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CHANGES IN MICROSTRUCTURE AND PORE STRUCTURE OF LOW-CLINKER CEMENTITIOUS MATERIALS DURING EARLY STAGES OF CARBONATION

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Key words: Carbonation, drying, cement paste, slag, early age, pore structure

Abstract. Carbonation is one of the main causes of reinforced concrete damage. It leads to decrease the pH of the concrete pore solution and to CaCO₃ formation from the reaction between CO₂ and cement hydrates. The mechanisms of carbonation are complex in low-clinker cementitious materials. At a given age, the hydration degree in low-clinker cementitious materials is reduced compared to CEM I materials which is due to the slower reaction of mineral additives and leads to the underdeveloped of microstructure. Therefore, the coupling between hydration, drying and carbonation at early age needs to be analysed, in order to understand and predict the durability of low-clinker cementitious materials. In this work, the coupled process leads to a faster drying of GGBS (Ground Granulated Blastfurnace Slag) cement paste at early age compared to the CEM I case. However, this trend changes when the GGBS reactions occur that are confirmed by the MIP results. Similar carbonation depths at early age are observed but a lower carbonation degree suggests a lower diffusivity and finer microstructure for GGBS paste.

1. INTRODUCTION

Rising energy cost and environmental considerations have shifted the cement manufacture industry to follow a low clinker cement approach. However the reactions of Supplementary Cementitious Materials (SCMs) are slower than clinker [1]. If they are cured like a CEM I system, the hydration does not occur completely [2] and consequently, the microstructure stays underdeveloped, more porous [3] and susceptible to the ingress of aggressive species, which primary starts with the process of carbonation as the cementitious structures are directly in contact with the atmospheric CO₂ after demoulding. Therefore, these kinds of materials require a specific curing condition [4]. In theory, a longer curing time is better for the durability as the microstructure is more developed. Nevertheless, in practice, it is impossible to keep the frameworks until material properties are fully obtained. A curing time of 3 days seems to be a favourable compromise.

In the CEM I cementitious materials, the carbonation reaction is sum up by the following equation:

\[ \text{CO}_2(g) + \text{Ca(OH)}_2 \rightarrow \text{CaCO}_3 + \text{H}_2\text{O} \] (1)

The use of supplementary cementitious materials (SCMs) causes a decrease in the Ca(OH)_2 content due to the dilution of clinker and its consumption by the pozzolanic reaction. In the blended cements, as the portlandite content decreases, the carbonation of the C-S-H cannot be
ignored ([5-7]). Consequently, the mechanisms of carbonation become more complex in low-clinker cementitious materials. These mechanisms depend on the hydration and saturation degree.

The importance of the curing time is shown by Aruhan [8], the carbonation depth decreases with the curing time. Drying causes a decrease in the internal relative humidity that leads to slow down or to stop the hydration reaction as shown by Jensen [9]. Furthermore, there is less hydrates phases which may react with the carbonates and the microstructure is coarser. Therefore, the carbonation depth progress is delayed with the curing time.

In this paper, the effects of coupling between carbonation and drying at early age (after 3 days of water curing) on the pore structure, the saturation and the microstructure profiles of GGBS cement pastes are studied and compared to CEM I cement pastes.

2. MATERIALS AND METHODS

2.1. Materials

Two paste systems (CEM I (PCM) and CEM I with replacement by 60% GGBS (Ground Granulated Blastfurnace Slag) (P6S) with W/B ratio of 0.57 were investigated. The replacement in the manufacture of the two systems was by volume. The chemical composition of CEM I 52.5 R and GGBS were determined by X-ray fluorescence (XRF) (Table 1). The clinker phase contents are calculated according to Bogue’s approach and shown in Table 2. The Blaine fineness is 4740 cm$^2$/g for the GGBS and 4900 cm$^2$/g for the CEM I.

<table>
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<tr>
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<th>SiO$_2$</th>
<th>Al$_2$O$_3$</th>
<th>Fe$_2$O$_3$</th>
<th>TiO$_2$</th>
<th>MnO</th>
<th>CaO</th>
<th>MgO</th>
<th>SO$_3$</th>
<th>K$_2$O</th>
<th>Na$_2$O</th>
<th>P$_2$O$_5$</th>
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<tbody>
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<td>20.50</td>
<td>4.60</td>
<td>2.40</td>
<td>0.30</td>
<td>0.00</td>
<td>63.40</td>
<td>2.00</td>
<td>3.60</td>
<td>0.74</td>
<td>0.13</td>
<td>0.30</td>
</tr>
<tr>
<td>GGBS (%)</td>
<td>(+1.33)</td>
<td>36.06</td>
<td>11.22</td>
<td>0.38</td>
<td>0.74</td>
<td>0.22</td>
<td>41.75</td>
<td>6.20</td>
<td>2.84</td>
<td>0.52</td>
<td>0.19</td>
<td>0.01</td>
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Table 2: Mineralogical composition of CEM I 52.5R

<table>
<thead>
<tr>
<th></th>
<th>C$_3$S</th>
<th>C$_2$S</th>
<th>C$_3$A</th>
<th>C$_4$AF</th>
</tr>
</thead>
<tbody>
<tr>
<td>CEM I 52,5R (% / cement)</td>
<td>68</td>
<td>10</td>
<td>9</td>
<td>7</td>
</tr>
</tbody>
</table>

2.2 Method

Samples were cast in PVC moulds (Ø=5.5 cm, h=19 cm) and were unmoulded after 24h. Then, they were kept in water during 48h. After this curing time ($t_0$=3 days), some samples were sawn. 3 cm of each sample on the Top and the bottom were removed to limit the heterogeneity. Two kinds of samples were produced: cut and uncut. Fig. 1 shows the two kinds of samples. The uncut samples were used to validate results of the cut samples by comparison between the mass loss kinetics of the two kinds of samples. The samples cut in slices allow one to obtain the porosity and the saturation profiles.

After this sawing operation, the bottom and the lateral sides of each specimen were sealed with two layers of aluminium foil sheets to obtain a moisture transfer in one dimension. All samples were kept in a chamber with a relative humidity (RH) of 65% at T=20°C in an environment without CO$_2$ (use of soda lime to trap CO$_2$ in the chamber). After $t_0$+28 days, a
part of samples were put in an environment with T=20°C, RH=65% and [CO$_2$]=3%. Throughout the rest of the article, the only drying condition is named condition D and the coupled drying-carbonation condition is named condition C. Characterisations using different techniques presented thereafter were performed on the samples at $t_0=3$ days, $t_0+7$, $t_0+28$ and $t_0+56$ days. In addition to these characterisations, weight monitoring was carried out for all samples.

Porosity accessible to water and saturation degree were obtained by hydrostatic weighting and drying at 80°C. This measurement is performed on 3 samples. For TGA, XRD and MIP analyses, samples were freeze dried for 24h at P=0.1 mbar. These measurements are performed on one sample. Thermogravimetric analysis (TGA) was collected using a NETZSCH STA 409E under nitrogen atmosphere for samples with GGBS and under air for the other samples, heating from 25°C to 1250°C at a heating speed of 10°C/min. For the samples with GGBS substitution, a nitrogen atmosphere is used to prevent slag compounds oxidation which leads to an increase of sample mass. This technique was used to quantify, the amount of Ca(OH)$_2$ and CaCO$_3$ and the chemically bound water content in the samples [10]. The temperature limit between the free water and the chemically bound water is difficult to estimate. Therefore, the following hypothesis was done: the water lost below 105°C is the free water [10]. The mass losses are measured by the derivative method because it is easy to apply and this method is appeared to be reproducible. The Portlandite content is calculated from Eq. 2 and the CaCO$_3$ one from Eq. 3.

$$x_{CH} = \frac{\Delta m_{T_1 \rightarrow T_2}}{m_{s,T=1100°C}} \cdot \frac{MW_{CH}}{MW_w} \cdot 100$$

(2)

where $x_{CH}$ (%) is the Portlandite content per g of cement, $\Delta m_{T_1 \rightarrow T_2}$ (g) is the mass loss between $T_1=465°C$ and $T_2=560°C$ in the CEM I case and between $T_1=430°C$ and $T_2=530°C$ for the GGBS case, $m_{s,T=1100°C}$ (g) is the sample mass at T=1100°C, $MW_{CH}$ (g.mol$^{-1}$) is the Portlandite molecular weight and $MW_w$ (g.mol$^{-1}$) is the water molecular weight.

$$x_{CCR} = \frac{\Delta m_{T=560°C \rightarrow T=1100°C}}{m_{s,T=1100°C}} \cdot \frac{MW_{CCR}}{MW_{CO_2}} \cdot 100$$

(3)
where $x_{\text{Ca}}$ (%) is the calcium carbonate content per g of cement in percent, 

$$\Delta m_{T=560^\circ C, T=1100^\circ C} \text{ (g) mass loss between } T=560^\circ C \text{ and } T=1100^\circ C, m_{T=1100^\circ C} \text{ (g) sample mass at } T=1100^\circ C, \ MW_{\text{Ca}} \text{ (g.mol}^{-1} \text{) calcium carbonate molecular weight and } MW_{\text{CO}_2} \text{ (g.mol}^{-1} \text{) CO}_2 \text{ molecular weight. The average degree of hydration (DoH) is also evaluated with TGA results [10], [11] by using the equation 4.}

$$DoH = \frac{W_D}{W_{D(\text{DoH}=100\%)}} \times 100 \quad (4)$$

where $W_D$ is the chemically bound water which is evaluated by the equation 5 and $W_{D(\text{DoH}=100\%)\text{}}$ is the chemically bound water for a DoH=100%. This value is equal to 24.6% for the CEM I and 23.6% for the CEM I +60% GGBS [12], [13].

$$W_D = W_{D,\text{CH}} + W_{D,\text{C-S-H}} + W_{D,\text{Other_hydrates}} + W_{D,\text{CH-CaCO}_3} + W_{D,\text{C-S-H-CaCO}_3} \quad (5)$$

where $W_{D,\text{CH}}$ is the chemically bound water for the portlandite, $W_{D,\text{C-S-H}}$ is the chemically bound water from the C-S-H, $W_{D,\text{Other_hydrates}}$ is the chemically bound water from the other hydrates, $W_{D,\text{CH-CaCO}_3}$ is the chemically bound water from the portlandite change in CaCO$_3$ and $W_{D,\text{C-S-H-CaCO}_3}$ is the chemically bound water from the C-S-H change in CaCO$_3$. In this study, we do the assumption that the quantity of the chemically bound water the C-S-H change in CaCO$_3$ is negligible.

X-ray diffraction (XRD) patterns were collected with Philips PW3830 equipped with CoK\text{a} radiation source between 4-76°2θ, a step size of 0.02 and a dwell time of 2.0 s were used. This technique was used to determine the presence of crystalline phases (C$_3$S, C$_2$S, C$_3$A, C$_4$AF, Portlandite, Calcite and Ettringite).

Finally, pore size distribution was obtained by Mercury Intrusion Porosimetry (MIP) with a commercial porosimeter (Micromeritics' AutoPore IV 9500 Series) using a maximum intrusion pressure of 400 MPa. This technique is used to assess the pore size distribution in a comparable manner and to obtain a qualitative evolution of this pore size distribution. The porosity obtained by MIP is calculated as the mercury intrusion volume.

The carbonation depth is measured by a colorimetric method based on phenolphthalein spraying tests. The obtained depth correspond to a pH value roughly 9. This implies a sharp carbonation front while this one is normally gradual (profile). Indeed, the main disadvantage of this method is that it can differentiate only between fully carbonated sample and other zones which might vary between being completely unaltered and being almost fully carbonated. This is why, the calcite profile measurements were proposed in parallel to determine the gradual front of carbonation.

3 RESULTS AND DISCUSSION

3.1 MICROSTRUCTURE

Fig. 2a shows the evolution of portlandite content with time and depth for CEM I cement paste. At $t_0$, the portlandite content per g of cement is the same in the entire sample and around 24%. For each depth, the portlandite content increases with the time between $t_0$ and 28 days. This is due to the hydration of clinker. Moreover, in the condition D, the Portlandite content is
constant between 28 and 56 days (blue and purple lines). This finding is the same in the condition D where the Portlandite content is also the same at the depths of 1-1.5 cm and 6.5-10.0 cm. The Portlandite content in the GGBS cement paste is lower than in the CEM I cement paste. This is due to the clinker dilution. Nevertheless, at the depth of 0-0.5 cm, the Portlandite contents decrease due to carbonation which is confirmed by the Fig. 3a&b where the CaCO₃ contents are constant at all the time except for the surface at t₀+56 days with the condition C for both binders. In the GGBS cement paste, the CaCO₃ contents stays lower (4.0%) than in the case of CEM I (7.8%). This result is confirmed by carbonation depth measurement at t₀+56 days with phenolphthalein spraying; a carbonation depth of 0.81±0.24 mm is obtained for the CEM I and 0.55±0.40 mm for the GGBS cement paste. These results need to be confirmed for longer time scales. For both binders at all the time in the condition D, the CaCO₃ contents near to the surface are higher than the other depths. This is due to the carbonation of a thin layer at the surface.

Figure 2: Evolution of Portlandite content with time for PCM (a) and for P6S (b)
D: drying condition – C: coupled drying and carbonation conditions

Figure 3: Evolution of CaCO₃ content with time for PCM (a) and for P6S (b), D: drying condition – C: coupled drying and carbonation conditions (dashed line = carbonation depth according to phenolphthalein)
Furthermore, the degree of hydration (DoH) is calculated by the method described by Bhatt and Pane [10], [11]. For the CEM I cement paste (see Fig. 4a), results can be summarized as following:
- At the depth of 0.0-0.5 cm, the DoH increases until \( t_0 + 28 \) days, it is unchanged for \( t_0 + 56 \) days in the condition D. Therefore, the saturation degree threshold, below which the hydration cannot be possible, is deeper than 0.5 cm between \( t_0 + 7 \) days and \( t_0 + 28 \) days. At \( t_0 + 56 \) days in the condition C, the DoH is higher than at \( t_0 + 28 \) days, which is probably due to the water produced during the carbonation reaction (see eq 1).
- At the depth of 1.0-1.5 cm, the DoH increases until \( t_0 + 28 \) days, it is unchanged for \( t_0 + 56 \) days in both conditions. Therefore the saturation degree threshold is deeper than 1.5 cm between \( t_0 + 7 \) days and \( t_0 + 28 \) days.
- At the depth of 6.5-10.0 cm, the DoH increases until \( t_0 + 56 \) days. Therefore at this depth the drying does not occur.

For the slag cement paste (see Fig. 4b), results can be summarized as following:
- At the depth of 0.0-0.5 cm, the trend is the same than the CEM I but the DoH is lower because the slag reaction rate is slower than the clinker one.
- At the depth of 1.0-1.5 cm, the degree of hydration increases until \( t_0 + 56 \) days in the drying condition. Therefore the saturation degree threshold is between 0.5 and 1.0 cm for a time \( t_0 + 28 \) days and \( t_0 + 56 \) days.
- At the depth of 6.5-10.0 cm, the DOH increases until \( t_0 + 56 \) days.

At \( t_0 \), the DoH is higher at the surface than in the bulk in both cases. The surface of GGBS cement paste dries faster before \( t_0 + 7 \) days than the CEM I one. After \( t_0 + 28 \) days, drying has an impact at the depth of 1.0-1.5 cm in the CEM I case while it is not the case for the GGBS cement paste. Therefore, after a faster drying at the surface at early age, the drying seems to slow down for the slag cement after \( t_0 + 28 \) days (see Fig 6). This can be due to a finer porosity of GGBS cement paste as see in Fig. 8b.

According to XRD results, the following phases are present in CEM I and GGBS cement pastes: Portlandite, Calcite, C-S-H, Ettringite, monosulfoalumiate, C\(_2\)S, C\(_3\)S, C\(_3\)A, C\(_4\)AF. Gehlenite and Wolastonite are only present in GGBS cement pastes. For the CEM I cement paste at a depth of 0.0-0.5 cm (Fig. 5a):

![Figure 4: Evolution of degree of hydration with time for PCM (a) and for P6S (b) [11]](image-url)
- The calcite peak seems unchanged for condition D except for condition C. It increases compared to that at $t_0+56$ days in condition D. This result is confirmed by the TGA.
- The $C_2S$ and $C_3S$ peak intensities seem reduced between $t_0$ and $t_0+28$ days, between $t_0+28$ days and $t_0+56$ days the intensities are quite similar.

For the GGBS cement paste (Fig. 5b):
- The calcite peak seems unchanged except for the condition C. It increases compared to that at $t_0+56$ days in condition D. This result is confirmed by the TGA. Vaterite and Aragonite are not found in the sample in opposite to other studies [14], [15]. Therefore the carbonation of C-S-H does not occur during the experiment, only the carbonation of Portlandite [14].

The calcite peak is higher in the CEM I paste than in the slag cement paste which is confirmed by TGA. This result can be explained by higher Portlandite and other hydrates contents in the CEM I paste compared to the slag cement paste.

![Figure 5: XRD pattern at the depth of 0.0-0.5 cm for PCM (a) and for P6S (b) at several times](image)

### 3.2 Mass loss kinetics

Evolutions of mass loss per drying surface versus the square root of time are shown in Fig. 6a for the CEM I cement paste and Fig. 6b for the GGBS cement paste. In the CEM I cement paste, the evolution is linear in the square-root of time scale with a slope of $-510$ g/($m^2$.d$^{0.5}$) and the repeatability seems correct. Therefore, there is no significant change in the microstructure during drying which started after 3 days of curing. In the case of CEM I +60% GGBS, the evolution is non linear (Fig. 6b). The slope changes around 1 week. This is due to the drastic microstructure change when the GGBS reactions take place [16]. Indeed, the result is confirmed by the attenuation of the slope in the GGBS case, before 1 week the slope is $-1200$ g/($m^2$.d$^{0.5}$) and after the slope value is $-210$ g/($m^2$.d$^{0.5}$). Therefore, the GGBS cement paste drying is slower than the CEM I one after 1 week. This can be due to a finer porosity and therefore a lower apparent diffusivity. In the Fig. 5a, the ratio mass variation to surface is smaller in the condition C than the condition D. This is due to the carbonation which leads to a mass increase by $CO_2$ binding. The difference between the conditions D and C is less important for the GGBS cement paste. This difference can be explained by lower $CO_2$ binding as shown with the TGA results.
8

3.3 Pore structure

Fig. 7a shows the pore size distribution at the surface for the CEM I cement paste. Between t₀ and t₀+56 days, in condition D, the pore structure becomes a little coarser while the porosity decreases during the hydration process. The pore diameters are smaller at t₀+56 days in the condition C than in only the condition D.

Fig. 7b shows the pore size distribution at the surface for a GGBS cement paste. Between t₀ and t₀+56 days in both conditions, the pore radius decreases due to the hydration. Concerning the case with or without carbonation, the pore size distribution stays quite the same in both conditions at t₀+56 days because this phenomenon is not enough developed in the slag cement paste, as shown by the carbonation depth measurements.

In Fig. 8a, the pore diameter decreases between t₀ and t₀+56 days from 220 nm to 20 nm due to hydration process. The same trend is highlighted in the Fig. 8b for the blended slag paste where the pore diameter decreases from 630 nm to 11 nm. At the depth of 6.5-10.0 cm, samples
are subjected to neither drying nor carbonation. At the surface, the GGBS cement pastes have a coarser porosity than the CEM I cement paste one due to the drying. The GGBS cement pastes have a finer pore structure than the CEM I cement paste one at a depth of 6.5-10.0 cm without drying. These results can explain the kinetics of the mass loss observed in Fig 6.

The comparison between the porosity obtained by MIP measurement and the porosity accessible to water is shown in Fig.9 and table 3. The MIP porosity values are smaller than water porosity values because the mercury does not have access to all the porosity. The difference between the 2 methods of measurement increases with the time. For both binders, in the condition D, the porosity decreases due to the hydration process which contributes to fill the porosity by hydrates formation. For the CEM I cement paste in the condition C, the porosity decreases for the depth 0.0-0.5 cm due to the carbonation treatment. Moreover, the MIP results show a decrease of pore size distribution. Therefore, the carbonation leads to filing of the porosity by formation of calcium carbonate, this observation is in agreement with the literature [17]. For the GGBS cement paste, the decrease of porosity for the depth 0.0-0.5 cm is smaller. The porosity increases for the GGBS cement paste in the condition C by MIP. This result may be due to cracking of sample. The GGBS cement pastes have a higher porosity around 10% (measured by MIP) more than the CEM I cement pastes. This is due to slower hydration kinetics as shown in Fig. 4.

Fig. 10 shows the evolution of saturation degree profiles at several times in the conditions C or D for a CEM I cement paste (Fig. 10a) and for the GGBS cement paste (Fig.10b). For both binders the saturation degree decreases when the depth goes to the surface due to drying. For the CEM I cement paste (Fig. 10a), the surface seems to be close to equilibrium with the atmosphere from \( t_0+28 \) days. The depth affected by the drying at \( t_0+7 \) days is 3 cm and 5 cm from \( t_0+28 \) days. For the slag cement paste (Fig. 10b), the surface seems to be close to equilibrium with the atmosphere from \( t_0+7 \) days. The depth impact by the drying at \( t_0+7 \) days is 4 cm and 6 cm from \( t_0+28 \) days. After 56 days profiles for both pastes are very similar. The difference of saturation evolution can be explained by an initial faster drying (\( t < 7 \) days) in the
slag cement paste due to a higher porosity. In both cement pastes, the saturation profiles are similar in the conditions C and D.

![Graph](image)

**Figure 9:** Evolution of porosity with time for PCM (a) and for P6S (b)

**Table 3:** Results of porosity obtained by MIP or accessible to water measurement at several times (Standard deviation (S.D))

<table>
<thead>
<tr>
<th>Depth (cm)</th>
<th>0.0-0.5</th>
<th>1.0-1.5</th>
<th>6.5-10.0</th>
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<tr>
<td></td>
<td>Hg</td>
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<td>water S.D</td>
</tr>
<tr>
<td>CEM I cement paste</td>
<td></td>
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<td></td>
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<td>40.6</td>
<td>47.2</td>
<td>0.16</td>
</tr>
<tr>
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<td>36.5</td>
<td>40.9</td>
<td>0.30</td>
</tr>
<tr>
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<td>38.9</td>
<td>2.57</td>
</tr>
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</tr>
<tr>
<td>56d_C</td>
<td>30.3</td>
<td>38.5</td>
<td>0.01</td>
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<td>GGBS cement paste</td>
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<tr>
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The above described experimental results show evidence difference of the two cement pastes behaviour when they are submitted to the coupled drying-carbonation condition at early age. Indeed, the Portlandite content is lower for the GGBS cement paste due to the dilution. The lower CaCO$_3$ shown by the TGA and XRD results for the GGBS cement paste can be explained by lower hydrates can be carbonated contents related to dilution of OPC. This result is confirmed by the carbonation depth measurement. For both binders at all the time in the condition D, the CaCO$_3$ contents near to the surface are higher than for the other depths. This is due to the carbonation of a thin layer at the surface. This cannot be avoided unless these experiments are performed under air void. The XRD results are only qualitative, to complete this work using of quantitative method is necessary. The drying of GGBS cement paste at early age compared to the CEM I case is faster. However, this trend changes when the GGBS
reactions occur, that are confirmed by the MIP results and by the saturation profiles. Indeed, the pore radii of GGBS cement paste are bigger at early age and consequently, the moisture transfer is easier. While after the GGBS reactions occur, the pore structure is thinner then moisture transfer is slowed down.

Figure 10: Evolution of saturation degree profiles with the time for PCM (a) and for P6S (b)

4 CONCLUSION

- In this work, the coupling process leads to a faster drying of GGBS cement paste at early age compared to the CEM I case. However, this trend changes when the GGBS reactions occur that are confirmed by the MIP results.
- After 3 days of hydration in water, the carbonation depths are similar at early age but a lower carbonation degree suggests a lower diffusivity and finer microstructure for GGBS paste.
- These results of drying and carbonation need to be confirmed for a longer time scale.

5 ACKNOWLEDGEMENTS

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6 REFERENCES


