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Experimental Determination of Temperature Thresholds for Portland Cement Storage in Tropical Climates via Spectro-Mechanical Techniques

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Abstract: The deterioration of Portland cement after manufacture was investigated considering that storage conditions such as temperature and humidity are the main causes of deterioration. Infrared spectroscopy, fluorescence microscopy and mechanical performance, X-ray diffraction (XRD) and ultrasonic tests were used. In this sense, an experimental procedure was designed to evaluate the chemical behavior of the hydration reactions of cement stored in a set of samples under conditions (dry, ambient and humid) with different temperatures. The results of infrared spectroscopy revealed a similar behavior between the cement in humid conditions and the cement with water addition. In addition, a pronounced impact of humid conditions on the characteristics of Portland cement when compared to drier environments was also verified. In addition, the fluorescence microscopy experiments easily identified signs of deterioration of cement stored under temperature conditions with high humidity. Ultrasonic tests reveal that specimens prepared with cement stored at 30 °C have greater porosity when the cement is stored for 45 days. Similarly to the compressive strength results, the ultrasonic pulse test identified that cement stored at 10°C and 50°C presented better mechanical behavior when added to the mortar. Finally, to preserve the quality of Portland cement, hot or cold storage temperatures are promising to be applied as a replacement for ambient temperatures, where problems due to its deterioration in tropical climates can be mitigated.

Keywords: Portland cement; Deterioration; Infrared spectroscopy; Kinects; Fluorescence microscopy.

1. Introduction

Portland cement (also called hydraulic cement) is an important material widely employed in civilian constructions such as: bridges; viaducts; and buildings. Despite the Portland cement is prepared at elevated temperatures, this inorganic material is often stored and employed at ambient conditions, Where the degradation condition of reinforced concrete structures is associated with the condition of exposure to temperature and humidity [1–3]. This is especially challenging for the Portland cement used in a tropical environment, in which several cases of deteriorations of both cement, as well as the cement-based materials, such as concrete and mortars, and their corresponding constructions have been reported [4–9].

The biodeterioration of the Portland cement has been studied, and several types of colonization of microorganisms were reported [4,5, 9–16,17–21]. An example of bacteria that can be found in Portland cement is the *H. neapolitanus* [9]. Besides, examples of fungi that can be present in the cement are both *Aspergillus niger* and *Mycelia sterile* [22]. Accordingly, during the life cycle of these microorganisms, substances that are capable to react with the components of the cement are emitted. Examples of substances include: sulfuric acid [9]; gluconic acid and malic acid [22]. In addition, Magniont *et al.* [23] reported that when cementitious materials, such as the concrete of agro-industrial and agricultural structures are in contact with liquid manure the bacterium of the type *Escherichia coli* can proliferate into the cementitious material. Indeed, the presence of Gram-negative Coliforms, such as *Escherichia coli*, *Salmonella*, and *Listeria monocytogenes* in construction materials are often in domestic areas, mainly due to the human and animal microbiomes [24]. In particular, *Sohn et al.* [25] reported that bacterial pathogens, such as *Escherichia coli and Salmonella* are capable to exhibit fluorescence properties when excited under UV-light.

On the other hand, the storage and usage of Portland cement in construction materials, such as the mortars and concretes, are often performed at ambient conditions [26]. Because the humidity of the environment is often associated with the deterioration of Portland cement materials [27], this condition can enhance its deterioration, and, therefore, its durability of use can be reduced. Besides, the purity of the Portland cement can be associated with the quality of its corresponding construction material, and, therefore, studies to identify its early deterioration when anhydrous material is important. In a recent work, a chemical formalism was proposed to estimate and understand the degradation of Portland cement subjected to degradation conditions [28].

Despite several works reported aspects on the durability of Portland cement and cement-based materials [29–38], for example, in terms of its properties and recommendations for its usage, there is still a gap with regard to academic studies on the optimal conditions of storage of Portland cement and what actually happens in commercial establishments and constructions located in tropical environments. In this sense, the Portland cement industries often recommend storing the Portland cement in an ambient without humidity. This recommendation may not be easy to perform in tropical environments, where the early deterioration of Portland cement can occur with a significant frequency. In addition, although important advances on physical-chemical aspects (kinetic and thermodynamics) of the complex reactions involving the Portland cement, such as its hydration [39–46].

In this article the near-infrared spectroscopy, as well as the fluorescence microscopy and physico-chemical investigations are performed to advance insights into storage conditions to improve the durability of use of the Portland cement in tropical environments. In this sense, chemical behaviors of the cement stored by considering a set of conditions (dry, ambient, humid) with different temperatures are investigated by near-infrared spectroscopy. Subsequently, the parameters of time and mass variation of Portland cement samples stored in tropical environment with different temperatures, and by considering 15-day intervals for an elapsed time of 90 days are investigated from a physical-chemical perspective. Finally, fluorescence microscopy analyses are performed in the Portland cement samples stored under different conditions of temperature in a tropical environment, when the following ages of use are studied: (i) 0 day (in an anhydrous form, which is an age predicted by the manufacturer for the material to present high quality), (ii) 45 days (which is also an age predicted by the manufacturer for the material to present high quality), and (iii) 120 days (which is an advanced age, and the material is not suitable for use).

2. Experimental

2.1 Infrared spectroscopy experiments and PCA analyses

For the infrared spectroscopy experiments, 4.2g of Portland of the type II (ASTM C595[47]) cement was placed in beakers and stored in different conditions by varying humidity (10%, 23%, 36%, 43%, and 99%), as well as by varying the temperature (13.3ºC, 24.6ºC and 34.6ºC). Besides, in a few samples, water was added (for each 0.5 g of cement evaluated, 0.5 ml of distilled water was added). These wet cement samples were homogenized. In order to obtain the spectra in the near infrared region, a Fourier transform infrared spectrophotometer (FTIR) (Perkin Elmer® Frontier) was employed, by considering the diffuse reflectance accessory (NIRA). The spectra were acquired in the region of 2500-800 nm, resolution 2.5×10^6 nm (4 cm^{-1}) and 16 scans. The white used was the spectralon and the measurements were performed in triplicate in the glass vial with 0.5 g of the cement sample. During the analysis, the conditions were humidity 43% and temperature 23.4^0C. Further, an exploratory analysis of the investigated samples was carried out, using data from 50 spectra (different storage conditions, varying temperature and humidity and cement samples in contact with water) to qualitatively assess the possible formation of clusters, which would indicate or not the similarity or difference between the samples. In this sense, principal component analysis (PCA) was considered where the data was centered on the mean and full cross validation. All data were processed using the software The Unscrambler® X, version 10.2.

2.2 X-ray fluorescence spectrometry

Disk samples of the Portland cement of the type II were prepared, and a load of 150 kN over 1 min was applied. Subsequently, the samples were left in an oven drying process at 90ºC. The XRF analysis was performed by employing an XRF 1800 Shimadzu equipment.

2.3 Fluorescence microscopy

The set of samples of the Portland cement were left for elapsed times of: 0 day; 45 days, and 120 days at the following temperatures: 10ºC; 30ºC; and 50ºC. These stored samples of Portland cement, as well as a sample of the anhydrous cement were fixed between slides with adhesive tape. The sheets were placed on an Axion Vision microscope with clear field to obtain fluorescence images in the following regions of the visible: blue; green; and red, that were employed the wavelengths of 366 nm, 488 nm, and 546 nm, respectively.

2.4. X-Ray Diffraction (XRD)

X-ray diffraction assessment was performed in a Bruker D2 Phaser diffractometer with the following configuration: angular step = 0.02°; scan range = 5 – 70° (2θ); rotation speed = 15 rpm; scan speed = 1.2°/min; current = 10mA and tension = 30 kV. Highscore Plus software was employed for the qualitative analysis.

2.5 Compressive strength

The compressive strength test was performed on 40 cylindrical samples of mortar made with cement at different temperatures. The cement was initially stored at room temperature and the first 10 samples were fabricated at a storage temperature of 30°C with a wet cure time of 28 days. Then, the cement was stored at two more temperatures (10ºC and 50ºC) for a period of 45 days and used for the manufacture of mortar specimens to perform the compressive strength test at 28 days.

2.6 Ultrassonic measurements

In the ultrasonic measurements, 19 samples of mortar were analyzed for each temperature of cement storage in the period of 1 day and 45 days. A record of direct transmission responses to longitudinal and transverse ultrasonic waves was kept at a frequency of 500kHz and pulse amplitude of 500V, and the position of the transducers and their applied pressure were kept constant during the experiment.

3. Results and discussion

3.1 Infrared spectroscopy and the Portland cement deterioration aspects

In order to characterize differences between the anhydrous Portland cement and the corresponding early deteriorated material due to tropical conditions, infrared spectroscopy was employed. In this sense, Fig. 1 shows the general profile of the spectra by considering the near-infrared region of the Portland cement samples by considering two situations: (i) anhydrous cement and (ii) wet cement. Fig.2 shows cement samples stored at different humidity and temperature.

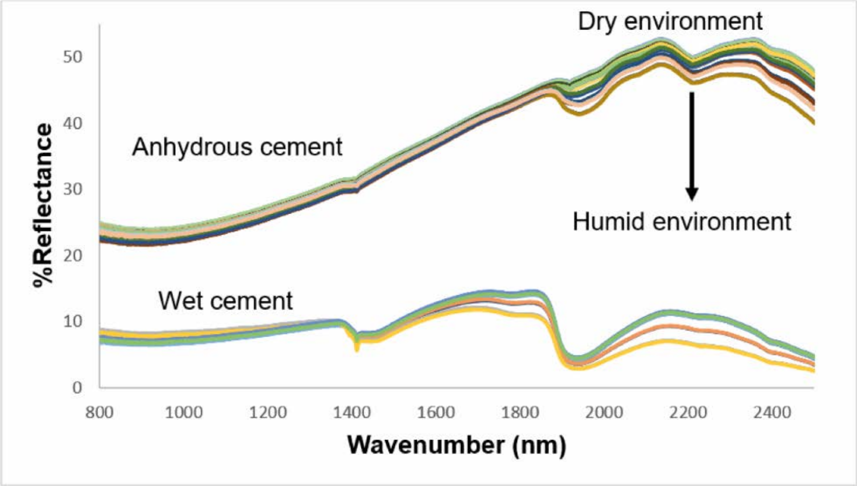

Figure 1. Near infrared spectra of the Portland cement samples stored in environments with different temperature and humidity.

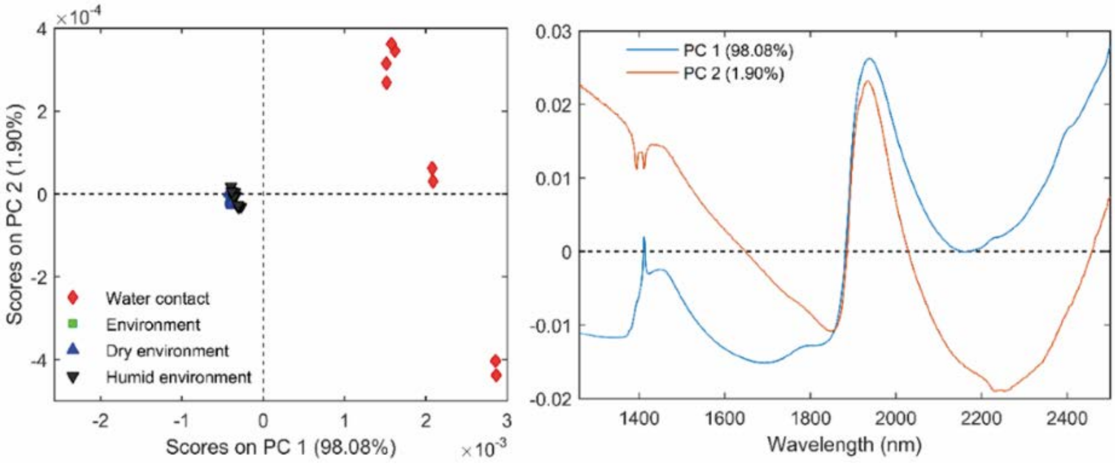

Figure 2. Scores (a) obtained by the PCA e loadings (b) from the spectral data of powdered Portland cement.

From Fig. 1, the signal on the main band appears 1937 nm, which is associated with the presence of water in the material, since it is indicative of the OH combination band of the water molecules. As expected, the drier the environment, the less pronounced this band is. Besides, from the infrared spectroscopy data, Principal Component Analysis (PCA) analysis was performed, as can be seen in Fig. 2.

From Fig. 2, it is verified that the two main components elucidated 99.98% of the total variation of the data (PC1 = 98.08% and PC2 = 1.90%). In PC1, two groups were formed where one group was composed of cement samples that had direct contact with water, and another group of the cement samples were stored in different environments. The samples that composed the second group of the PC1 did not have direct contact with water. Besides, the loading graph revealed that the band in 1937nm contributes more significantly to differentiate the samples on the PC1 curve.

Fig. 3 shows the PCA analysis of this set of NIRA spectra to evaluate the case that Portland cement interacts with the environment's water molecules.

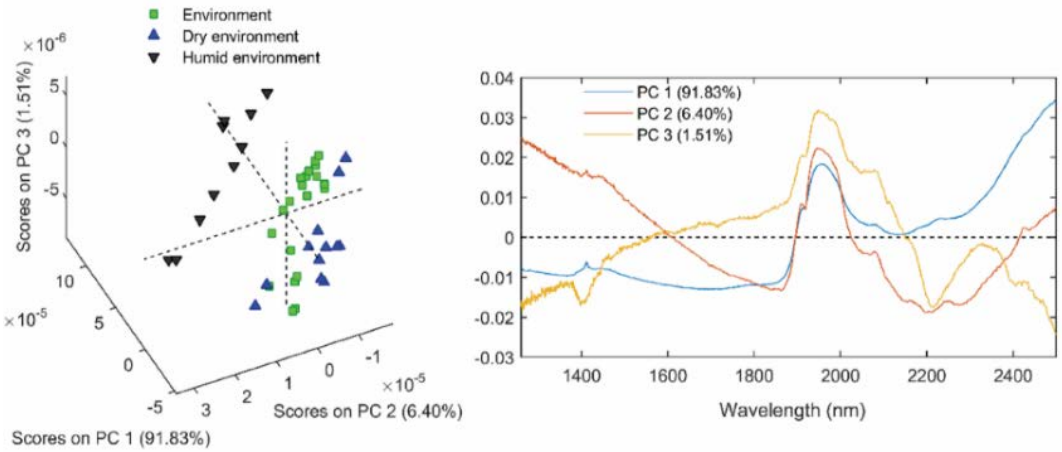

Figure 3. Scores (a) obtained by the PCA e loadings (b) from the spectral data of Portland cement stored in three conditions: environment; dry environment; and humid environment.

The PCA of the NIRA spectra associated with the Portland cement stored at different conditions (dry, ambient, and humid) indicated the formation of two groups, Figure 3. In this sense, the three main components elucidated 99.74% of the total variation of the infrared spectroscopic data (PC1 = 91.83%, PC2 = 6.40% and PC3 = 1.51%). On the one hand, the cement samples that were stored by employing a humid environment (humidity of 99%) and on the other hand, the samples that were stored in a dry environment (humidity ranging from 10% to 43%), regardless of the storage temperature.

In summary, the infrared spectroscopy revealed a similar behavior between the cement maintained at humid conditions, and the cement with added water. Besides, there is a large impact of the humid conditions on the characteristics of the Portland cement when compared with drier environments, even varying the temperature. In this sense, a tropical environment is now being evaluated, where the impact of different storage strategies on the physico-chemical properties of the processes associated with cement deterioration is evaluated.

3.2 Fluorescence microscopy and XRD structural characteristics

In order to identify microscopic signals of the early deterioration of the Portland cement stored in ambient conditions, a sample of the anhydrous material was evaluated by fluorescence microscopy, as can be seen in Fig. 4.

From Fig. 5, the reference sample of the cement exhibits, in a qualitative manner, a poor RGB fluorescence, i.e., the anhydrous Portland cement studied under UV lights was capable of emitting red, green, and blue lights. Besides, to evaluate the impact of the temperature on the deterioration of the Portland cement, a humid ambient condition and three temperatures were considered: (i) 10ºC; (ii) 30ºC; and (iii) 50ºC for an elapsed time of 45 days, that is its half-life. After this period, the Portland cement stored at ambient conditions exhibited stone aspects whereas in its anhydrous form this material is a fine powder. When the cement was stored at both 10ºC and 50ºC, the fine powder characteristic of the anhydrous material was, seemingly, conserved. In this sense, Fig. 5 shows, in an illustrative manner, all cement samples studied in this article after a storage time of 45 days.

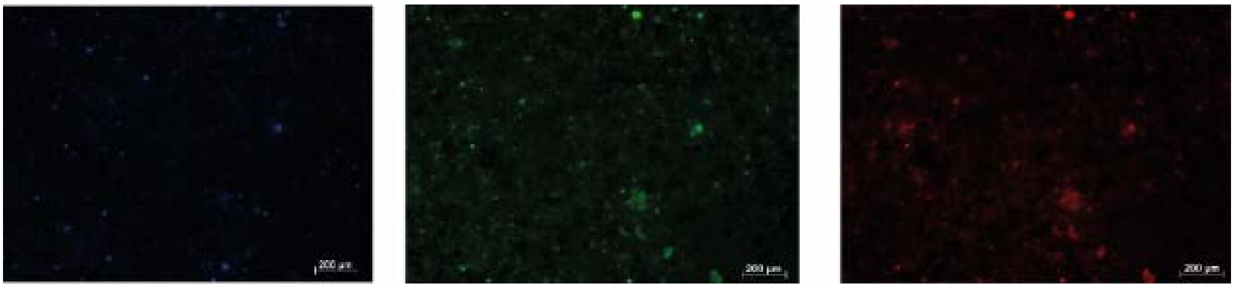

Figure 4. Fluorescence microscopy data of the Portland cement anhydrous of type II under UV-light with the following wavelengths: 366 nm (for blue emission), 488 nm (for green emission), and 546 nm (for red emission). All fluorescence microscopy experiments were considered with a resolution of 200 µm.

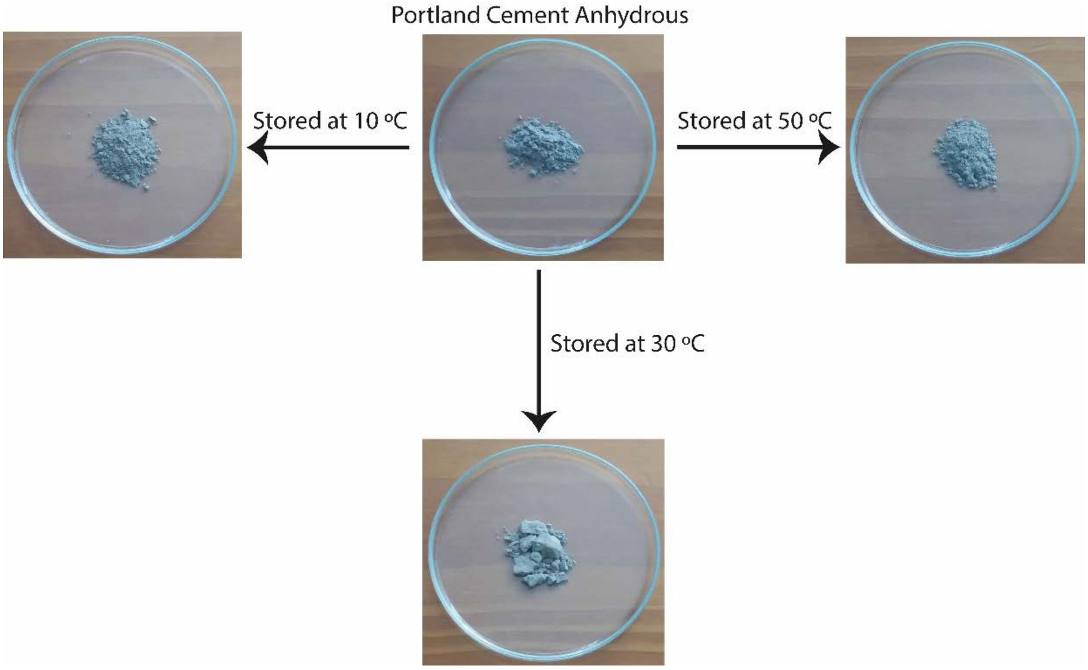

Figure 5. Images of the anhydrous Portland cement, as well as their samples stored for 45 days at 10ºC, 30ºC, and 50ºC in a tropical environment.

Fig. 5 shows that, as expected, the ambient condition strongly affects the macrostructural aspects of Portland cement. Besides, Fig. 7 shows the images of fluorescence microscopy experiments for these samples stored at 10º C, 30º C, and 50 ºC temperatures, considering the half-life of Portland cement (45 days).

From Fig. 6, the Portland cement stored at 50ºC exhibited poor fluorescence in the three visible regions investigated: blue, green, and red. A similar poor RGB fluorescence was verified when the cement was stored at 10ºC. A substantial RGB fluorescence was verified when the cement was stored at ambient conditions (30°C). Our hypothesis is that the storage temperatures ranging from 10°C to 50°C are not capable of significantly affecting the chemical and physical aspects of the main inorganic compounds that compose the Portland cement (C_3S, i.e. $3CaO.SiO_2$), (C_2S, i.e. $2CaO.SiO_2$), (C_2A, i.e. $3CaO.Al_2O_3$), and (C_4AF, i.e. $4CaO.Al_2O_3.Fe_2O_3$). This is because the energetic stability of the ionic bonding nature of the main components of the Portland cement is capable of supporting even more variations of temperatures.

In particular, Ortega-Morales *et al.* [4] reported an interesting and extensive meta-analysis of the aspects associated with the biodeterioration of a set of stone buildings located in Argentina, Belize, Brazil, Cambodia, Laos, and Mexico, where two tropical and subtropical conditions were investigated: (i) polluted and (ii) unpolluted ambientes. The authors identified several types of cyanobacterial systems and evaluated the diversity of these systems for both cases. The results of Ortega-Morales *et al.* [4] revealed that for the polluted tropical environments, the diversity of cyanobacteria was lower when compared to unpolluted environments. However, pollution-resistant cyanobacteria, such as the *Chroococcus*, were also detected [4]. Besides, the authors identified the genus *Gloeocapsa* for most of the cement-based samples, which, seemingly, is responsible for the black coloration that a part of the building in these places exhibits over time [4]. Therefore, in the present article, the results of fluorescence microscopy of cement samples stored at different temperatures in a tropical climate, seemingly, is due to the presence of fluorescent microorganisms that are probably affecting the durability of the use of Portland cement.

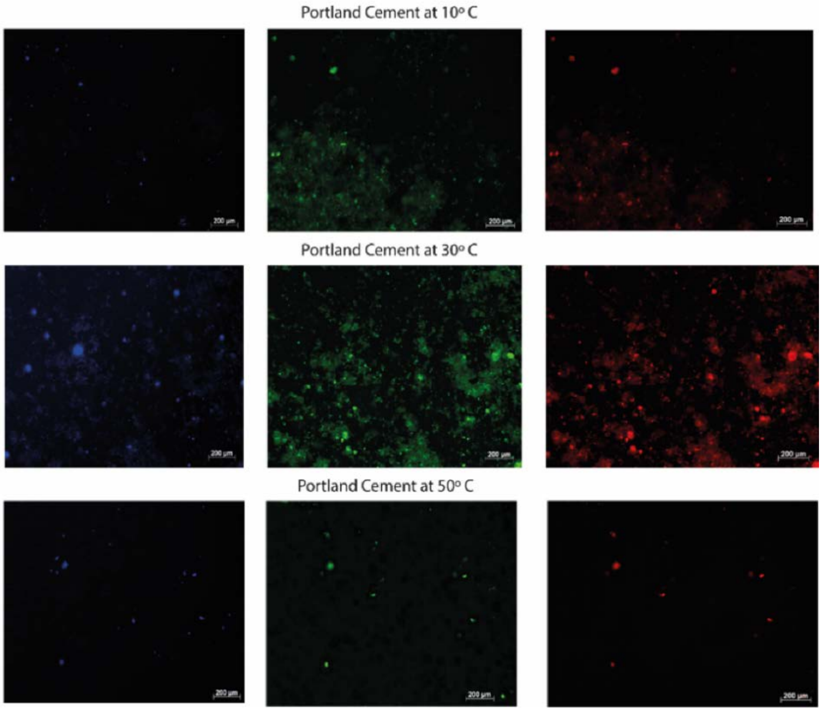

Figure 6. Fluorescence microscopy data of the Portland cement samples stored at 45 days under UV-light with the following wavelengths: 366 nm (for blue emission), 488 nm (for green emission), and 546 nm (for red emission). All fluorescence microscopy experiments were considered with a resolution of 200 µm,

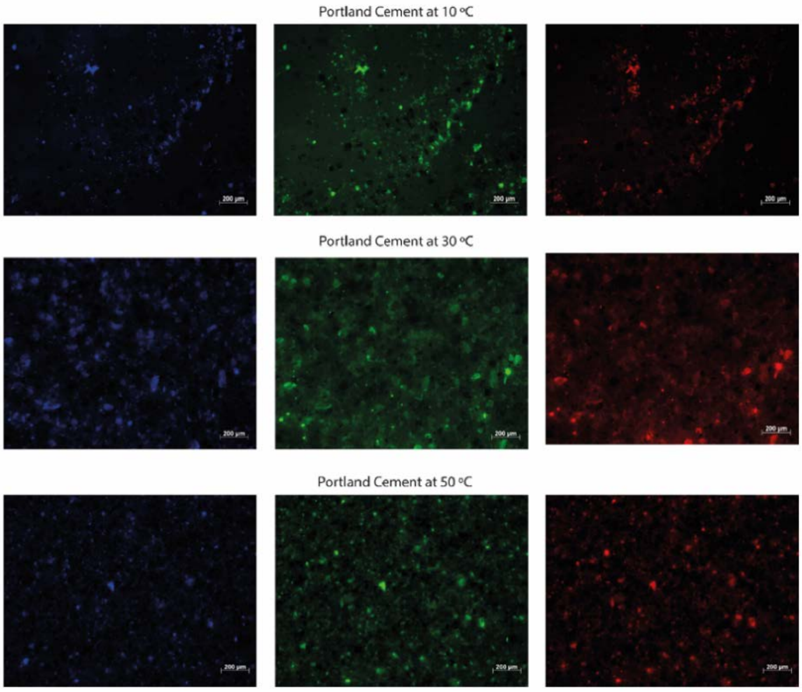

Figure 7. Fluorescence microscopy data of the Portland cement samples stored at 120 days under UV light with the following wavelengths: 366 nm (for blue emission), 488 nm (for green emission), and 546 nm (for red emission). All fluorescence microscopy experiments were considered with a resolution of 200 µm

Further, by considering a period longer than the shelf life of Portland cement (120 days), fluorescence microscopy experiments were also performed for samples stored in tropical environments at different temperatures (10°C, 30°C, and 50°C), as can be seen in Fig. 6(b)

Fig. 6(b) shows that, as expected, due to the advanced age of the stored cement samples, fluorescence microscopy was also able to easily identify a more advanced deterioration (for all temperatures considered) for all samples of Portland cement.

In summary, fluorescence microscopy can be employed to easily identify signals of Portland cement deterioration in different ages (anhydrous material, half-life material, and old-age material) for the cement stored in tropical climates. In particular, because the ambient condition of storage led to an early deterioration of cement, alternative storage temperatures, such as hot or cold temperatures instead of ambient temperatures, should be employed in order to preserve the quality of the original material, at least during its shelf life. By considering these promising storage temperatures, the quality of the original Portland cement can be improved in tropical environments, where problems due to its early deterioration in tropical climate can be mitigated.

On the other hand, by emploing the X-ray diffraction technique, it was possible to identify the mineralogical phases of the cement samples after 45 days of storage. This analysis aimed to observe the appearance of hydrated products, which could indicate cement hydration due to storage conditions. Figure 8 shows expected cement constituents such as gypsum ($CaSO_4$), larnite (Ca_2SiO_4), hatruite (Ca_3SiO_5), calcite ($CaCO_3$) and quartz (SiO_2). It was not possible to verify the presence of portlandite ($Ca(OH)_2$) because there is peak overlapping at 18° and 34° with usual phases found in cement, such as larnite and brownmillerite ($Fe_{1.33}Al_{0.67}Ca_2O_5$).

Nevertheless, the sample storage at 30°C presented some hydration evidence. First, there was a small peak at 8.74° (2θ) owing to the presence of ettringite ($Ca_6Al_2(SO_4)_3(OH)_{12}.25H_2O$), which can be better analyzed by the ampliation. Moreover, the peak of gypsum at 11.62° (2θ) is lower for the cement stored at 30°C, which suggests that part of the gypsum could be consumed to produce ettringite.

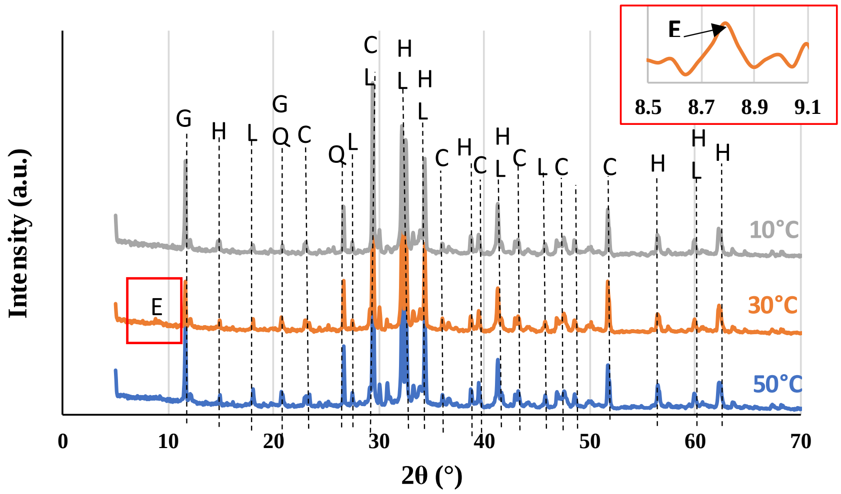

Figure 8. X-ray diffractograms for the cement samples stored at 10, 30, and 50°C. The phases identified were: ettringite (E), gypsum (G), hatruite (H), larnite (L), quartz (Q) and calcite (C).

3.3 Mechanical investigations

Table 1 shows the compressive strength results of mortars made with cement stored at three different temperatures. The results show a reduction in compressive strength after 45 days of storage compared to cement used soon after receipt. The material stored at room temperature showed the lowest values, with a 38% reduction in the compressive strength of the mortars. The storage of the material at 50°C showed greater conservation of the cement characteristics when compared with the other temperatures considered. Cement stored at high temperatures achieved a reduction of only 11% when incorporated into cement-based material.

In addition**,** the ultrasonic pulse test is a nondestructive test and a qualitative analysis that, through the propagation speed of longitudinal waves, provides information regarding the material's homogeneity and porosity. The evolution of concrete's compressive strength is related to changes in the material's density. Table 1 presents the results of the ultrasonic pulse test in microseconds and meters per second.

From Table 2, evaluating the behavior of specimens prepared with cement stored at 30°C reveals greater porosity when the cement is stored for 45 days, and these results were also lower than at other temperatures. Analogously to the compressive strength results, the ultrasonic pulse test identified that the cement stored at 10°C and 50°C presented better mechanical behavior when added to the mortar.

Table 1. Compressive strength results of concrete mixes from three storage temperature

Storage time/ Temperature	1 day / 30 ºC	45 days/ 10 ºC	45 days/ 30 ºC	45 days /50 ºC
Compressive strength average (Mpa)	27.12	23.93	16.80	24.24
Standard deviation (Mpa)	1.11	1.08	1.76	2.55
Variation coeficiente (%)	4.10	4.51	10.49	10.50

Table 2. Ultrasonic testing data of cement-based mortars.

Sample	1 day (30°C)		45 days (10°C)		45 days (30°C)		45 days (50°C)	
	µs	m/s	µs	m/s	µs	m/s	µs	m/s
1	24.40	4,098.00	24.50	4,082.00	24.70	4,049.00	24.40	4,098.00
2	24.10	4,149.00	23.90	4,184.00	24.90	4,016.00	24.20	4,132.00
3	24.00	4,167.00	24.30	4,115.00	25.00	4,000.00	24.20	4,132.00
4	24.30	4,115.00	24.40	4,098.00	25.10	3,984.00	24.60	4,065.00
5	24.60	4,065.00	24.40	4,098.00	24.40	4,098.00	24.20	4,132.00
6	24.00	4,167.00	24.20	4,132.00	24.80	4,032.00	24.40	4,098.00
7	24.40	4,098.00	24.70	4,049.00	25.10	3,984.00	24.40	4,098.00
8	24.40	4,098.00	24.50	4,082.00	24.50	4,082.00	24.90	4,016.00
9	24.20	4,132.00	24.50	4,082.00	24.90	4,016.00	23.90	4,184.00
10	24.00	4,167.00	24.10	4,149.00	24.50	4,082.00	24.50	4,082.00
11	24.10	4,149.00	23.80	4,202.00	25.00	4,000.00	24.40	4,098.00
12	23.90	4,184.00	24.40	4,098.00	24.60	4,065.00	24.30	4,115.00
13	24.40	4,098.00	24.30	4,115.00	24.10	4,149.00	24.50	4,082.00
14	23.90	4,184.00	24.50	4,082.00	24.80	4,032.00	23.80	4,202.00
15	23.90	4,184.00	24.40	4,098.00	24.40	4,098.00	24.40	4,098.00
16	24.40	4,098.00	24.80	4,032.00	24.80	4,032.00	24.20	4,132.00
17	24.00	4,167.00	24.40	4,098.00	25.40	3,937.00	24.80	4,032.00
18	24.40	4,098.00	24.40	4,098.00	25.40	3,937.00	24.40	4,098.00
19	23.90	4,184.00	24.40	4,098.00	25.80	4,031.00	26.50	3,962.00
Average	24.17	4,136.95	24.36	4,104.84	24.85	4,032.84	24.47	4,097.68
Standard Deviation	0.23	39.12	0.24	40.44	0.41	54.76	0.56	54.92

4. Conclusions

The conclusions of this article are presented as follows:

1) The infrared spectroscopy revealed a similar behavior between the cement maintained at humid conditions, and the cement with added water. In addition, a pronounced impact of the humid conditions on the characteristics of the Portland cement was verified when compared with drier environments.

2) The fluorescence microscopy experiments easily identified signals of Portland cement deteriorations in different ages (anhydrous material, half-life material, and old-age material) for the material stored in a tropical environment.

3) From XRD, it was possible to identify the mineralogical phases of the cement samples after 45 days of storage. This analysis aimed to observe the appearance of hydrated products, which could indicate cement hydration due to storage conditions, such as gypsum ($CaSO_4$), larnite (Ca_2SiO_4), hatruite (Ca_3SiO_5), calcite ($CaCO_3$) and quartz (SiO_2);

4) Ultrasonic testing reveals that the specimens prepared with cement stored at 30°C have greater porosity when the cement is stored for 45 days, and these results were also lower than at other temperatures. Analogously to the compressive strength results, the ultrasonic pulse test identified that the cement stored at 10°C and 50°C presented better mechanical behavior when added to the mortar.

5) Finally, this article points in the direction of new studies, in which optimal storage conditions for Portland cement can be developed, considering even more complex situations, such as the wide range of climates worldwide.

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