Effect of cement type on pore pressure, temperature, and mass loss of concrete heated up to 800 °C

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ABSTRACT. *Pore pressure, temperature, and mass (PTM) tests up to 800 °C have been carried out on concrete specimens made with CEM III cement. Tests were carried out according to the test methods described by Kalifa and co-workers [1]. The present paper discusses the influence of cement type on the experimental results. Prismatic concrete specimens size of 300 x 300 x 120 mm^3 were manufactured in the laboratory conditions using CEM III/A 42.5 cement and the results were compared with CEM II/A 42.5 cement concrete. Three different thermal loads of slow heating up to 600 °C (after 1 hour of heating), moderate heating up to 600 °C (after 5 minutes), and rapid heating up to 800 °C (after 5 minutes) were applied and these temperatures were maintained for 6 hours. After 6 hours of stabilization, samples were naturally cooled down to the room temperature. The temperature, pressure, and mass loss were measured simultaneously during the test. Measured pore pressures were lower than 1 MPa for CEM III tested concrete specimens. These pore pressures are about half of the ones determined on CEM II concrete specimens due to its higher value of permeability at all the heating levels.*

KEY WORDS: *cement, concrete, temperature, pore pressure, mass, permeability.*

1 Introduction

Blast furnace slag cement, by-product of steel production, had been used since the beginning of the 20th century as partial replacement of Portland cement. It has demonstrated very good durability performances as low chloride diffusion. It is then mainly used in structures exposed to aggressive chemical environments as building foundations, tunnels and bridges. More recently, the cement industry has shown a significant interest in using blast furnace slag based cement (CEM III in Europe) because of its lower carbon footprint. However, the fire behaviour of blast furnace slag based cement concrete is not clear in the existing literatures. Most of the fire research work has been done on concrete made with Portland cement. Very few results are available in the existing literature, which does not provide the detailed investigation of the fire behaviour of concrete made with CEM III, especially spalling process of concrete in fire. This investigation provides insight on some of the physical properties of CEM III cement concrete at high temperature related to spalling. This paper presents the results of an experimental investigation of the influence of cement type on pore pressure build-up, temperature, and mass loss of normal strength concrete specimens subjected to different thermal loading conditions. In addition, gas permeability of concrete was also investigated to better understand the behaviour of concrete at elevated temperature.

2 Experimental Procedure

2.1 Concrete mix design, curing conditions and mechanical properties

An ordinary concrete (B40) made with CEM III (CEM III/A 42.5 N CE CP1 NF) cement has been investigated and the results were compared with CEM II (CEM II/A-LL 42.5 R PM-CP2) cement concrete [2-3]. The CEM III cement contains 54 % of clinker and 43 % of slag, while CEM II cement contains 92 % of clinker and no slag. The CEM III concrete specimens, 24 h after casting, have been covered by plastic film for 7 days inside the laboratory room. After 7 days of curing period, the specimens were stored in an indoor climate condition without plastic film up to the day of the test. The CEM II concrete specimens, were covered, after demoulding, with a plastic film in the climatic room (20 °C and 50 % RH) until the day of the test. Concrete mix design and properties at the fresh and hardened state are reported in Table 1. Compressive strength, modulus of elasticity, and tensile strength (splitting) have been measured on Ø 110 mm x h 220 mm cylindrical samples for CEM III concrete and Ø 160 mm x h 320 mm cylindrical samples for CEM II concrete.

Table 1: *Concrete mixture proportions and properties of fresh and hardened concrete*

	CEM III concrete	CEM II concrete
Cement (kg/m^3)	350	350
Calcareous 8/12.5 gravel (kg/m^3)	330	330
Calcareous 12.5/20 gravel (kg/m^3)	720	720
0/2 sand (kg/m^3)	845	845
Water (l/m^3)	188	188
Superplasticizer (by mass of cement)	1%	1%
Water / cement ratio (w/c)	0.54	0.54
Slump (cm)	19	8.8
28 days compressive strength (MPa)	42.8	37
90 days compressive strength (MPa)	49.1	-
28 days modulus of elasticity (GPa)	37.9	36
28 days tensile strength (splitting) (MPa)	4.2	2.4
Water content (after drying at 80 °C) (%)	2.96	2.59

2.2 Test procedure

2.2.1 Gas permeability tests

The residual gas permeability of concrete was measured on 150 mm diameter and 50 mm thickness concrete disc using a Cembureau constant pressure permeameter with nitrogen as the neutral percolating gas [4]. Three different levels of inlet pressures were applied to the samples depending on preheating temperatures in order to determine the intrinsic permeability of the material according to the Klinkenberg's approach [5]. However, the gas permeability of concrete was investigated at room temperature after applying thermal loads of 120, 250, 400 and 600 °C at a slow heating rate of 1 °C/min. After reaching the target temperatures, the temperatures were stabilized for 28, 10, 6 and 6 hours, respectively, for the temperatures of 120, 250, 400 and 600 °C to reach a uniform temperature and water content distribution in the concrete. The specimens were then cooled inside the closed furnace to ambient temperature. The average cooling rate was 0.5 °C/min to avoid thermal shocks. Moreover, the reference specimens were dried in oven at the temperature of 80 °C until constant value of mass was reached. Stabilization was considered achieved, when difference between 2 masses determined in an interval of more than 24 h was lower than 0.1 %.

2.2.2 PTM (pressure, temperature and mass) tests

PTM tests were carried out according to the test methods described by Kalifa and co-workers [1]. The experimental campaign consisted of 12 test specimens for 3 different thermal loads of PTM tests, 6 specimens for each type of cement. Two specimens for each heating levels were used to provide a measure of repeatability. Prismatic concrete samples of 300 x 300 x 120 mm^3 were used for all PTM tests. Specimens were instrumented with five gauges placed at 10, 20, 30, 50 and 80 mm depths from the exposed surface of concrete specimens for simultaneous pressure and temperature measurements (Figure 1). The measurement of the gas pore pressure and temperature was performed by using capillary steel pipes (inner diameter 1.6 mm), fitted with sintered metal round plate (Ø 12 x 1 mm). The specimen was placed on a balance in order to monitor its mass during heating. For more details about the experimental set-up, the reader should refer to Kalifa and al [1].

In order to understand the role played by cement type in fire, particularly the influence of heating severity on both thermo-hygral behaviour and instability risk of concrete, 3 different thermal loads have been used. The heating system consists of one radiant panel positioned 3 cm above the surface of the prismatic samples. The sample lateral faces are insulated with porous ceramic blocks to favour quasi-unidirectional heat transfer. The thermal heating loads were as follows.

<u>Slow heating</u>

The radiant panel was controlled with a heating rate of 10 °C/min up to 600 °C (after 1 h of heating) and then this temperature was maintained for 6 hours. After 6 hours of stabilization, samples were naturally cooled down to the room temperature (20 °C).

<u>Moderate heating</u>

The radiant panel is controlled in such a way that the temperature rapidly reached 600 °C (after 5 minutes of heating). The heating panel is maintained at this temperature for 6 hours. After that, samples were naturally cooled down.

<u>Rapid heating</u>

The heating system is the same as the moderate heating; the only difference is that the radiant panel temperature rapidly reached 800 °C (after 5 minutes of heating).

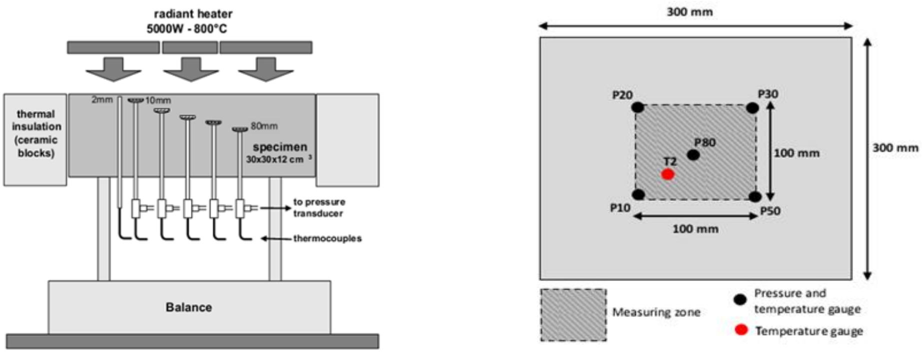

Figure 1: *Experimental set-up (left) and measuring position of the temperature and pressure (right).*

3 Experimental results and discussion

3.1 Gas permeability

The residual intrinsic permeability of the CEM II and CEM III tested concrete specimens subjected to different preheating temperatures are presented in Figure 2. In Figure 2, the y-axis presents the intrinsic permeability in $\log_{10}$ scale and the x-axis presents temperature in arithmetic scale. It can be seen that the gas permeability of concrete increases with temperature. After being exposed to the same temperature, the increase of gas permeability of CEM III concrete is higher than the one of CEM II concrete. It is particularly notable that a sharp increase in permeability was observed from 80 °C to 120 °C for the CEM III concrete while a more gradual increase was found for the CEM II concrete (Figure 2). In this range of temperature, no visible surface cracks were observed. However, this trend is in good agreement with observed apparent porosity measurements. These measurements have been carried after cooling on the same samples and at the same temperatures.

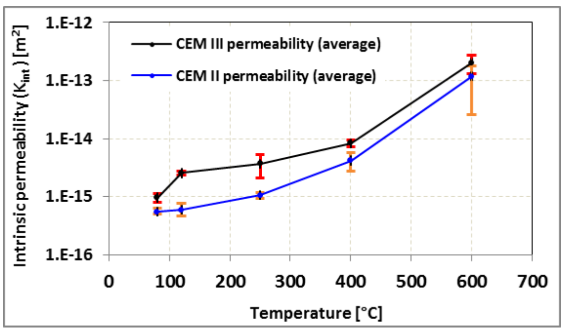

Figure 2: *Residual gas permeability of concrete as a function of temperature.*

The results show that CEM II concrete porosity has not increased in the temperature between 80 °C – 120 °C, while CEM III concrete porosity has increased about 3 % in this range of temperature. No exact explanation is given yet by the authors to explain this sudden increase of porosity and permeability from 80 °C to 120 °C in CEM III concrete. Research is needed to analyse the microstructure of both concretes at these temperatures.

In a more general way, the increase in permeability is attributed to the removal of free water from the porous network, dehydration of the cement gel and, at temperature higher than 300 – 400 °C, the development of cracks mainly caused by the thermal incompatibility between the cement paste and the aggregates [2, 6].

3.2 PTM tests

3.2.1 Mass loss

Mass loss of CEM III and CEM II concretes were measured during PTM tests is illustrated in Figure 3 (left). As written in the previous paragraph, 2 samples for each thermal load were tested. The curves corresponding to one of the two tests are presented here. Higher mass loss rate and higher value of final mass loss was observed in rapid heating of CEM III and CEM II concretes compared to the slow and moderate heating of the 2 concretes. This behaviour is firstly due to the higher temperature which accelerates the vaporization. It is also due to the higher temperature gradient into the concrete which strongly modify the transport properties inside the concrete making easier the movement of fluids. Finally, thermal damages were visible in both concrete samples when exposed to rapid heating (Figure 3 right). Then, induced thermal damage could be one of the main reasons explaining the higher mass loss rate observed during rapid heating tests.

The other noticeable result was that a higher mass loss was observed in the CEM II concrete compared to the CEM III concrete specimens for all thermal loads. However, from table 1, it can be observed that water contents of the 2 concretes samples were close. No explanation is given yet by the authors to explain the 2 concretes behaviour difference.

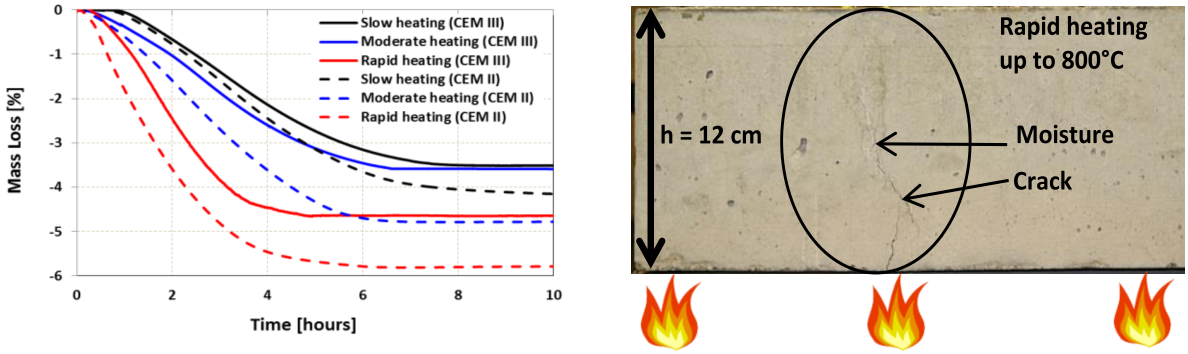

Figure 3: *Mass loss of concretes as a function of time (left). Cracking and released of hot moisture (right).*

3.2.2 Temperature distribution

Figure 4 presents the temperature measured into the concrete at different depths from the exposed surface. CEM II concrete exhibited little higher temperature for all depths with all thermal loads compared to the CEM III concrete. For all heating levels and for each measuring depths, a slight plateau has been observed. The temperature plateau is caused by the water phase change (vaporization). This transformation is endothermic and consumes part of the energy that is brought by heating. As a consequence, the temperature rise of concrete sample is slowed down. It can be emphasized that water vaporization can induce additional temperature gradients [7]. It is also observed that the length of the temperature plateau depends on the intensity and the level of thermal load. Rapid heated and slow heated specimens exhibited shorter temperature plateau. Figure 5 represents the evolution of temperature and pressure as a function of time that have been measured at 20 mm from the heated surface (similar behaviour has been observed for other depths of concrete). Vaporization plateaus are noticeable (Figure 5) around 165 to 175 °C for slow heating, 170 to 190 °C for moderate heating, and 170 to 185 °C for rapid heating. For slow and rapid heating, the pressure peak coincides with the end of the vaporization plateaus. For moderate heating, the pressure peak occurs before the end of the plateau.

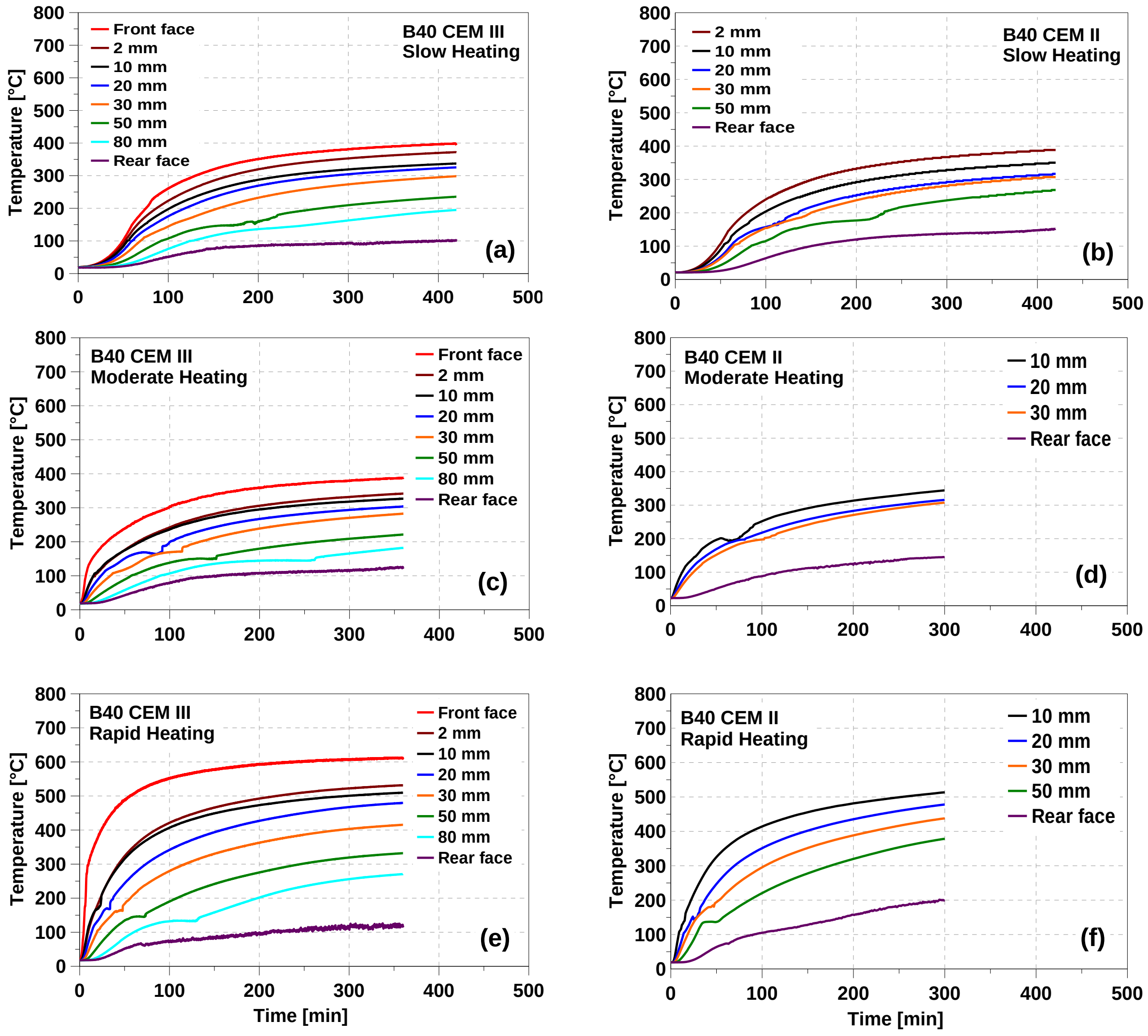

Figure 4: *Evolution of temperatures inside the 2 concretes exposed to the 3 thermal loads versus time.*

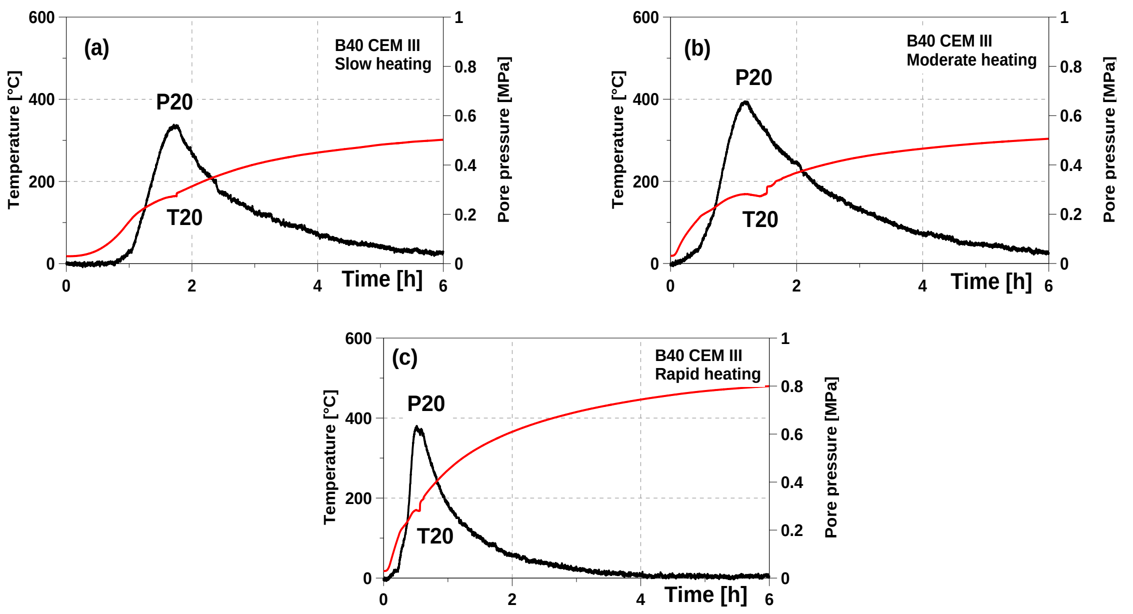

Figure 5: *Evolution of temperature and pore pressure at 20 mm depth from the heated surface of CEM III concrete.*

Before vaporization occurs in a given area, the increase of pressure is due to arrival of water coming from warmer region (closer to the heated surface). When vaporization occurs, this involves increase of permeability and decrease of pore pressure.

3.2.3 Gas pore pressure development

Low pore pressures were observed for CEM III tested concrete specimens exposed to 3 different thermal loads, which is lower than 1 MPa (Figure 6). These pore pressures are about half of the ones determined on CEM II concrete specimens. This behaviour could be attributed to the lower value of permeability of the CEM II concrete (Figure 2). Indeed, lower permeability reduces transport of water vapor inside the concrete and then induces faster build-up of pore pressures and higher value of measured gas pore pressure [1, 8].

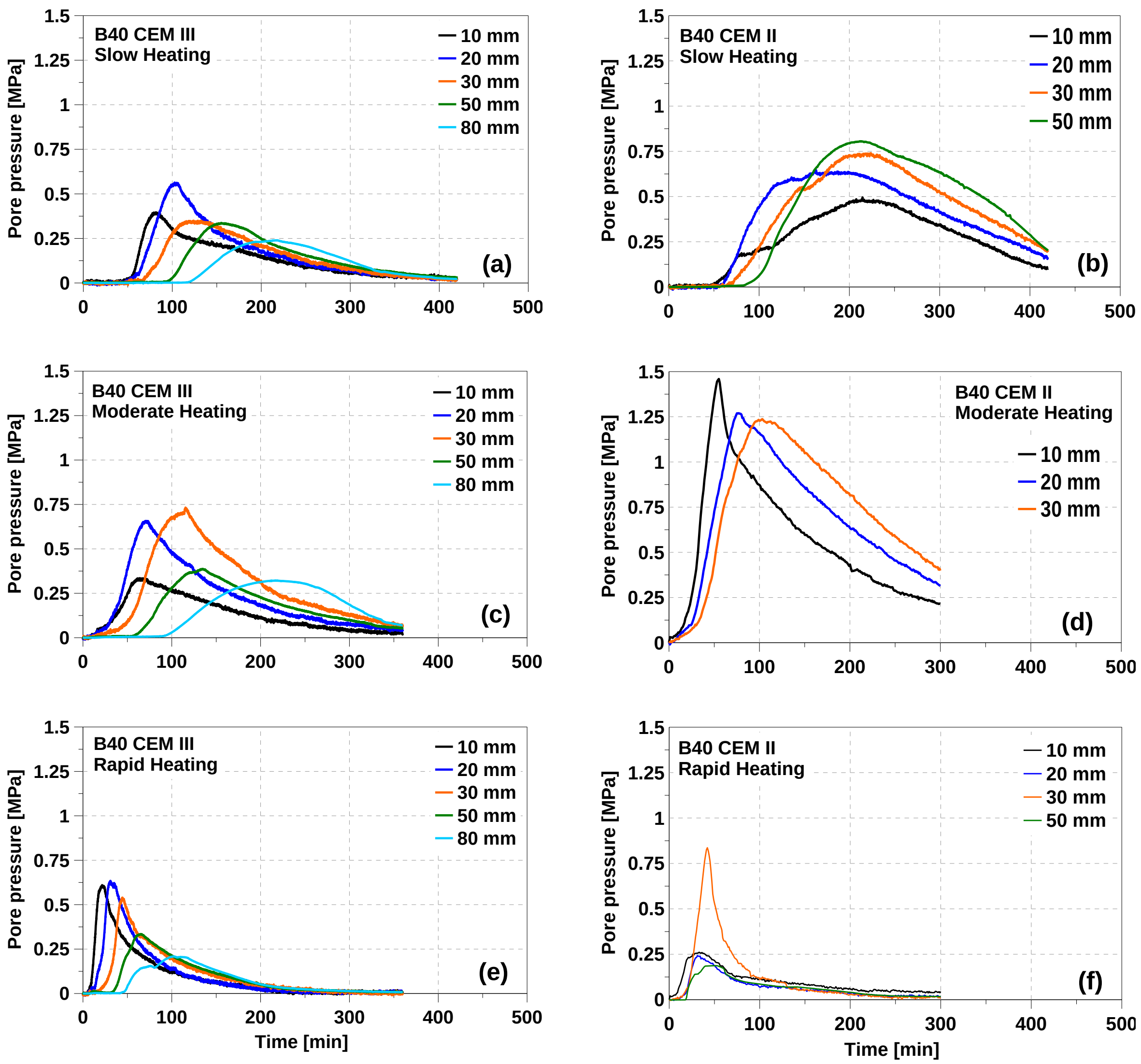

Figure 6: *Gas pore pressure development inside the concrete at various depths from heated surface.*

Higher gas pore pressures were observed for moderate heating of CEM III and CEM II concretes, while lower pore pressures were measured in slower heating concrete. Sharp increase in pore pressure as well as sharp decline of pore pressure was found in all depths of rapid heating concrete specimens (Figure 6e and 6f). A network of cracks on the exposed surface and few large vertical cracks (around 1 mm) on the lateral side of the specimens were observed (Figure 3 right). Release of moisture was also observed around the cracks, while no cracking of this type was observed for slow and moderate heating concrete specimens.

Figure 7 represents the experimental results of pressure-temperature together with the saturation vapour pressure P_{vs} (T) curve. The saturation curve represents a limit condition of pressure-temperature relation.

Theoretically, pressure-temperature curves cannot pass beyond the saturation curve. It can be observed that measured pressures of CEM III and CEM II concretes followed the P_{vs} curve during the ascending branch. Except for rapid heating, at a 80 mm depth of CEM III concrete and a 50 mm depth of CEM II concrete measured pore pressure is slightly higher than the saturation vapour pore pressure curve (Figure 7e and 7f). This overpressure could be attributed to the partial pressure of the dry air enclosed within the porous [1, 7]. The partial pressure of air in the pore is strongly dependent on its liquid water saturation: the higher the water saturation, the lower is the free volume available to the air to expand during heating.

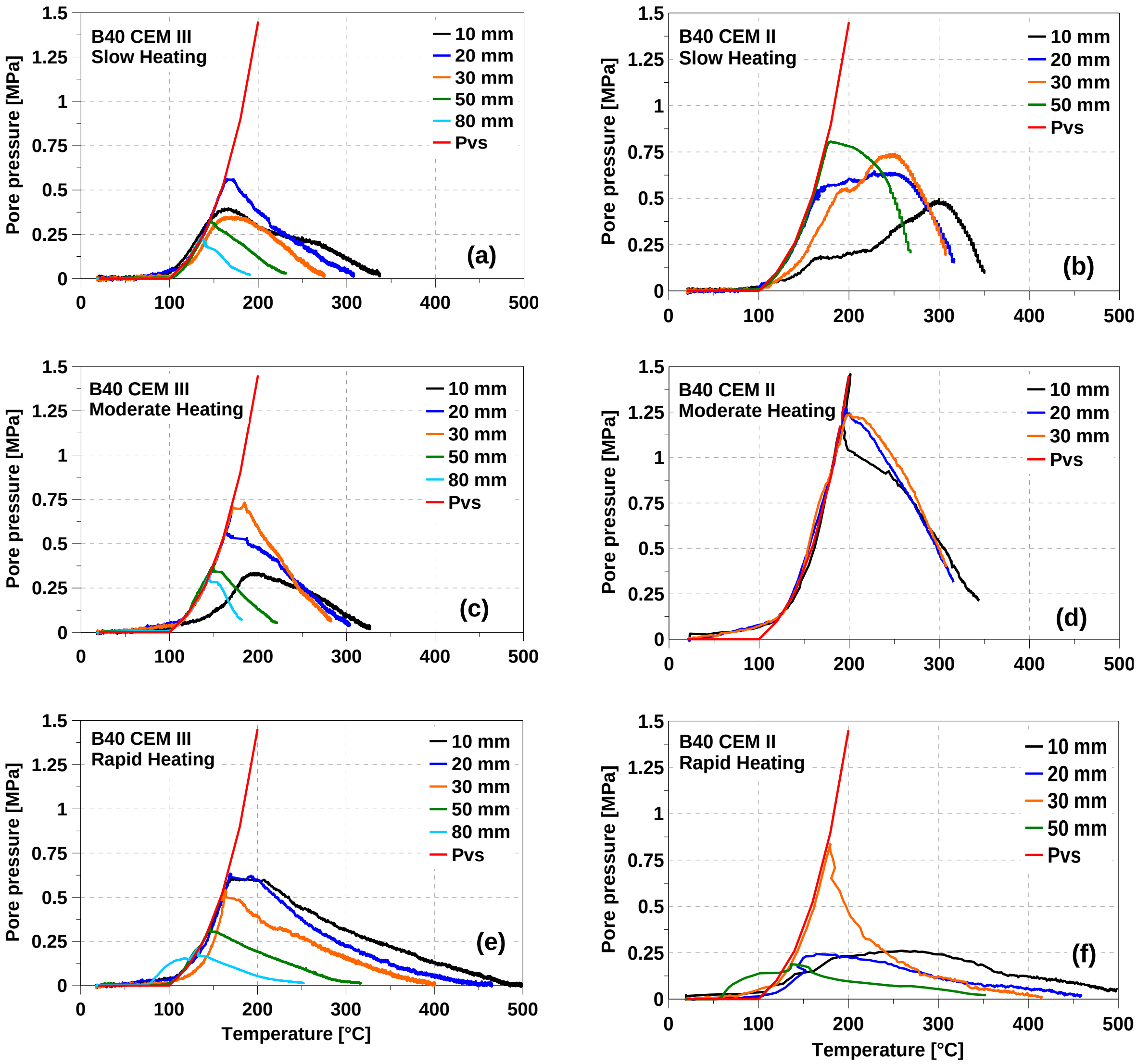

Figure 7: *Pore pressure versus temperature, plotted together with the saturation vapour pressure* P_{vs} *(T).*

4 Conclusions

This research aims at providing the pore pressure development at different thermal loads on blast furnace slag cement based concretes and at comparing the obtained results with CEM II based concretes. To better understand the pore pressure behaviour of the two different cement concretes, the gas permeability of concretes were also studied with different preheating temperatures. The following conclusions can be drawn based on the results presented in this study.

No significant change in maximum pore pressures were observed for the CEM III tested concrete specimens exposed to 3 different thermal loads, which is lower than 1 MPa.

CEM II concrete presented higher pore pressure for all the thermal loads than the CEM III concrete. This result should be explained by lower CEM II concrete permeability at high temperature.

During rapid heating more surface cracks and few big cracks were observed due to the higher thermal gradient of rapid heating which leads to lower maximum pore pressure inside the concrete. The cracks allow significant amounts of vapor and liquid water to be drained out from the specimen. Perhaps this could be a reason for higher mass loss of both concretes when heated to rapid heating test. This suggests that the internal cracking is an important factor for pore pressure development when concrete are exposed to fast heating rate.

Based on these experimental results, it can be seen that the replacement of CEM II cement by CEM III cement in the same mix design has different behaviour at elevated temperature. As written before, CEM III concrete exhibited higher value of permeability and lower value of pore pressure at all the thermal loads. These results tend to show that the studied CEM III concrete could be less sensitive to spalling. However, it has been shown that fire spalling cannot be only explained through the build-up of pore pressure [3].

Permeability is linked to crack opening and crack opening is influenced by mechanical stress that may be applied on a concrete member. Research is needed to quantify the influence of mechanical loading on pressure build up and spalling. This research is underway at SIAME and CSTB. The results will be published soon.

5 References

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