

Study of Rehydration of Portland Blast-Furnace Slag Cement Subjected to Heat Treatment

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Abstract – Researchers have studied the dehydration and rehydration of Portland cement. In this study, hydrated cement pastes (HCPs) prepared from Portland blast furnace slag cement (PBFSC), were heat treated at 700°C and 900°C and used in the preparation of rehydrated cement powders in air (RCPs-air) and rehydrated cement paste (RCPs) with water/cement ratios (w/c) of 0.3 and 0.5. X-ray diffraction (XRD) analyses of the RCPs-air and RCPs indicated the occurrence of pozzolanic reactions during rehydration. RCPs were also prepared from 0, 10, 20 and 40% RCPs-air relative to PBFSC. RCPs prepared with w/c=0.5 showed higher temperatures than RCPs prepared with w/c=0.3. Pastes prepared with w/c=0.5 from 0, 10 and 20% RCP-air and paste prepared with w/c=0.3 from 0 and 10% RCPs-air, showed similar properties. For concentrations greater than these, there was a decrease in compressive strength.

Keywords – Cement Paste; Rehydrated Cement Paste; Rehydration; Compressive Strength; Porosity.

I. INTRODUCTION

Researchers have studied the behavior of hydrated cement pastes (HCPs) subjected to high temperatures [1], [2], [3], [4], [5], [6], [7], [8], [9], [10], [11]. It is known that HCPs subjected to temperatures between 400 and 800°C can retrieve its hydration capacity [12-14]. The type of cement and the state of the sample influence the dehydration of the hydrated cement paste (HCP) [12]. During heat treatments several changes occur in HCP in certain temperature ranges [12], [16], [17], [18]. At around 400-550°C portlandite (CH) is converted to calcium oxide (CaO) and water [12], [19], [20]. After being exposed to 850°C, the calcium silicate hydrated (C-S-H) with larger 1.5 CaO/SiO₂ ratio, transforms into new nesosilicate whose composition is close to larnite (C₂S) [2], [14], [15]. During the heat treatments the dehydration of C-S-H also contributed to CaO formation [18]. The dehydrated cement powder (DCP) is not stable in humid atmosphere, thus the rehydration and carbonation processes occur in this atmosphere [12], [15], [18]. The dehydrated phases in the DCP react rapidly with water due to the high reactivity of CaO with

it [14]. The rehydrated cement paste (RCP) does not recover the mechanical strength of the original HCP [14]. The RCP has voids that are not filled with C-S-H. The CaO could participate in the rehydration process forming C-S-H, but reacts mainly with water to form Ca(OH)₂ [14], [18]. The first products to be formed in the rehydration process are similar to portlandite and C-S-H [18]. In general the carbonation reactions involve CH, C-S-H, Ca₂SiO₄ (C₂S) and Ca₃SiO₅ (C₃S) [18], [21], [22]. CaCO₃ formed by CaO carbonation during dehydration processes coexists with rehydrated solid phases [14], [21]. The rehydration capacity of DCP is influenced by the dehydration temperature and method of rehydration [14]. For example, if rehydrated cement pastes (RCPs) are soaked in water for 7 days they reach lower compressive strengths than, RCPs exposed to 100% relative humidity for the same period [19].

The dehydration and rehydration of HCPs have been studied under different conditions. Studies have shown that, the x-ray diffraction (XRD) pattern of HCP samples treated at 700°C detected C-S-H as crystalline phase, while samples

treated at 800°C revealed that C-S-H decomposes into β -C₂S and C₃S [23]. Studies have also shown that, the compressive strength of the RCPs prepared with a high w/c ratio was lower than the resistance of the prepared RCPs with a low w/c ratio [24]. Research has shown that RCPs prepared with up to 40% of the cement powders submitted to heat treatment presented similar physical and mechanical properties [25]. It is known that the excess dehydrated cement powders may reduce the cement amount in the system, which can cause a reduction of the compressive strength [26], [27]. However, it is also known that the addition of granulated blast furnace slag (GBFS) contributes to the improved resistance of rehydrated cement [19]. The reason for this is the rehydration reaction between the newly formed CH and the unhydrated pozzolanic particles, that fill the capillaries and decrease the total porosity of the cement paste [19], [28]. Several authors study these pozzolanic reactions under different conditions [29], [30], [31], [32], [33]. Researchers prepared HCPs from different Slag/Ca(OH)₂ ratios. The study shows that the presence of CH substantively activates the slag hydration [34]. In another study, were prepared specimens from slag and heat-treated HCP at 600°C. Slag and dehydrated cement powders were mixed at the mass ratios of 3:1, 2:1, 1:1, 1:2, and 1:3, respectively. The strength of the specimens reached the maximum value when the mass ratio was 1:2 [28]. Researchers prepared specimens from CEM I 42.5 R Portland cement and GBFS. The test specimens were placed in water for 27 days and were subsequently heated in a furnace at different temperatures. The amount of mixing water was variable with the aim of achieving standard consistency for RCPs. The XRD analysis of the PCR samples showed signs related to Portlandite and gismondine. The RCPs prepared starting from the heat treatment of the HCPs at higher temperatures showed higher compression strength [19].

The objective of the present research is to prepare and to study by X-ray diffraction (XRD), rehydrated cement powders in air (PCRs-air) and rehydrated cement pastes (RCPs) prepared using water/cement ratios (w/c) 0.3 and 0.5, starting from hydration, heat treatment at 700 and 900°C and rehydration of Portland blast-furnace slag cement (PBFSC). This research also aims to investigate rehydration temperatures, physical properties and compressive strength of pastes prepared from 0, 10, 20 and 40% of RCPs-air in relation to PBFSC.

II. EXPERIMENTAL

2.1 Materials and methods

In this study the Portland blast-furnace slag cement (PBFSC) was used as the binder for the experiments. The

physical and chemical properties of cement are listed in Table I. The hydrated cement pastes (HCPs) were prepared by using the water/cement (w/c) ratios of 0.3 (0.3-HCP) and 0.5 (0.5-HCP). The HCPs were left in the molds for 24 hours and then air-cured at laboratory temperature ($25 \pm 2^\circ\text{C}$) for 28 days. The 0.3-HCP and 0.5-HCP were heat treated at 700°C, termed 0.3-700-HCP and 0.5-700-HCP, respectively. The 0.3-HCP and 0.5-HCP also were heat treated at 900°C, termed 0.3-900-HCP and 0.5-900-HCP, respectively. After the curing period, HCPs were crushed into particles smaller than 0.7 mm by a laboratory jaw crusher. The HCPs powders were sieved and submitted to the controlled heating regime. The heating rate was 10 °C/min starting at room temperature. When the desired temperature was reached the powders were held for 1.5 hours in the oven. After cooling, these HCPs treated at 700 and 900°C were rehydrated in air at relative humidity (RH)>70% (RCPs-air) for 28 days, then 0.3-700-HCP, 0.5-700-HCP, 0.3-900-HCP and 0.5-900-HCP were denominated as, 0.3-700-RCP-air, 0.5-700-RCP-air, 0.3-900-RCP-air, and 0.5-900-RCP-air, respectively. After this time, the rehydrated cement pastes (RCPs) were prepared from 0.3-700-RCP-air, 0.5-700-RCP-air, 0.3-900-RCP-air and 0.5-900-RCP-air being denominated as, 0.3-700-RCP, 0.5-700-RCP, 0.3-900-RCP and 0.5-900-RCP, respectively. The w/c ratios of 0.3 and 0.5 were used to prepare the RCPs. All HCPs, RCPs-air and RCPs were separated for testing. Specimens of HCPs and of RCPs were prepared. Prior to the preparation of the RCPs specimens, RCPs-air and PBFSC were mixed in a mechanical mixer for 10 min. RCPs specimens were prepared from 10, 20 and 40% RCPs-air relative to the PBFSC for each w/c ratio. The RCPs were left in the mold for 24 hours and then cured at laboratory temperature ($25 \pm 2^\circ\text{C}$) for 28 days, then separated for testing. Twenty specimens of HCPs and RCPs were prepared for each condition, obtaining a total of 280 specimens.

Tabela I: Propriedades físicas e composição química do cimento.

Chemical analysis (%)	
SiO ₂	21.85
Al ₂ O ₃	4.96
Fe ₂ O ₃	1.83
CaO	63.57
SO ₃	1.58
MgO	1.47
TiO ₂	0.16
Alkalis	0.67
L.O.I	4.98
Physical properties	
Specific gravity (g/m ³)	3.11

Surface area (m ² /g)	0.64
Initial setting time (min)	228
Final setting time (min)	395
Requiring water for normal consistency	0.28
Components of Cement (%)	
Clinker + Gypsum	56-94
Granulated blast-furnace slag	6-34
Calcareous	0-10

2.2 Characterization and tests

Samples of HCPs, RCPs-air and RCPs were characterized by X-ray diffraction (XRD). The phase analysis was done by a Shimadzu XRD 6000 equipment. The phases were identified by comparison with the JCPDS database. For the temperature tests, samples of 0.3-HCP, 0.5-HCP and of RCPs were prepared from 10, 20 and 40% of RCPs-air in relation to the PBFSC. During the semi-adiabatic measurements (Langavant's calorimeter) increases in the temperatures of HCPs and RCPs were monitored. The temperature tests were determined according to the adapted NBR 12006 standard.

The physical and mechanical properties were obtained for 0.3-HCP, 0.5-HCP and RCPs specimens prepared from 10, 20

and 40% of RCPs-air in relation to PBFSC. Water absorption and apparent porosity were determined according to the adapted BS EN ISO 10545-3 standard, with 10 specimens being used for each condition. The dimensions of the cylindrical specimens were 50 mm in diameter and 50 mm in height. The relationship between the accessible pore volumes was calculated by evaluating the difference between saturated and dry weights.

The compressive strength tests were performed at room temperature in a universal mechanical testing machine Shimadzu AG-X plus (100 kN), with 10 specimens being used for each condition. The specimens were prepared according to the specifications provided by the adapted NBR 7515. The dimensions of the cylindrical specimens were 50 mm in diameter and 100 mm in height.

III. RESULTS AND DISCUSSION

3.1 Phase Analysis by X-Ray Diffraction (XRD)

The X-ray diffractograms of hydrated cement pastes (HCPs), rehydrated cement paste in air (RCPs-air) and rehydrated cement pastes (RCPs) samples prepared from Portland blast-furnace slag cement (PBFSC) are shown in Figure 1.

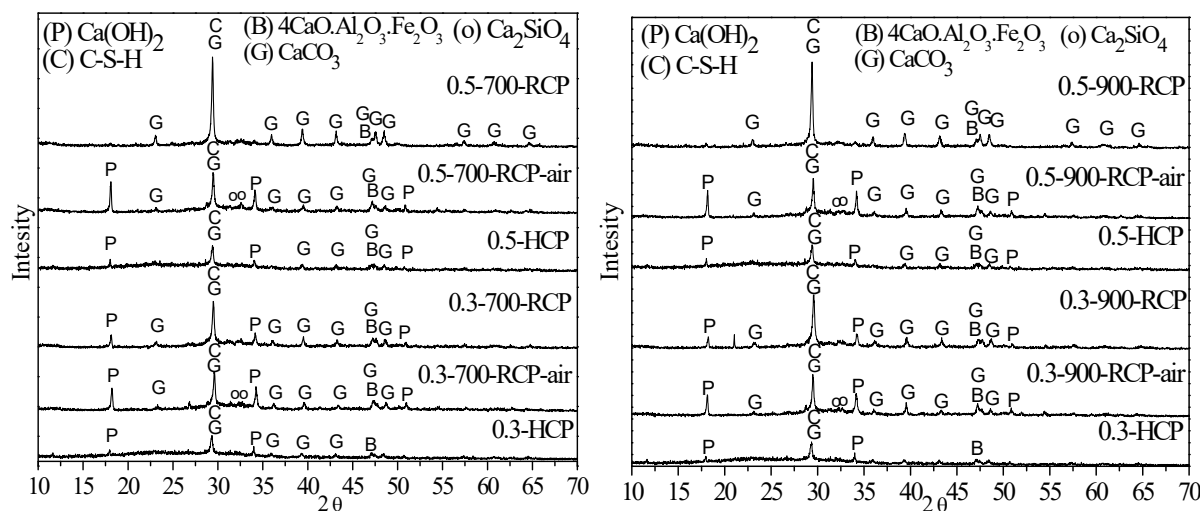


Figure 1: X-ray diffractograms of the hydrated samples and rehydrated samples prepared from the rehydration of 0.3-700-HCP and 0.5-700-HCP (left). X-ray diffractograms of the hydrated samples and rehydrated samples prepared from the rehydration of 0.3-900-HCP and 0.5-900-HCP (right).

During curing of a cement paste the formation of $\text{Ca}(\text{OH})_2$ [14, 15, 20] and C-S-H [24], [35], [36], [37], [38] occur. XRD-analysis of the HCP samples revealed $\text{Ca}(\text{OH})_2$ and C-S-H as the principal hydrated components. The peaks at around

18.21° and $34.18^\circ(2\theta)$ can be attributed to $\text{Ca}(\text{OH})_2$, and the peak at around $29.35^\circ(2\theta)$ can be attributed to C-S-H. Peaks around 29.35° , 36.01° , 39.47° , 43.18° and $48.50^\circ(2\theta)$ were identified in all samples, and can be attributed to the CaCO_3

present in PBFSC and to products of carbonation reactions. The high reactivity of the heated cement powders causes it to react with the water present in the air [12], [14], [15], [18], [24]. XRD analysis of all RCPs-air samples presented intense peaks that can be attributed to Ca(OH)_2 and C-S-H. This can also be related to the fact that the heat treated powders are stored in air with relative humidity (H.R.) > 70%. Low-intensity peaks at around 32.01° and $33.12^\circ(2\theta)$ which can be attributed to C_2S and C_3S respectively, were also identified in a RCPs-air samples. XRD-analysis of the 0.3-700-RCP and 0.3-900-RCP samples revealed the presence of Ca(OH)_2 and C-S-H. The characteristic peak of C-S-H increases whereas the peaks which can be attributed for Ca(OH)_2 decrease, when compared to the XRD-analysis of the 0.3-700-RCP-air and 0.3-900-RCP-air samples. XRD-analysis of the 0.5-700-RCP and 0.5-900-RCP samples did not reveal presence of Ca(OH)_2 . The 0.5-700-RCP and 0.5-900-RCP samples presented more intense peaks attributed to C-S-H than the samples of 0.5-700-RCP-air and 0.5-900-RCP-air, respectively. This may have occurred due to the marked advance of the pozzolanic reactions in the RCPs samples prepared with w/c ratios of 0.5. This advance consists of the reaction between the newly formed Ca(OH)_2 and the unhydrated pozzolanic particles. The continuous activation of the slag by CH allows formation of C-S-H.

3.2 Semi-adiabatic calorimetry analysis

Figure 2 show the semi-adiabatic calorimetry curves for hydrated cement pastes and (HCPs) and rehydrated cement pastes (RCPs) samples prepared from 10, 20 and 40% of RCPs-air in relation to PBFSC. Slag mixed in Portland cement has a complicated hydration mechanism and its reactivity is significantly influenced by CH, which acts as an activator inducing a pozzolanic reaction to produce binders [19], [32], [39], [40]. According to some researchers this activation is effective when the pH of the aqueous phase is higher than 11.5 [41]. The high concentration of OH^- anions causes the progressive dissolution of the slag and precipitation of C-S-H, leading to the increase in temperature of the cement paste [42], [43], [45]. The data from semi-adiabatic calorimetry show that RCPs samples prepared with higher concentrations of RCPs-air, presented higher temperatures than RCPs samples prepared with lower concentrations of RCPs-air. Additionally it is possible to say with the study of the semi-adiabatic calorimetry curves that, the RCPs samples prepared with w/c=0.5 presented higher temperatures than the RCPs samples prepared with w/c=0.3. This may have occurred due to the marked advance in the dissolution of the slag and the pozzolanic reactions in RCPs samples prepared with more water.

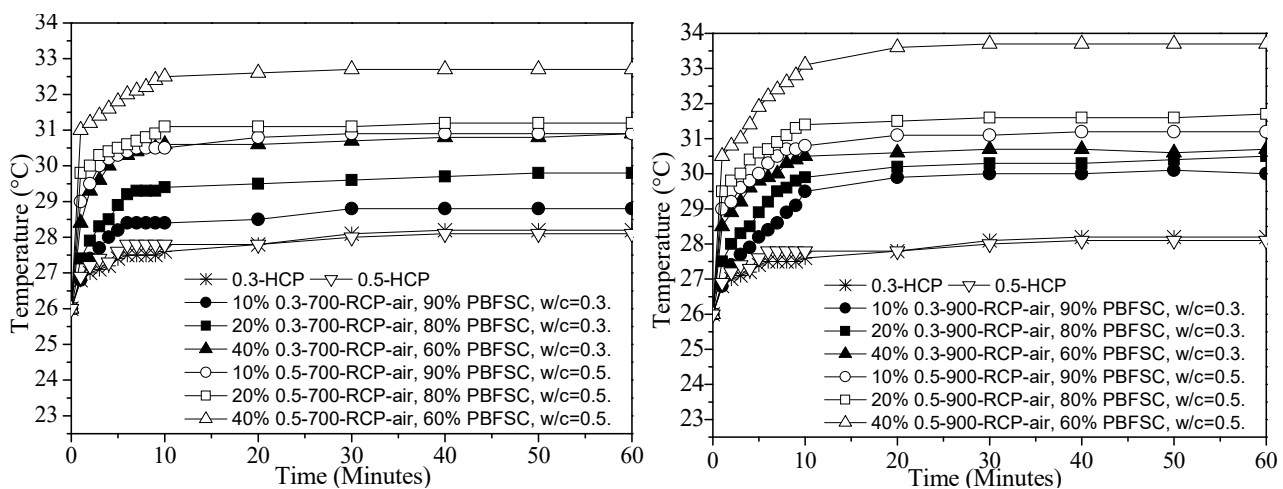


Figure 2: Semi-adiabatic calorimetry curves for HCPs and RCPs prepared from 10, 20, and 40% of 0.3-700-RCP-air and 0.5-700-RCP-air (Left) and semi-adiabatic calorimetry curves for HCPs and RCPs prepared from 10, 20, 40% of 0.3-900-RCP-air and 0.5-900-RCP-air (Right).

3.3 Physical properties

Figure 3 show the water absorption for, hydrated pastes and cement (HCPs) and rehydrated cement pastes (RCPs) prepared from 10, 20 and 40% of RCPs-air in relation to Portland blast-furnace slag cement (PBFSC). Figure 4 show

the apparent porosity for, HCPs and RCPs prepared from 10, 20 and 40% of RCPs-air relation to PBFSC. It is known that it is not possible to recover cement hydrates completely in a RCP [14], [18]. The results show that, 0.3-HCP and RCPs prepared from 10% of 0.3-700-RCP-air and from 10% of 0.3-

900-RCP-air, presented similar values of these physical properties. The results also show that, 0.5-HCP, RCPs prepared from 10 and 20% of 0.5-700-RCP-air and from 10 and 20% of 0.5-900-RCP-air also presented similar values of these properties. On the other hand, RCPs prepared from 20 and 40% of 0.3-700-RCP-air and from 20 and 40% of 0.3-900-RCP-air, presented higher values of these properties when compared to the 0.3-HCP. RCPs prepared from 40% of 0.5-700-RCP-air and from 40% of 0.5-900-RCP-air also presented higher values of these properties when compared to the 0.5-HCP. For example, the mean values of water absorption and apparent porosity for 0.3-HCP were 3.65% and 6.59% respectively, and the mean values of these properties for 0.5-HCP were 6.58% and 10.54% respectively. The mean values of water absorption and apparent porosity for RCPs prepared from 40% of 0.3-900-RCP-air were 25.29% and 29.93%

respectively, while RCPs prepared from 40% of 0.5-900-RCP-air presented mean values of water absorption and apparent porosity of 12.02% and 19.53%, respectively. The mean values of these properties for RCPs prepared from 40% of 0.3-700-RCP-air and from 40% of 0.5-700-RCP-air were similar, assuming mean values of water absorption and apparent porosity around 11.80% and 18.80% respectively. The increase of these properties may be related to the formation of voids in RCPs that were not filled with cement hydrates. Although certain amounts of C-S-H exist, the polymerization of rehydration products to form a dense body can not take place. The increase of these physical properties of the RCPs specimens prepared with $w/c=0.5$ may have been attenuated by pozzolanic reactions, that occurred more intensively in these cement pastes.

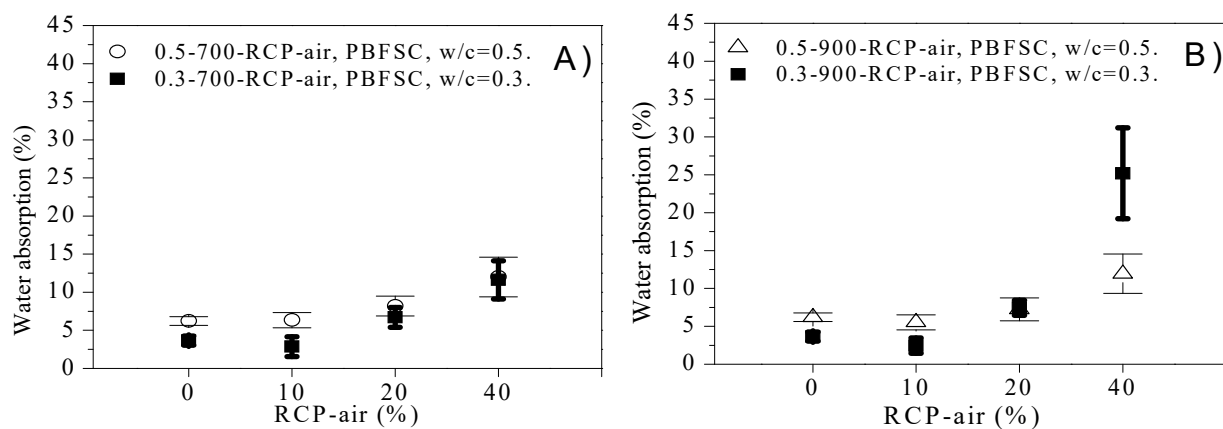


Figure 3: Water absorption for HCPs and RCPs prepared from 10, 20 and 40% of 0.5-700-RCP-air and 0.3-700-RCP-air in A) and for HCPs and RCPs prepared from 10, 20 and 40% of 0.5-900-RCP-air and 0.3-900-RCP-air in B).

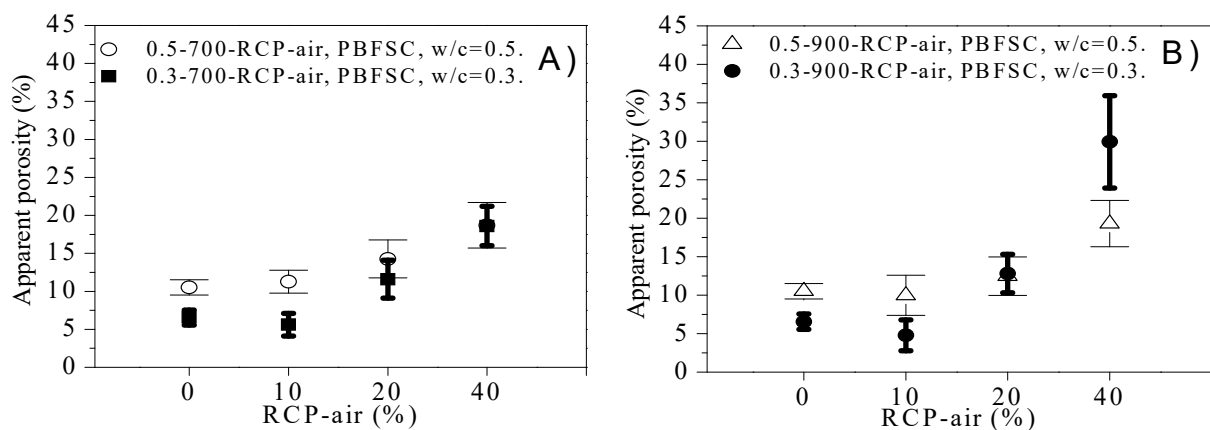


Figure 4: Apparent porosity for HCPs and RCPs prepared from 10, 20 and 40% of 0.5-700-RCP-air and 0.3-700-RCP-air in A) and for HCPs and RCPs prepared from 10, 20 and 40% of 0.5-900-RCP-air and 0.3-900-RCP-air in B).

3.4 Compressive strength

Compressive strength for hydrated pastes and cement (HCPs) and rehydrated cement pastes (RCPs) prepared from 10, 20 and 40% of RCPs-air in relation to PBFSC are shown in Figure 5. The results show that 0.3-HCP and RCPs prepared from 10% of 0.3-700-RCP-air and 10% of 0.3-900-RCP-air, presented similar values of compressive strength. The results also show that, 0.5-HCP and RCPs prepared from 10 and 20% of 0.5-700-RCP-air and 10 and 20% of 0.5-900-RCP-air, also presented similar values of this mechanical property. On the other hand, RCPs prepared from 20 and 40% of 0.3-700-RCP-air and 20 and 40% of 0.3-900-RCP-air, presented lower values of this mechanical property when compared to 0.3-HCP. RCPs prepared from 40% of 0.5-700-RCP-air and 40% of 0.5-900-RCP-air, also presented lower values of compressive strength when compared to 0.5-HCP. For example, the mean values of compressive strength for 0.3-

HCP and 0.5-HCP were 38.18 MPa and 24.0 MPa, respectively. The mean value of compressive strength for RCPs prepared from 40% of 0.3-900-RCP-air was 3.06 MPa, while RCPs prepared from 40% of 0.5-900-RCP-air presented a mean value of compressive strength of 12.73 MPa. The mean values of this property for RCPs prepared from 40% of 0.3-700-RCP-air and from 40% of 0.5-700-RCP-air are similar, around 14.59 MPa. This decrease is related to the decrease of cement hydrates that are responsible for the mechanical resistance, and the increase of voids present in the RCPs that occurs when it is prepared from higher RCP-air concentrations. It is also possible to note that the decrease in compressive strength is less pronounced for RCPs specimens prepared with w/c ratios of 0.5. This occurs because RCPs prepared with more water allow the advance of chemical reactions with formation of C-S-H, involving the pozzolanic reactions between Ca(OH)_2 and slag.

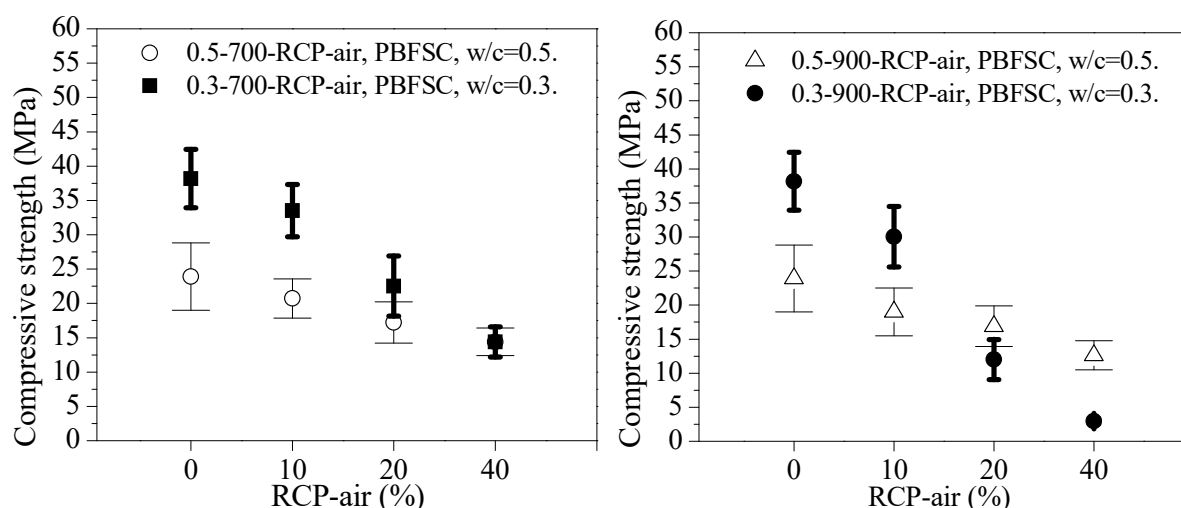


Figure 5: Compressive strength for the RCPs prepared from 10, 20, and 40% of 0.3-700-RCP-air and 0.5-700-RCP-air (Left) and compressive strength for the RCPs prepared from 10, 20, 40% of 0.3-900-RCP-air and 0.5-900-RCP-air (Right).

IV. CONCLUSION

Based on the results of this research, the main final considerations are summarized as follows: i) It is possible to obtain air rehydrated cement powders with relative humidity (RH) > 70% (RCPs-air) containing Ca(OH)_2 , starting with heat treatment at 700°C and at 900°C of hydrated cement pastes (HCPs) prepared from Portland blast-furnace slag cement (PBFSC); ii) Pozzolanic reactions involving Ca(OH)_2 and slag, occur in the rehydrated cement pastes (RCPs), being more intense in the cement pastes prepared with w/c=0.5 than in the pastes prepared with w/c=0.3; iii) HCPs prepared with a/c=0.3 and RCPs prepared from 10% RCPs-air in relation to PBFSC, have similar physical and mechanical properties.

HCPs prepared with a/c=0.5 and RCPs prepared from 10 and 20% RCPs-air, also have similar physical and mechanical properties. For concentrations greater than these, the decrease in compressive strength for RCPs occur. This decrease in compressive strength for RCPs is less pronounced as these are prepared with higher amounts of water, this is due to the greater advance of the pozzolanic reactions with the formation of C-S-H in these rehydrated pastes. Thus, these RCPs prepared with more water present greater potential for the development of pastes with lower porosity and higher mechanical resistance; iv) RCPs prepared with w/c=0.3, prepared from 20 and 40% of 0.3-HCP heat treated at 900°C, shows lower compressive strength than RCPs prepared from 20 and 40% of 0.3-HCP heat treated at 700°C.

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