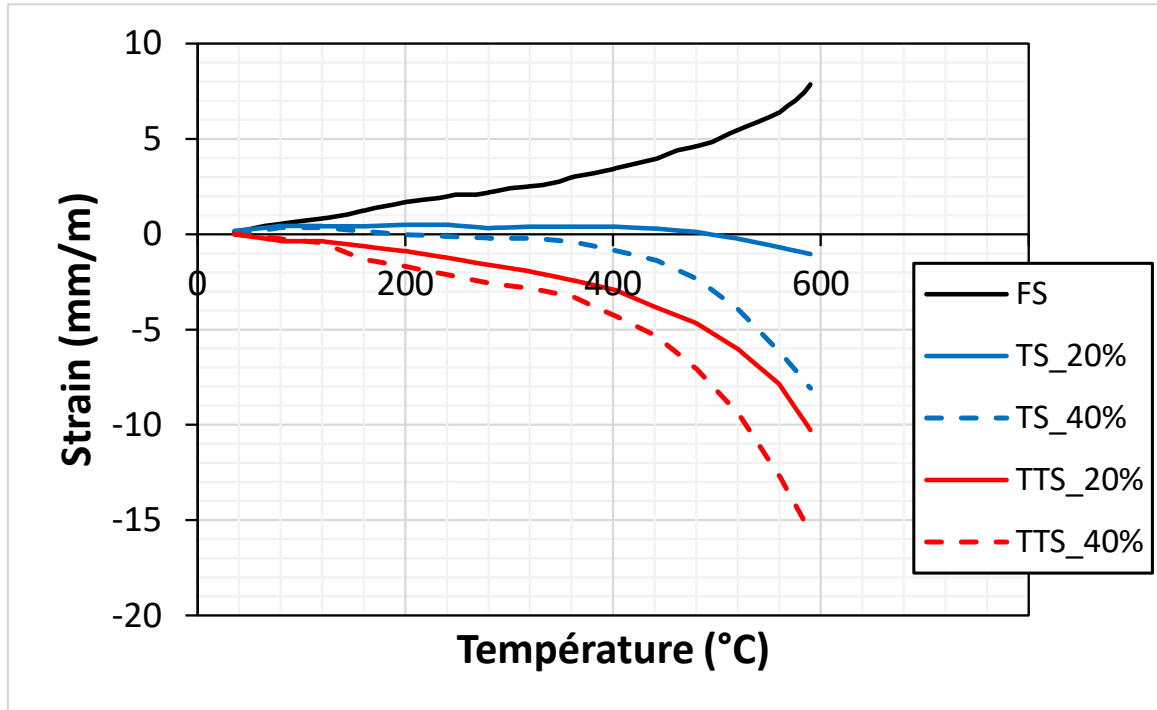


Figure 9: free thermal strain (FS), total strain under load (TS) and transient thermal strain (TTS) of M100C. Heating is done at 1 °C/min and applied loads are 20 % or 40 % of the 20 °C compressive strength of M100C.

### 3.7. Compressive strength and modulus of elasticity

The same device described in § 3.5 has been used for measuring the compressive strength and modulus of elasticity of concretes [1[2], [3], [7] and [8]. Tests have been done at ambient temperature and then in hot conditions at 120, 250, 400 and 600 °C on samples (diameter 104 mm and height 300 mm). The measured properties and their evolution with temperature are presented in Table 9 and Figures 10 for the compressive strength, and Table 10 and Figures 11 for the modulus of elasticity. The modulus of elasticity is defined as the slope of the stress / strain curve between the origin of the curve and the point corresponding to 30 % of the compressive strength of the material.

Compressive strength (MPa)	20 °C	120 °C	250 °C	400 °C	600 °C
<b>M100C</b>	120.7	90.8	73.1	80.8	35.6

Table 9: compressive strength (hot measurements, mean values).

Modulus of elasticity (GPa)	20 °C	120 °C	250 °C	400 °C	600 °C
<b>M100C</b>	50.8	41.5	33.4	26.8	7.6

Table 10: modulus of elasticity (hot measurements, mean values).

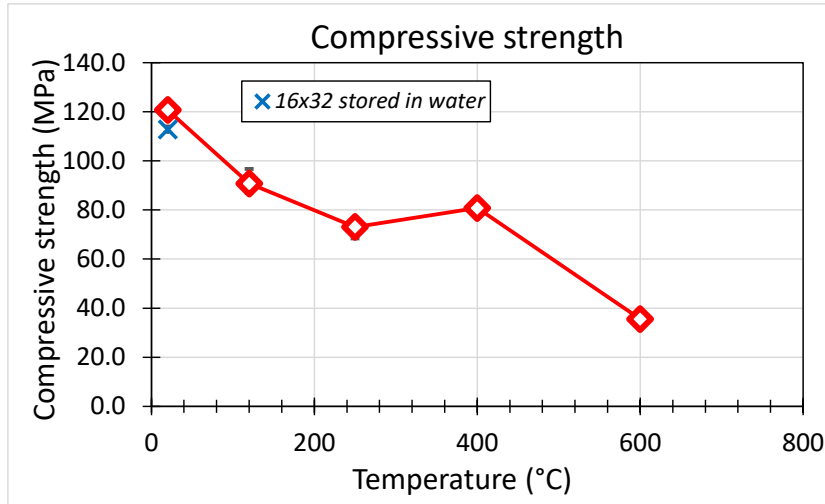


Figure 10: compressive strength vs. temperature for M100C.

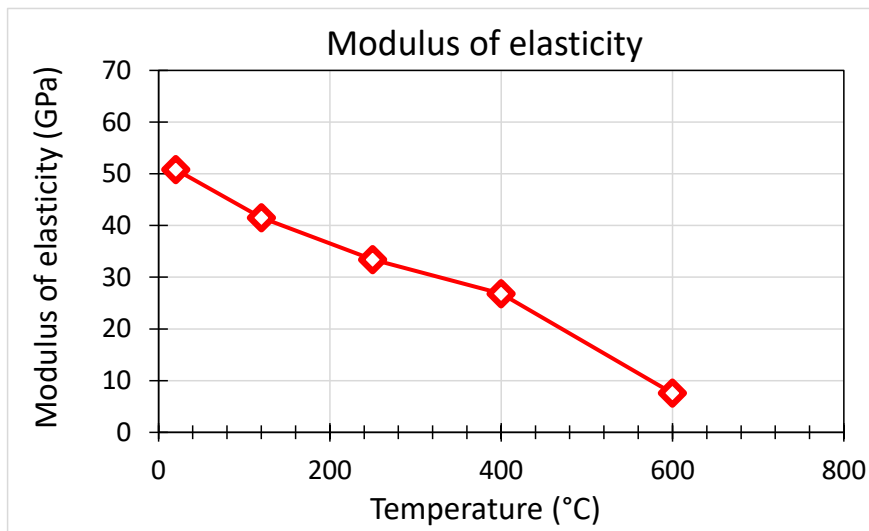


Figure 11: modulus of elasticity vs. temperature for M100C.

### 3.8. Pore pressure, temperature and mass loss measurement

#### 3.8.1. Experimental settings

The tests were performed in CSTB laboratory in Grenoble [5] and [6]. Tests were carried out on prismatic samples ( $300 \times 300 \times 120 \text{ mm}^3$ ) heated on one side. The experimental device allows temperature and pore gas pressure to be measured in a concrete specimen during a transient heating (see Figure 12). Before concrete casting, five metallic tubes (inner diameter of 1.6 mm) are placed in the molds. A sintered metal round plate (diameter 12 mm, thickness 1 mm) is wedged to the extremity of the tube that is placed into concrete. Just prior to tests, type K thermocouples (diameter 1.5 mm) are put in these tubes. It is worth noting that the free volume between the tube and the thermocouple ( $130 \text{ mm}^3$ ) were not filled with any sort of fluid, which can be different than other similar studies. The gas pressure in concrete is first transmitted via the metallic tubes to an external "T-junction", and then to an external pressure transducer (piezo-electrical transducer) via plastic tubes filled with silicon oil. By this way, temperature and pore gas pressure are simultaneously measured at the same location. Measurements are done at 2, 10, 20, 30, 40, and 50 mm from the heated surface. The thermal loading is ensured by a ceramic radiant panel (5 kW), covering the entire surface of the specimen and located 3 cm above it. The four sides of the concrete specimen are insulated by porous ceramic blocks. The heat flow can be then considered as unidimensional.

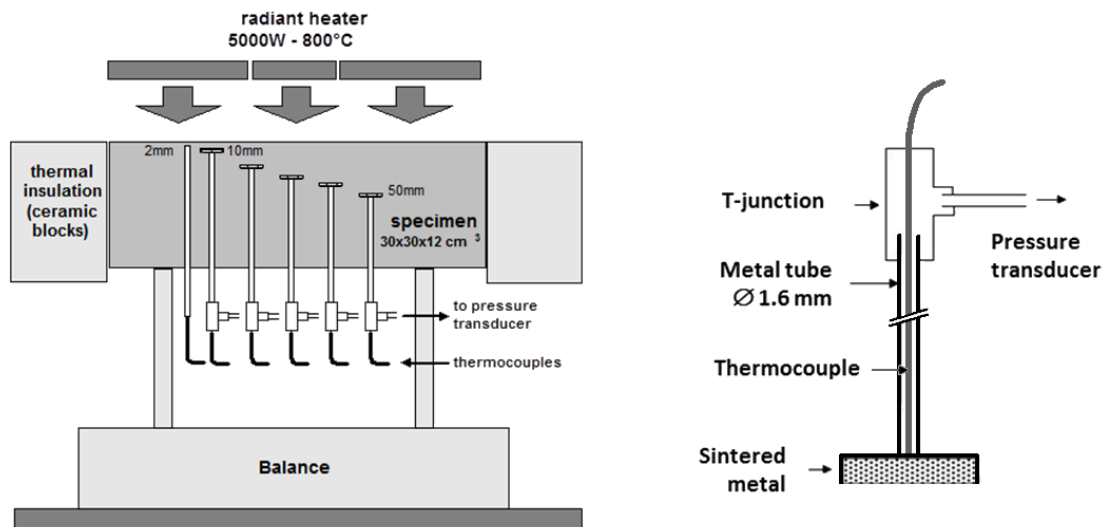


Figure 12: Scheme of the experimental set-up for small-scale tests (left). Right: detail of a metal tube filled with a thermocouple and connected to a "T-junction".



The hydral state before tests are given in Table 11 [6].

	<b>M100C</b>
<b>Days before test</b>	>90 days
<b>Water content at the day of test (%)</b>	2.95
<b>Water porosity (%)</b>	9.27
<b>Bulk density (kg/m3)</b>	2 340
<b>Degree of saturation (%)</b>	74

Table 11: hydral state of the samples before tests (mean values).

Most of the tests carried out by Kalifa [5] and [6] were done according to the following heating procedure:

- the radiant heater is turned on and its power is controlled in such a way that the temperature measured inside the panel reaches 600 °C as fast as possible. Generally this phase takes around 5 minutes,
- then power of the heater is regulated in such a way that the temperature in the radiant heater keeps constant during 6 hours
- finally the radiant heater is shut down and temperature naturally decreases until room temperature

In addition to this heating procedure, Kalifa carried out few tests where the radiant heater temperature was regulated at 800 °C and 450 °C (with the same procedure than previously described).

Figure 13 presents the evolution of the radiant heater temperature for the tests carried out at 600 and 800 °C. It also presents the evolution of the temperature measured at 10 mm from the exposed surface of the concrete sample, when the heater is controlled at 450, 600 and 800 °C.

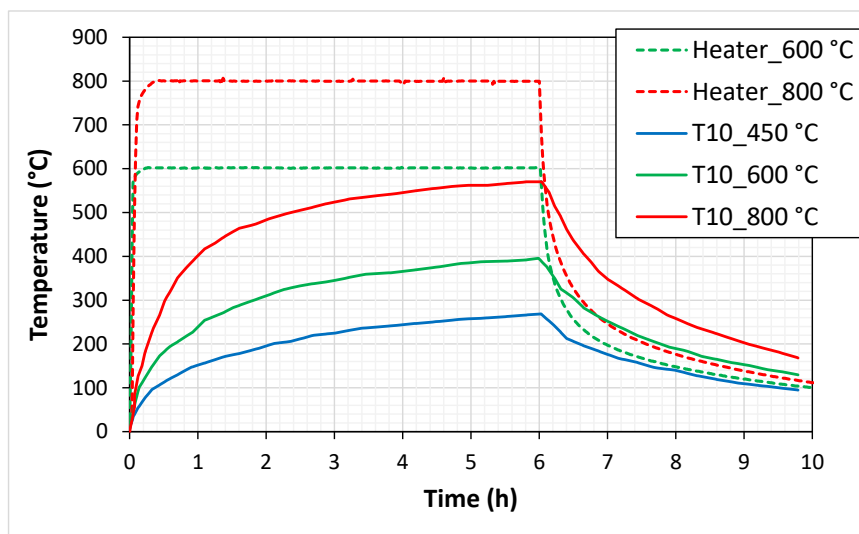


Figure 13: Evolution of the temperature in the radiant heater for 800 and 600 °C tests (dashed lines) and at 10 mm from the exposed surface in concrete for 450, 600 and 800 °C tests (continuous lines).

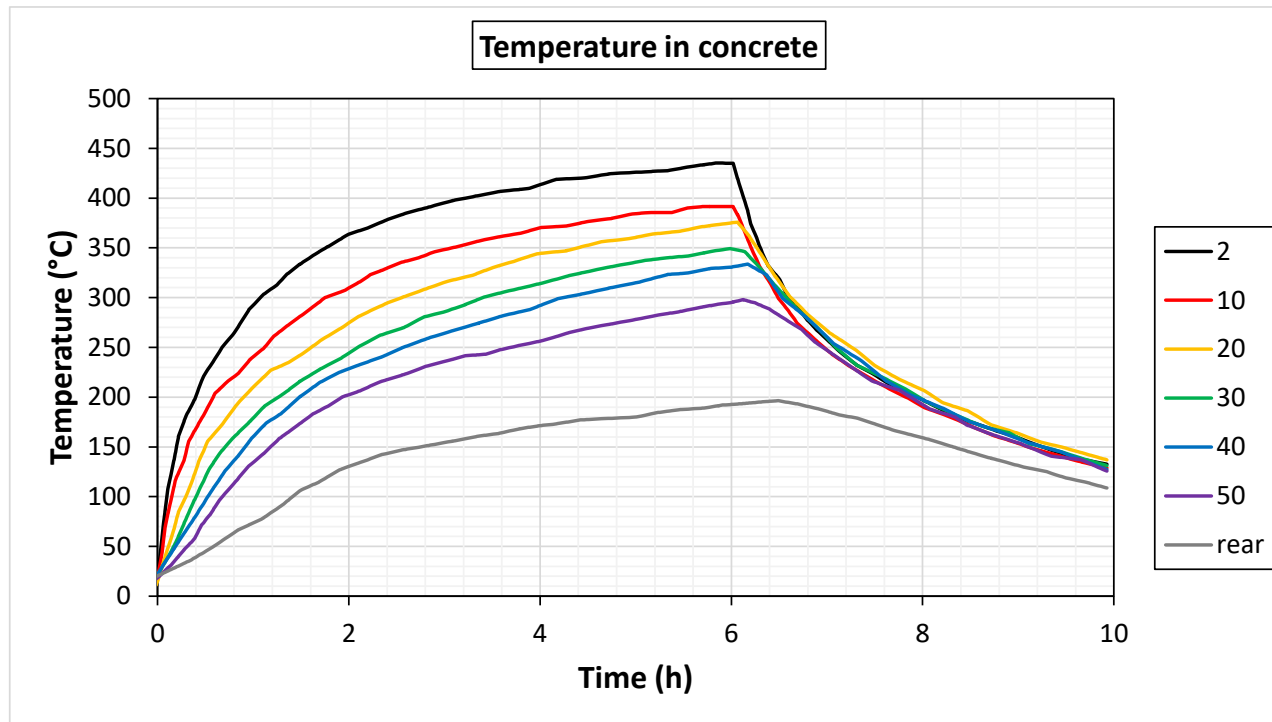
### 3.8.2. Results

For M100C concrete, only “moderate” heating has been carried out.

The time evolution of the temperature, the pore gas pressure and the mass loss of each sample are presented in the next pages.

### 3.8.2.1. Temperature measurement

Rear = unexposed side

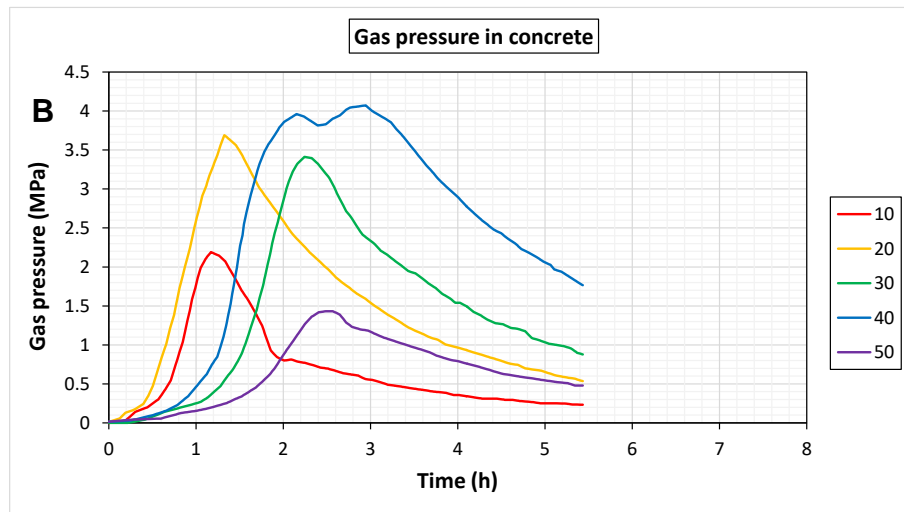
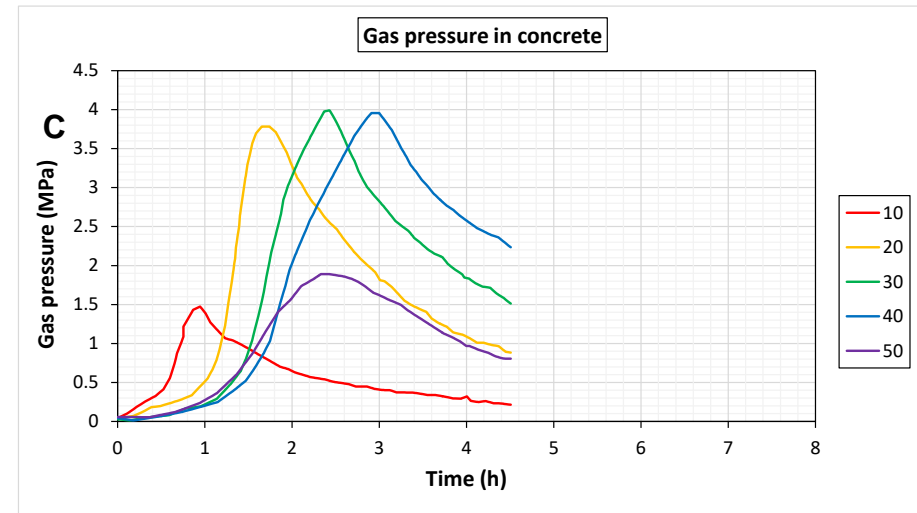
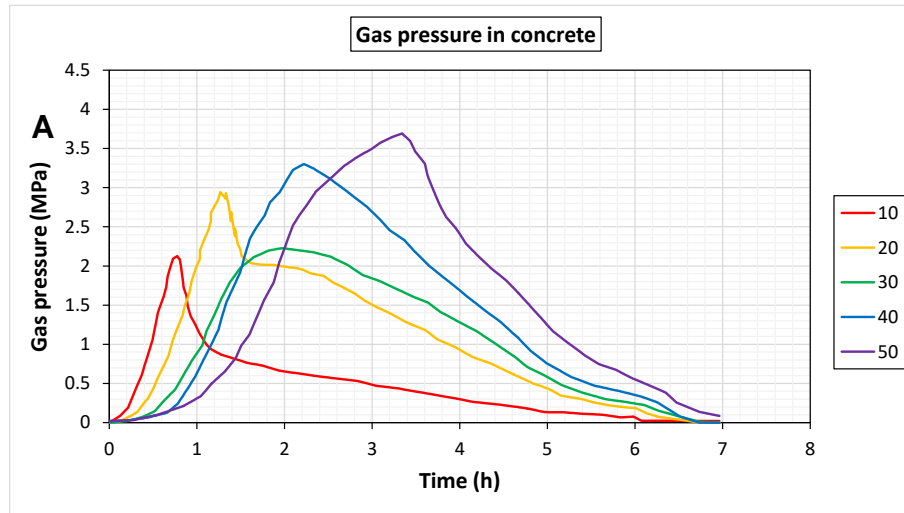


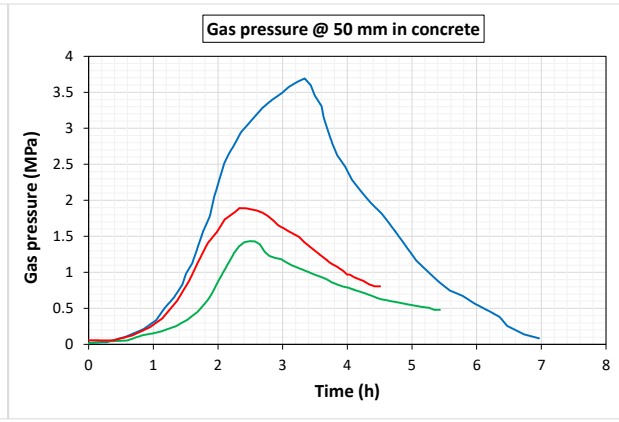
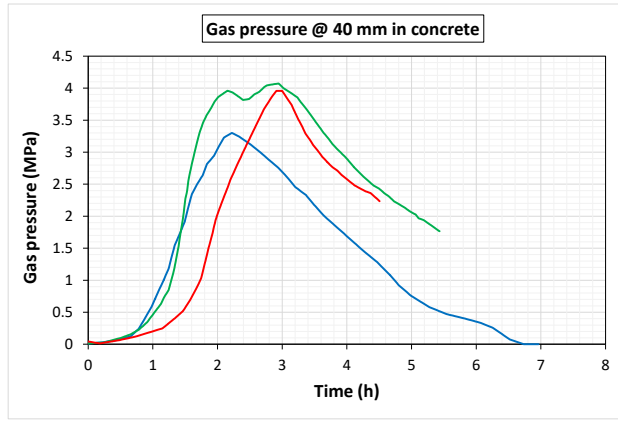
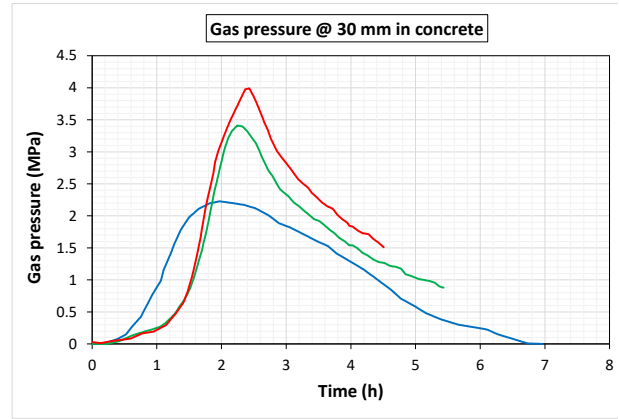
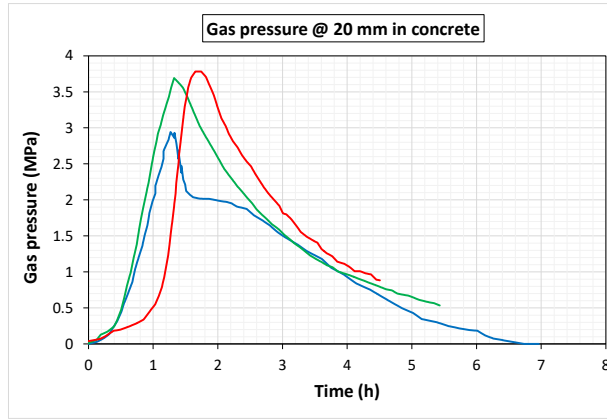
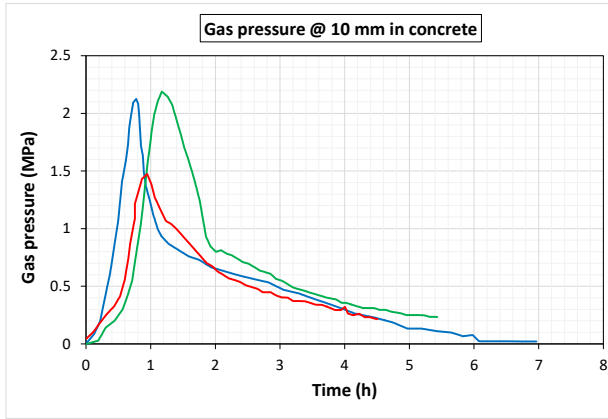
### 3.8.2.1. Pressure measurement

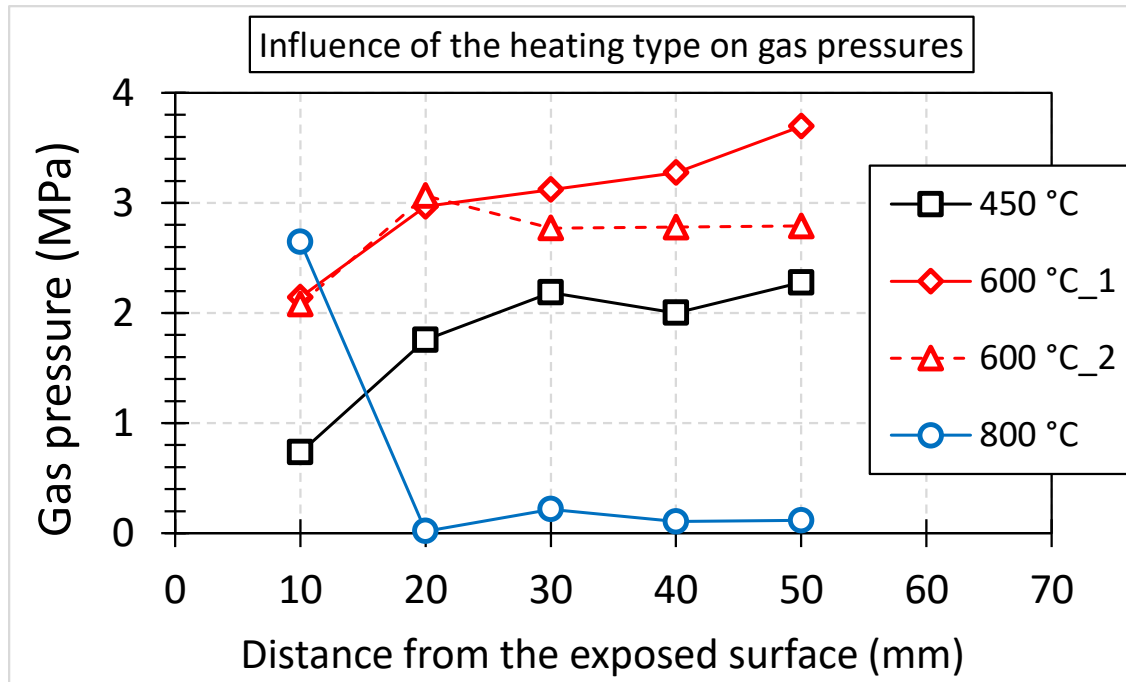
Figure A presents the evolution of gas pressure in M100C coming from 3 600 °C heating tests.

Figure B presents the comparison of pressure between these 3 tests, for each position into concrete.

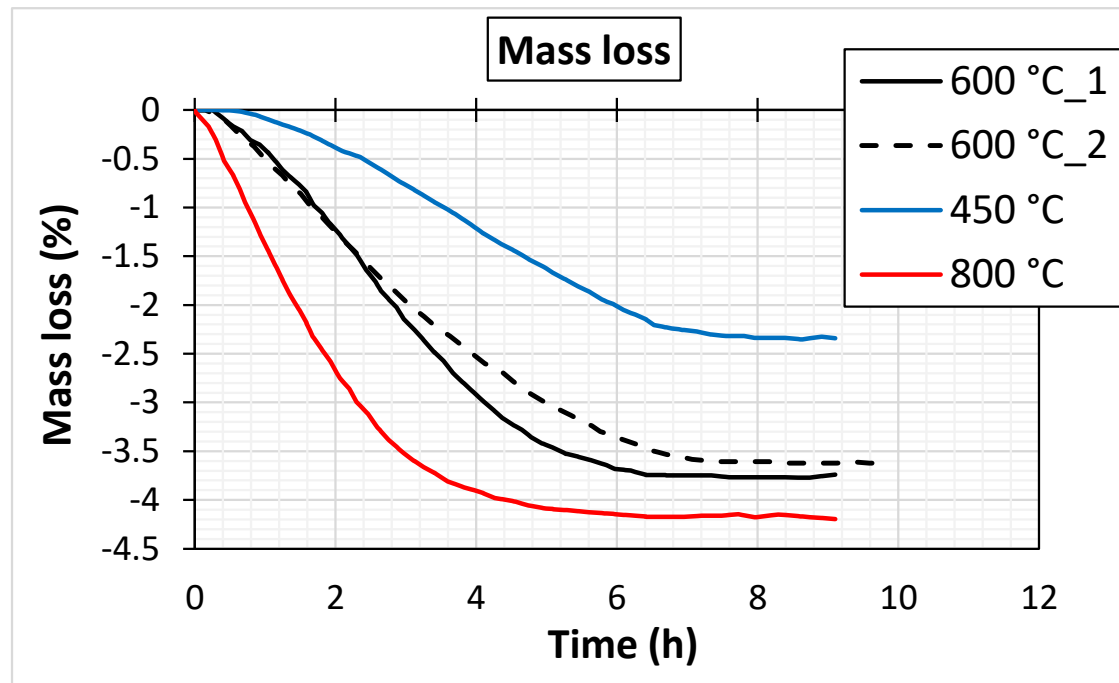
Figure C allows analyzing the influence of the heating procedure on the gas pressure. It presents the profile of the maximal pressure measured at each depth, and for the 3 heating procedures (450, 600 and 800 °C tests).







3.8.2.1. Mass measurement



#### 4. OTHER PROPERTIES OF B40 AND B60 CONCRETES

We have reported on section 3 the main properties (mass transport, thermal, mechanical) which have been studied on concrete B40 and B60. We have also reported results from the test PTM (Pressure, Temperature and Mass).

In this section 4, we give the references of the publications on fire exposed large-scale specimens made in the frame of the National French Project BHP 2000.

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*Fire spalling mechanism tests on large-scale slabs*

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Fire spalling tests on large scale beams, walls, and columns exposed to ISO [3], [4]  
834-1 under loaded conditions. The articles compare the behaviour of M100  
concrete with and without polypropylene fibers

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