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STUDY OF THE HYDRATION PROCESS OF PORTLAND CEMENT BY DIFFERENT CHARACTERIZATION TECHNIQUES

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Abstract. *Portland cement is a hydraulic binder that hardens by reacting with water. The phase composition and structure of Portland cement, as its mechanical properties, are related to its hydration process. In this work samples of Portland cement with a water/cement ratio of 0.5 were prepared and the samples analyzed after 1, 3, 7, 14, 21 and 28 days. The hydrated samples were characterized by their mechanical compression strength, phase composition by x-ray diffraction analysis and infrared spectroscopy (FTIR) and the microstructure by scanning electron microscopy (SEM). The hydration process during curing has been verified by the formation of hydrated calcium silicate (CSH) and portlandite $Ca(OH)_2$. The microstructural analysis revealed the formation of needle shaped ettringite crystals and portlandite. This work pretends to contribute to the study of the microstructure and mechanical properties due to the hydration process of Portland cement.*

Keywords: *Portland cement, hydration, mechanical properties, microstructure.*

1. INTRODUCTION

Portland cement is considered one of the most important materials of humanity. (Escalante-Garcia, J.-I and Sharp, J.H, 2004). Thus the knowledge of its hydration process is of paramount importance, since this gradual process gives important physical and mechanical properties. The hydration process of Portland cement occurs after contact with water, starting the hardening of the folder. rapidly between its components and starts hardening of the paste. This hydration can be monitored and analyzed by observing the change of the microstructure as well as the increase in the compressive strength.

According to Dias, *et al* 2006 hydration CP evolves with time, and after 28 days of curing about 70-80% degree of hydration and practically completing the 365 days. The hydration process depends on the kind and fineness of the cement, the ratio between water and cement, the curing temperature and the presence of chemical additives and minerals, as well as the temperature in the initial phase (Lothenbach, B *et al* 2007).

The products of hydration responsible for contributing to the mechanical strength is dicalcium silicate and tricalcium silicate (Birchall, J.D *et al* 1978). The product of the hydration of silicate is CSH (calcium-silicate-hydrate). The hydration of alite is primarily responsible for the initial resistance and by its characteristic reaction to occur quickly, along with the tricalcium aluminate (C3A), still considered your hydration reaction is even faster, with greater release of heat hydration and requires the addition of gpsita (gypsum) to retard the reaction rate (Stutzman, P. 1999). This reaction leads to the formation of ettringite ($C_6AS_3H_{32}$) known to have needle shape and it is possible to observe the formation of the same moments after the start of the handle of the cement (Andersen, M *et al* 2004). It is considered as a balance of hydration phase between two and three months (Nielsen, E.P *et al* 2005). The reaction that follows describes this hydration:

$$C_6AS_3H_{32} + 2\ C_3A \rightarrow C_4ASH_{18} \quad (1)$$

Subsequently starts hydration of silicates. The hydration reactions of silicates to form CSH and portlandite $Ca(OH)_2$. From images of scanning electron micrograph is possible to observe the formation of fibers of hexagonal plates of CSH and portlandite. The reaction that follows describes the hydration of silicates:

$$2C_3S + 6H_2O \rightarrow CSH + 3\ Ca(OH)_2 \quad (2)$$

$$2\ C_2S + 4H_2O \rightarrow CSH + Ca(OH)_2 \quad (3)$$

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The study of hydration of CP confers knowledge imprensindível for better structural properties. (Monteiro, 1993. Feldman, 1972). Were studied the hydration of Portland cement paste (CP-V) by different characterization techniques, these being: X-ray diffraction, infrared spectroscopy, scanning electron microscopy and axial compression test in order to show the process of hydration CP-V from its initial phase.

2. MATERIALS AND METHODS

In order to better study the hydration process of the CP evaluated the hydration process by various characterization techniques.

Initially samples were prepared at a ratio of CP H2O/CP 0.5 and characterized by X-ray diffraction (XRD), infrared spectroscopy (FTIR) and Scanning Electron Microscopy (SEM) at a curing time of 1 , 3, 7, 14, 21 and 28 days. Samples prepared with the same ratio were mechanically tested at a curing time of 7, 14, 21 and 28 days. The cement used for this work was the CP V - ARI characteristic for presenting grabs fast and high early strength.

Cimento Portland CP V – ARI:

According to Costa, C. et al (2009) cement is usually marketed in the form of particles having dimensions between 1 and 90 microns and essentially consisting of: tricalcium silicate (C3S - 30 to 70%), dicalcium silicate (C2S - 10 to 55%); aluminate tricalcium phosphate (C3A - 0 to 15%); aluminoferrato tetracalcium (C4AF, 5 to 15%) and calcium sulphate ie, gypsum (CS - 3 to 8%).

In its most general concept is the cement hydraulic binder able to handle both in the air and in the water. The CP V - ARI stands for designating types of cement with the main characteristic of high early strength. According to the standard (NBR 5733), the composition of CP V ARI understands the data contained in Table 1:

Table 1. Concentrations of the components of Portland cement of high early strength

	Components (% mass)	
Sigla	Clinker + sulphates	Carbonate material
CP V ARI	100 - 95	0 – 5

Characterization of CP paste:

Was performed to identify hydrated products via X-Ray Diffraction (XRD) held in a device of the diffractometer model Shimadzu XRD-6000, which performs measurements of X-ray diffraction powder. The hydrated pastes were crushed and sieved through sieve 80 MESH. The results prove the hydration of the samples.

Using infrared spectroscopy (FTIR) brand Perkin Elmer the process of hydration of the samples was studied by CP in the infrared spectra. It was possible to identify bands CSH and portlandite Ca (OH) 2, both products of hydration.

The technique of Scanning Electron Microscopy (SEM), allows to study the microstructure of CP hydrated, thus being able to better monitor the hydration process of the CP compounds. From waste fractures CPs held allowing the characterization also qualify the hydration products.

Preparation of test specimens and mechanical compression:

The preparation and curing were carried out under the NBR 5739 with dimensions 10 cm high and 5 cm in diameter. The specimens were demolded after its curing time, to perform the compression tests. The specimens were prepared mechanically tested in axial compression machine, as shown in Figure 1.

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Figure 1. Photo of the axial mechanical compression.

3. RESULTS AND DISCUSSION

X-ray diffraction (XRD):

CP samples were crushed and sieved analyzed. The results of XRD analysis are shown in Figure 2.

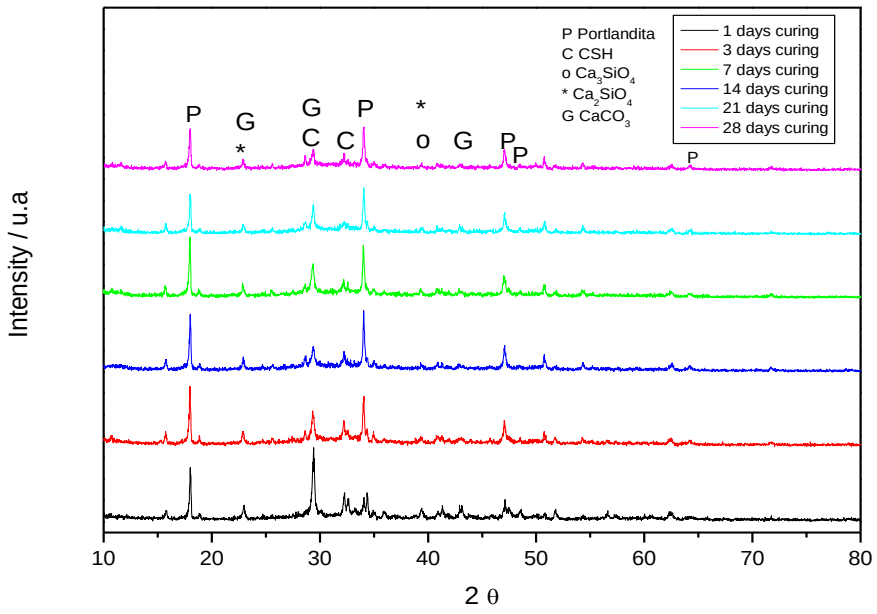

Figure 2. X-ray diffraction of samples of CP hydrated.

Peaks in the XRD patterns show hydration since its inception, with the formation of hydration products, as well as CSH, Portlandite, Alita and Belita since the beginning of the healing process. The main crystalline phase hydrated CP detected in all five peaks were identified as calcium hydroxide (portlandite). CSH peak was identified at 29.5 ° and 32.2 ° (2θ). Has located a peak sicilicato tricalcium was located 39.2 ° (2θ). At 22.6 ° and 39.2 ° (2θ) peak was located dicalcium silicate. Possibly, other peaks were not identified by the possibility of major peaks in subreposição. All stages analyzed qualitatively show peaks related to hydrated products.

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Infrared spectroscopy (FTIR):

CP hydrated samples were analyzed by infrared region. Results are shown in Figure 3:

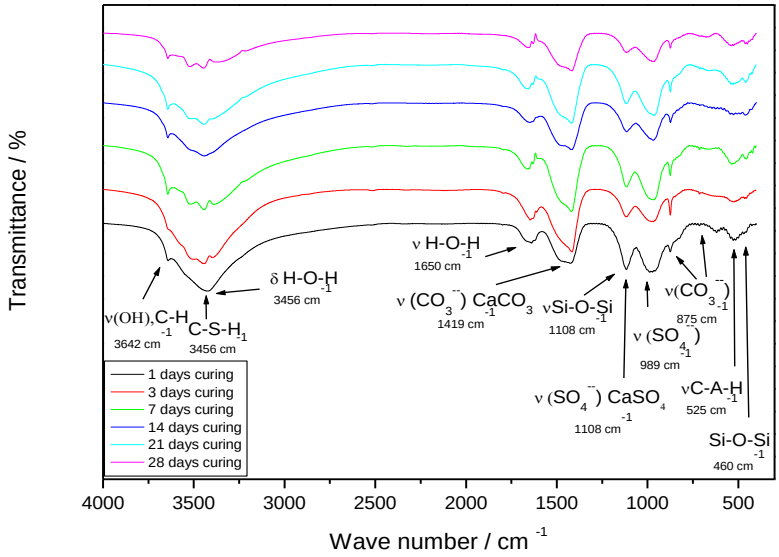

Figure 3. Infrared spectra of samples of CP hydrated.

With the analysis of Figure 3, it is possible to observe the sample spectrum bands CP located at 3456 cm^{-1} and 1650 cm^{-1}, which can be attributed to stretching and deformation vibration of bonds present in the molecules of water physically adsorbed to sample. It is possible to observe the presence of the band located at 3642 cm^{-1}, which can be attributed to stretching of OH bond in the present portlandite. CSH bands can be observed at 3456 cm^{-1}. Silicates bands can be identified in 1108 cm^{-1}. Bands were identifed aluminate at 525 cm^{-1}. The spectra show the hydration of samples of CP.

Scanning Electron Microscopy (SEM):

The results of the SEM analyzes in Fig 4 from a fractured surface of the PC, shows the hydration process. The results have for 1 day cure, has not yet been detected by the hydration products perfectly. In other images we note the appearance of ettringite needles, fibrous matrix of CSH and portlandite crystals, performing in hexagonal plates.

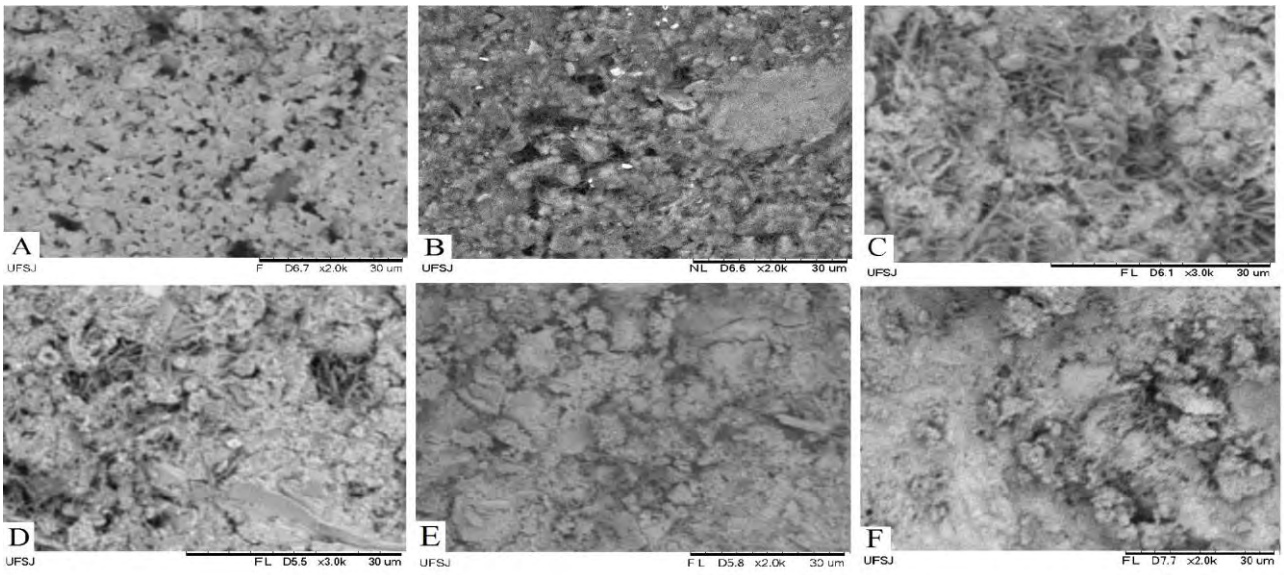

Figure 4. Scanning Electron Microscopy of CP hydrated.

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In figure A and B, the first 3 days of curing, it is possible to note the onset of hydration grains CP. The appearance of the hydration products can not so clear, but it is possible to note the initial crystals foramação portlandite, which can not be well to note the formation of ettringite needles and fibrous deposits CSH. In Figures C and D after 7 and 14 days of curing, the needles of ettringite, CSH fibrous deposits and portlandite crystals can be clearly vizualizadas. E and F in Figures 21 and 28 days of curing, presents itself as a large formation of hydrated products with large plates portlandite well defined. The results of SEM and show correlate with other techniques resutlados obtained in the characterization.

Mechanical compression axial:

Table 2 shows the results of the compressive strength (Rc):

Table 2. Result of axial compression

Cure time	Rc (Mpa)
7 days	28,3
14 days	31,2
21 days	31,7
28 days	34,6

As expected, the strength increases gradually with Portland cement hydration process of the compounds conferring mechanical resistance to the cement paste.

The mechanical test results correlate well with the other techniques, showing good alternatives for the study of cement hydration.

4. CONCLUSION

All such techniques are a good tool for correlating resutlados, in order to obtain an improvement in moisture data to the PC. Note that in 28 days of curing, even with its high speed of reaction of some compounds of the CP, did not get their full hydration. The knowledge and mastery of the hydration process of the CP facilitates control of its mechanical strength, porosity and the interface between the cement and aggregates. This work contributes to the understanding of the process of hydration of Portland cement.

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