EUROPEAN ACADEMIC RESEARCH
Vol. VIII, Issue 10/ January 2021

Impact Factor: 3.4546 (UIF)
DRJI Value: 5.9 (B+)

ISSN 2286-4822
www.euacademic.org

Normative Verification of Three Types of Portland Cements Commercialized in Manaus/AM/Brazil

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Abstract

Ensuring the quality of products offered to consumers is one of the fundamental roles of public authorities, but when demand becomes greater than its operational capacity, it tends to serve the industry. With this, the importance of a study that validates normative prerequisites for elementary material for civil construction emerges and which, if not met, endanger the integrity of consumers. Therefore, the present research seeks to evaluate the normative prerequisites for Portland cement marketed in the city of Manaus, capital of the State of Amazonas in Brazil. The experimental program aims to attend from a brief market survey to check which types and manufacturers of cement *are most frequently sold, to the verification of resistance in comparison with what the standard recommends, including the physical and rheological characterization of the most used material in construction in general. Therefore, tests will be carried out to determine the fineness and axial compression strength measured at 14 and 28 days of age. With the results found, we intend to compare them with the values recommended by Norma and check if they fully meet, if they are in the variation range or if they fail.*

Keywords: Normative, Portland cements, Amazonas, fineness, axial compression.

1. INTRODUCTION

Civil construction over the last decades has had a surprising evolution and is therefore characterized by the Civil Construction Industry, as it develops a productive chain of materials separated into groups and subgroups, moving direct and indirect jobs.

Inserted in this production chain is cement as raw material for mortar, concrete made for foundations, structures, finishes and regularization. Cement is used in environments with the most diverse characteristics of aggressiveness, such as humid soils, saline environments, carbonation, acid rain in large cities, attacks on sulfates and others are some of these degrading agents that attack the cement material, compromising its structure resulting in decreased durability.

Portland cement is the name given to this material widely used in civil construction as only cement. It is a fine powder that has agglomerating, binding or binding properties, having its chemical activation in the presence of water and that over the days undergoes a process of hardening and resistance gain (Portland, 2003).

Cement is acquired from the clinker grinding with other materials, called the generic form of additions, Figure 2. The clinker is manufactured mainly from limestone and clay, which are crushed, ground and mixed in defined quantities. It is a material that is brought to high temperatures in special ovens. The clinker production procedure is, in a simplified way, a combination of exploration and processing of mineral substances, transformed through heat (ABDI, 2012).

The present work aims to determine the fineness and resistance of axial compression measured at 14 and 28 days of age. With the results found, we intend to compare them with the values determined by the Standard and check if they fully meet, if they are within the variation range or if they fail.

2. THEORETICAL REVIEW

Portland cement is the product of burning a well-proportioned mixture of raw materials containing four main oxides - CaO, SiO_2, Al_2O_3 and FeO_3 - which produces clinker, one of the two basic ingredients required to make Portland cement. The other is calcium sulfate in the form of plaster ($CaSO_4.2HO$) or hemidrate ($CaSO_4.1/2H_2O$) or calcium anhydrite or sulfate ($CaSO_4$), or a mixture of two or three of them (Bye, 1993).

The chemical composition of raw materials in terms of CaO, SiO_2 and Al_2O_3 is adjusted in such a way that the mixture falls into the composition triangle limited by C_3S, C_2S, and C_3A in the ternary phase diagram SiO_2 - CaO - Al_2O_3, Figure 1, because it is only in this area that these three phases can exist simultaneously (Philips and Muan, 1959).

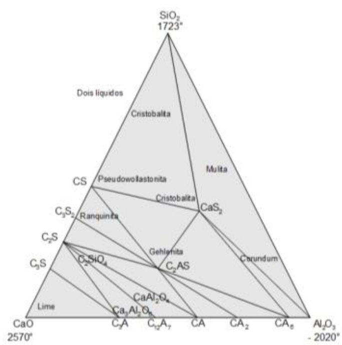

Figure 1: Triangle of cement composition
Source: Philips and Muan (1959)

Portland cement clinker is the end product of a highly complex pyroprocessing technology that transforms raw materials into phases of calcium silicates and calcium iron aluminate. Also relevant is the nature of the fuel used in the cement kiln, or to be more precise, the nature of the impurities present in it. In particular, the content of sulfur and ash can play a critical role during the formation of clinker: ash as a source of impurities and sulfur in the formation of volatile alkaline sulphates.

From a composition point of view, Portland cement clinker comes out of the rotary kiln as a mixture of two phases of well crystallized silicates, C_3S e C_2S, and an interstitial phase composed of C_3A e C_4AF, more or less crystallized, Figure 2, and a few "impurities" such as periclase (MgO), hardened calcined calcium oxide (CaO) and alkaline sulfates.

Thus, in order to stabilize the silicates in their respective forms, the clinker must be cooled quickly after passing through the cliqurization zone in the oven. Rapid cooling solidifies the different phases in their forms at high temperature, not allowing them to follow natural phase transformation, which would have occurred under very slow cooling. The microstructure of the clinker after this cooling significantly influences the hydraulic properties: two Portland cements can have exactly the same chemical composition, but different microstructures and hydraulic behaviors.

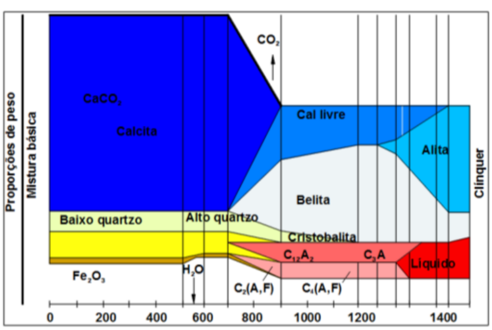

Figure 2 - Phases that happen with the heating of the clinker raw material

Fonte: Adapted from Isaia (2005).

Clinker is a complex material with multiple phases, whose characteristics and properties depend not only on the composition of the ground raw material, but also on pyrotechnology that transforms it into clinker. Two clickers can have exactly the same chemical composition, but very different microstructural characteristics. They can have exactly the same phase composition and still have different hydraulic properties because, for example, after rapid cooling, the average sizes of the alita crystals can be very different from one clinker to another, which is a characteristic that influences strongly reactivity and resistance after grinding (Shachelford, 2008).

To produce Portland cement, the clinker has to be mixed with an optimum amount of calcium sulfate. The role of this sulfate is to control the initial hydration of Portland cement. In its absence, the pure clinker undergoes an instant catch that causes an irreversible hardening of the mixture. Considering that C_3A reacts very quickly with water to form hydrated calcium aluminate, if some calcium sulfate is mixed with the clinker, C_3A reacts with it and water to form a more or less impermeable etringed envelope that inhibits the reaction of C_3A with water (Collepardi et. al., 1979); (Regourd, 1978); Mindess and Young, 1981).

In fact, there are five common types of Portland cement, as shown in Table 1. They differ in the relative concentrations of four minerals that contain calcium. The matrix is formed by adding water to the appropriate cement powder. The particle sizes for cement powders are relatively small compared to the finer aggregates. Variation in the size of the cement particles can strongly affect the rate at which the cement hydrates. It is this hydration reaction that hardens the cement and produces the chemical bond between the matrix and the aggregated particles.

By examining the complex compositions of Portland cement, the chemistry of the hydration process is equally complex. The main hydration reactions and the associated final products are shown in Table 2.

Table 1: Portland cement compositions

Composition (% p)						
Type ASTM	Features	C_3S	C_2S	C_3A	C_4AF	Others
I	Standard	44	27	11	8	9
II	Reduced heat of hydration and greater resistance to sulphates	45	31	5	13	7
III	High initial resistance (associated with high hydration heat)	53	19	11	9	8
IV	Low heat of hydration (less than type II and especially good for solid structures)	28	49	4	12	7
V	Sulphate resistance (better than type II and especially good for marine structures.	38	43	4	9	6

Source: (Shachelford, 2008).

Table 2: Main hydration reactions of Portland cement

Hydration reactions of Portland cement	
(1)	$2C_3S + 6H \longrightarrow 3Ch + C_3S_2H_3$ (tobermorita)
(2)	$2C_3S + 4H \longrightarrow Ch + C_3S_2H_{12}$
(3)	$C_3S + 10H + CsH_2 \longrightarrow C_3ACSH_{12}$ (hydrated calcium aluminum monosulfate)
(4)	$C_3S + 12H + Ch \longrightarrow C_6AChH_{12}$ (hydrated tetracalcium aluminate)
(5)	$C_4AF + 10H + 2Ch \longrightarrow C_6AFH_{12}$ (hydrated calcium aluminum)

Source: (Shachelford, 2008).

1. 2 Compressive strength of cement

To assess the strength of a concrete to compression, it is necessary to perform a number of tests on specimens. The resistance values provided by the different specimens are more or less dispersed, varying from one mixture to another and also according to the rigor with which the concrete is made (Carvalho and Figueiredo Filho, 2019).

Cement is produced by grinding and intimately mixing clay and minerals containing lime in appropriate proportions, and then heating the mixture in a rotary kiln to approximately 1400°C (2550°F); this process, sometimes called calcination, produces physical and chemical changes in raw materials. The resulting product, "clinker", is then ground into a very fine powder, to which a small amount of plaster is added ($CaSO_4$ - $2H_2O$) to slow the setting process, then forming the Portland cement. The properties of Portland cement, including its setting time and final strength, also largely depend on its composition (Callister Jr., 2020).

Several different constituents are found in Portland cement, the main ones being tricalcium silicate ($3CaO$-SiO_2) and dicalcium silicate ($2CaO$-SiO_2). The setting and hardening of this material results from relatively complicated hydration reactions that occur between the various constituents of the cement and the water that is added. For example, a hydration reaction involving dicalcium silicate is as follows:

$$2CaO\text{-}SiO_2 + xH_2O \quad = \quad 2CaO\text{-}SiO_2 - xH_2O$$

where x is the variable and depends on the amount of water available. These hydrated products are in the form of crystalline substances or complex gels that form the cement bonds. Hydration reactions begin immediately after adding water to the cement. These reactions first manifest themselves as the handle, which takes place shortly after mixing, usually within a few hours. The hardening of the dough continues as a result of additional hydration, in a relatively slow process that can continue for periods as long as several years. It must be emphasized that the process by which cement hardens is not a drying process, but rather a hydration process, where water effectively participates in a chemical reaction (Callister Jr., 2020).

Portland cement is said to be hydraulic because its hardness develops through chemical reactions with water. It is mainly used in mortar and concrete to agglutinate aggregates of inert particles (sand, pebbles and gravel) into a cohesive mass; these products are considered composite materials.

1.3 Microestrutura da Pasta de Cimento Hidratada

Anhydrous cement is a gray powder composed of angular particles with dimensions that normally vary from 1 µm to 50 µm. It is produced by grinding a clinker with a small amount of calcium sulfate, the clinker being a heterogeneous mixture of various compounds produced by reactions at high temperature, between calcium oxide and silica, alumina and iron oxide. The chemical composition of the main components of the clinker corresponds approximately to C_3S, C_2S, C_3A, and C_4AF. In ordinary Portland cement, their respective quantities normally vary between 45% and 60%, 15% and 30%, 6% and 12%, and 6% and 8% (Mehta and Monteiro, 2008).

When the cement is dispersed in water, calcium sulphate and the calcium compounds formed at high temperature start to come into solution, and the liquid phase quickly becomes saturated with various species of ions. As a result of the interaction between calcium, sulfate, aluminate and hydroxyl ions, and within a few minutes of cement hydration, acicular crystals of hydrated calcium trisulfoaluminate, known as etringite, begin to appear. A few hours later, large prismatic crystals of calcium hydroxide and small fibrous crystals of hydrated calcium silicate begin to fill the empty spaces previously occupied by water and dissolving cement particles. After a few days, depending on the alumina-sulfate ratio of Portland cement, the etringite may become unstable and decompose to form the hydrated monosulfoaluminate, which is in the form of a hexagonal plate. The hexagonal plate morphology is also a characteristic of hydrated calcium aluminates that are formed in hydrated Portland cement pastes containing low sulfate or high C_3A content.

A model of the essential phases present in the microstructure of a well-hydrated cement paste is shown in Figure 3. It can be noted that the various phases are neither uniformly distributed nor uniform in size and morphology. In solids, microstructural heterogeneity can have negative effects on strength and other mechanical properties, because these properties are controlled by the microstructure that generates the weakest link, and not by the average microstructure. Thus, in addition to the evolution of the microstructure as a result of chemical changes, which occur after the cement comes into contact with water, attention must be paid to certain rheological properties of the cement paste in the fresh state, which also influence the microstructure of the cement paste. hardened cement. Anhydrous cement particles tend to attract and form flakes, which trap large amounts of water in the mixture. Obviously, local variations in the water / cement ratio would be the main sources of evolution of heterogeneity in the microstructure. In a highly flocculated cement paste system, not only the size and shape of the pores, but also the crystalline hydration products should be different when compared to a well dispersed system (Mehta and Monteiro, 2008).

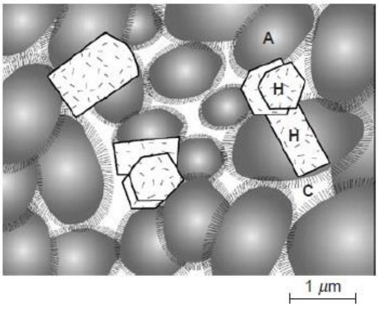

Figure 3: Diagrammatic representation of well-hydrated cement
Source: (Mehta and Monteiro, 2008).

3. MATERIALS AND METHODS

To achieve the objectives, the experimental program was designed to verify the conditions imposed by the Brazilian standard on cement. According to the established objectives, the research was divided into three stages.

3.1 First step: Field data collection

In this stage, it was verified which types of cement are sold in Manaus/AM, in the main building materials stores. In this stage, cement suppliers and their compositions will be recognized. Data collection will be through observation during visits to building materials stores distributed in the most diverse areas of the city. The stores of greatest significance in the supply of cement by area of the city were surveyed and, based on these results, verify the three largest types of cement sold for use in civil construction.

With the results of this first stage, the results were analyzed, the information was homogenized, and the three most commercialized cements were extracted with information from the manufacturer, type and class. After this survey, the cements were acquired and stored at the Laboratory of Construction Materials and Material Resistance of the Federal Institute of Education, Science and Technology of Amazonas - IFAM.

3.2 Second Stage: Characterizations of cements

This stage comprised the second and third objectives of the research, in which the basics of the studied cements were carried out. The tests in question were carried out according to the current Brazilian standards presented in Table 3.

Table 3: Determination of standards

STANDARD	DESCRIPTION	ANO
NBR 11579	Portland cement - Determination of fineness through a 75 micrometer sieve (N°. 200).	2012
NBR 16605	Portland cement and other powder materials - Determination of specific gravity.	2017

Source: Authors

3.3 Third Stage: Mechanical characterizations

In the third stage, the verification imposed by the standard for resistance to axial compression will be performed. For this, cylindrical specimens of dimensions 5 x 10 cm were molded, kept in a humid chamber and until broken at the ages of 14 and 28 days. The equipment used to carry out the mechanical tests was a universal electromechanical testing machine UMC 60 tons with the aid of the pavitest global-UMC software, belonging to the Laboratory of

Construction Materials and Strength of Materials, from the Federal Institute of Education, Science and Technology of the Amazonas - IFAM.

3.4 Test script

The mixture was made in a manual mortar, as shown in Figure 4, for three specimens in the proportion of 312 ± 0.2 g of cement, four proportions of normal sand, being 234 ± 0.2 g of coarse sand (2, 4 to 1.2 mm), 234 ± 0.2 g of coarse medium sand (1.2 to 0.6), 234 ± 0.2 g of fine medium sand (0.6 to 0.3), 234 ± 0.2 g of fine sand (0.3 to 0.15) and 300 ± 0.2 g of water.

(a) (b)

Figure 4: Preparation of materials

Source: Tests at the IFAM Construction Materials Laboratory

After mixing the specimens were molded in cylinders of dimensions 5 x 10 cm filled in four almost equal layers, each layer receiving 30 strokes evenly distributed.

After demoulding the bodies were broken at 14 and 28 days of cure, the tolerances of rupture were ± 3 hours and 4 hours, respectively, with axial compression load to the specimen at the speed of (0.25 ± 0.05) MPa / s and no adjustment in the machine when the sample at the moment of deformation when approaching its rupture Figure 5 (a) and (b).

(a)

(b)

Figure 5: Preparation and disruption of the specimens

Source: Tests at the IFAM Construction Materials Laboratory

4 RESULTS AND DISCUSSIONS

Table 4 indicates the fineness of the study cements, the results obtained are the values found in a single determination. The test defined the fineness index and demonstrated that the samples were satisfactory for the three types of cement, in compliance with the provisions of NBR 11579/2013 and consolidating the concepts related to the fineness of cement.

Table 4: Cement fineness

FINANCE OF CEMENTS	
Cements	**Fineness (%)**
MIZU CP II-Z-RS	1,75
FORTE CP II-Z-RS	1,86
POTY CP II-F-32	1,22
Standard deviation	0,03
Coefficient of variation (%)	2,11

Source: The authors

The granulometry of the cement affects the speed of the hydration reaction, as this can speed up the reaction if a cement of less granulometry is used, due to the fact that the smaller the area of the cement grain, the faster its reaction with water.

The fineness parameters presented in Table 4 obtained in the tests of agro-ager fineness tests show the profile of the discrete particle size distribution of the three types of agglomerates. Its performance as a nucleating agent for hydrated faces depends on the size of the cement particles. The particles must necessarily be equal in size or smaller than the particles (Lawrence, 2003).

According to Paula (2006), the fineness and the specific area of the binders are important requirements with regard to their ability to agglomerate. When in an aqueous medium the surface area of the binders is important in the reaction rate of hydrated products, and a consequent increase in the speed of gain of mechanical resistance. High surface area particles tend to agglomerate even in the dry state, requiring high activation energies, decreasing the efficiency of the process, requiring dispersive additives, directly influencing the workability of the paste and the exudation of water.

Table 5 shows the results of rupture stresses at 14 days for the three studied cements. Poty cement presented the best resistance results, the minimum, maximum and average resistance values showed the best results, however the highest values of the standard 4.17 and coefficient of variation 18,10. Meanwhile, the Mizu cement that showed the lowest strength values showed the best values of Standard Deviation, and a variation coefficient of 1.53 and 7.69, respectively.

Table 5: Axial rupture stress at 14 days

AXIAL BREAKING TENSION (MPa) - 14 DAYS			
MIZU CP II-Z-RS	MIZU CP II-Z-RS	FORTE CP II-Z-RS	POTY CP II-F-32
CP- 1	20,02	21,22	24,82
CP- 2	20,73	21,02	24,22
CP- 3	21,53	22,10	24,82
CP- 4	19,72	19,22	19,53
CP- 5	19,03	20,42	22,22
CP- 6	18,03	22,62	22,67
Minimum	18,03	19,22	19,53
Maximum	21,53	22,62	24,82
Average	19,84	21,10	23,05
D.P	1,53	1,47	4,17
C.V. (%)	7,69	6,95	18,10

Source: The authors

The paste is much more unstable, even if the production procedures of the samples are the same, the same during the hydration process retract during the hydration and drying process and expanding when moistened, allowing for increased porosity and resistance to rupture of the samples. The cement can still release heat during its hydration and induce cracking problems, the heat of hydration being one of the reasons for the difference in strength of the three types of cement studied. In addition, the creep phenomenon, that is, the effect of slow deformation suffered by the samples, comes from the viscoelastic properties of the cement paste, which are much more sensitive to this phenomenon than the sand involved in the mixture.

The finer the cements, the greater the reactive capacity of the binder, in addition to acting as a filler effect, decreasing the void volume, consequently decreasing porosity, reducing permeability and improving mechanical resistance and durability, and consequently promoting permanent effects on the material (Mehta and Monteiro, 2008); (Macedo, 2009).

We observed that the Poty cement among the studied cements was the one with the least fineness and, consequently, the greatest mechanical resistance. The increase in the final resistance and impermeability due to the refinement of the pores, the resistance to attack by sulfate, the minimization of the alkali-aggregate reaction and the corrosion of the reinforcement. The improvement of these properties directly reflects the increase in the durability of materials produced with Portland cement.

In Figure 6, the experimental results were grouped in pairs in order to allow a comparison between all the resistances at 14 days, all the binders presented resistance between 18.03 MPa and 24.83 MPa, we also realized that despite the cements being produced by different manufacturers the resistances are relatively close, such similarity occurs, mainly, when we make comparison with the samples tested by the same manufacturer.

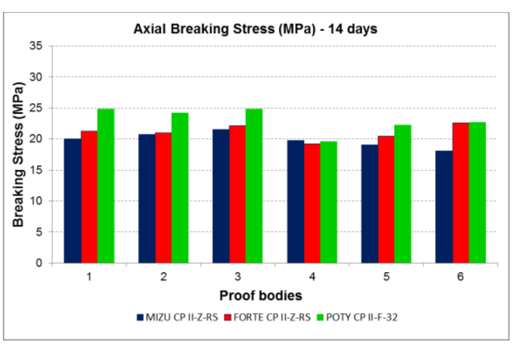

Figure 6: Grouped resistance results

Source: The authors

The maximum, minimum, average, standard deviation and variation coefficient values obtained for the rupture stresses of the three types of cement at 28 days are shown in Table 6. Of the three studied cements, Poty presented the best strengths, also presented, the best values of average resistance, standard deviation and coefficient of variation, 33.37 MPa, 0.67 and 2.00%, respectively.

The work carried out allowed us to identify not only the mechanical behavior of the cement samples, but also to observe that the Portland cement paste is much more unstable, shrinking during the hydration and drying process and expanding when moistened. The cement can still release heat during its hydration and induce cracking problems in pieces of large volume of concrete.

The relationship between the fineness and the reactivity of Portland cements has been shown by several studies, which, by increasing the fineness, consequently increases the potential reaction area of the material. Table 6 shows the variations found in the resistance of the tested cements, with the same amount of kneading water to obtain the same workability of the cement mortars and in the pozzolanic activity indexes, however we observed that the measure that its fineness changed, also modified the breaking strengths.

Table 6: Axial rupture stress at 28 days

AXIAL BREAKING TENSION (MPa) - 28 DAYS			
MIZU CP II-Z-RS	MIZU CP II-Z-RS	FORTE CP II-Z-RS	POTY CP II-F-32
CP- 1	34,56	31,71	34,61
CP- 2	30,21	30,42	32,22
CP- 3	33,86	33,41	33,71
CP- 4	32,86	34,21	32,86
CP- 5	33,41	29,71	33,66
CP- 6	33,51	32,41	33,21
Minimum	30,21	29,71	32,22
Maximum	34,56	34,21	34,61
Average	33,07	31,98	33,38
D.P	2,28	2,97	0,67
C.V (%)	6,88	9,30	2,00

Source: The authors

In Figure 7, the experimental results were also grouped in pairs allowing a comparison between all resistances at 28 days, all the binders presented resistance between 29.71 MPa and 34.61 MPa, we also realized that despite the fact that cements are produced by manufacturers resistances are relatively close. Although one sample of the Mizu cement and three samples of the Forte cement were below 32 MPa, as specified by the NBR, however, practically all the cements showed average resistance within the satisfactory condition.

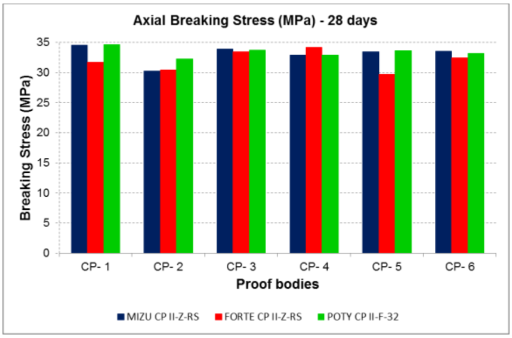

Figure 7: Grouped resistance results
Source: The authors

5. FINAL CONSIDERATIONS

The most relevant results obtained to classify a quality cement is the strength test after 28 days, this test demonstrates that the CPII-Z-32-RS cement from the manufacturer MIZU, had a value below its specification in only one of the six samples having a mean strength of the samples a value of 33.07 MPa which demonstrates a satisfactory comparison with the standard. For the cement CPII-Z-32-RS from the manufacturer FORTE, three of the six samples obtained results below its specification, presenting an average of 31.98 MPa that does not comply with the value informed by the manufacturer. For the CPII-F-32 cement from the manufacturer POTY, no sample was below the specified with an average of 33.38 MPa, thus satisfying its specification.

Of the three cements studied, Poty presented the best strengths, also presented the best values of average strength, standard deviation and variation coefficient, 33.37 MPa, 0.67 and 2.00%, respectively.

According to the data obtained and in disagreement with the result of the FORTE cement, there may be some error at the time the paste was made, for this reason it did not obtain the specified value. As has been seen, there are divergences between the manufacturer's results and the results found in the laboratory. The checks have the benefit of both the consumer and the manufacturer.

The work carried out allowed us to identify not only the mechanical behavior of the cement samples, but also to observe that the Portland cement paste is much more unstable, shrinking during the hydration and drying process and expanding when moistened. Being able to release heat during its hydration and induce cracking problems in pieces of large volume of mortar and concrete.

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