

Behavior of mortar blended with quartzite residues when subjected to natural aging

Comportamento de argamassas misturadas com resíduos de quartzito quando submetidas ao envelhecimento natural

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Abstract

The mixture of mortars with quartzite residues is a recycling technique that has been widely used to promote the adequate management of solid waste generated in the municipality of Várzea-PB, Brazil. However, there are no reports in the literature that prove the durability of the mortar mixed with these residues when subjected to environmental conditions over time. Thus, the present work has as objective to study the durability of mortars incorporated with quartzite residues when submitted to natural aging. The reference mortar and incorporated with quartzite were prepared according to NBR 13281 and submitted to natural aging for 28, 60,

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120, 180 and 360 days. Then, they were characterized by x-ray diffraction, thermogravimetric analysis and simple compression strength and carbonation and pozzolanic measurements. The results showed that, despite the decrease in compressive strength of quartzite containing mortars, the durability was not compromised, since the values were maintained in the range suggested by NBR 13281. Finally, it was concluded that mortars produced with quartzite residues presented good mechanical behavior when submitted to natural aging.

Keywords: Quartzite residue; Durability; Natural aging; Mechanical behavior

Resumo

A mistura de argamassas com resíduos de quartzito é uma técnica de reciclagem que tem sido amplamente utilizada para promover o adequado gerenciamento de resíduos sólidos gerados no município de Várzea-PB. No entanto, não há relatos na literatura que comprovem a durabilidade da argamassa misturada a esses resíduos quando submetidos a condições ambientais ao longo do tempo. Assim, o presente trabalho tem como objetivo estudar a durabilidade de argamassas incorporadas com resíduos de quartzito quando submetidas ao envelhecimento natural. As argamassas de referência e incorporadas com quartzito foram preparadas conforme NBR 13281 e submetidas ao envelhecimento natural por 28, 60, 120, 180 e 360 dias. Em seguida, foram caracterizadas por difração de raios-x, análise termogravimétrica, resistência a compressão simples e medições de carbonatação e pozonalidade. Os resultados revelaram que, apesar da diminuição da resistência à compressão das argamassas com incorporação de quartzito, a durabilidade não foi comprometida, uma vez que os valores foram mantidos na faixa sugerida pela NBR 13281. Por fim, conclui-se que as argamassas produzidas com resíduos de quartzito apresentaram bom comportamento mecânico quando submetido ao envelhecimento natural.

Palavras-chave: Resíduo de quartzito; Durabilidade; Envelhecimento natural; Comportamento mecânico

1 Introduction

After the act of the Brazilian Federal Law 12,305/2010, Article 27, the Brazilian mining companies that deal with ornamental rocks became responsible for the integral implementation of a residue management plan so that they could be licensed to explore and sell the ornamental rocks (Brazil, 2010). According to Article 13 of that Law, the residues generated in the extraction and beneficiation of quartzite are considered solid mining residues. This fact led to the widespread of recycling techniques applied to the residues produced in the town of Várzea/PB. Some of these techniques employ the residues, called artificial sand and crushed stone for construction, as feedstock for the production of mortar. However, the durability of mortar produced with artificial sand must be tested, so that this practice can be considered an effective way of reducing of the environmental impact, creating new job positions and income, settling conflicts between sawmill, and environmental agencies and the community, that is, given the prerogatives of Federal Law 12,305/2010 (BARROS et al., 2019; DANTAS et al., 2018; DANTAS et al., 2017).

There has been much research on recycling techniques in Brazil and other emerging countries. Such techniques focus on the use of residues of ornamental rocks as alternative feedstock to be used in construction with the main purpose of reducing environmental impacts and adding economic value to the residues (CRUZ; DANTAS; RAMOS, 2019; ERCIKDI; KULEKCI; YILMAZ, 2015; ULUBEYLI; ARTIR, 2015; BACARJI et al., 2013). The proper management of residues of ornamental rocks can be achieved by mixing them with cementitious matrices (ULUBEYLI GC, ARTIR, 2015; GAMEIRO et al., 2014). However, Farias Filho (2011) states that besides the definition of a proper technique to promote the recycling of ornamental rock residues for construction ends, it is necessary to perform studies that reveal the behavior and the properties of the alternative materials over time, that is, their durability.

Durability is an important feature in the development of alternative materials, which must have, in addition, similar properties to conventional ones in order to achieve market acceptance (VIJAYALAKSHMI; SEKAR; PRABHU, 2013). Moreover, it contributes to diversify their use in various industrial applications, especially in concrete and mortar (FARIAS FILHO

et al., 2011). One of the most used parameters to verify the lifetime of concrete is its simple compressive strength (SCS), which can indicate if there will occur or not its deterioration over the aging process (BINICI et al., 2008). According to Barros et al. (2016), the durability analysis of mortar blended with residues is a hard task, due to the complex systems of deterioration agents that interact with the components of these mortar compositions along their lifetime.

The deterioration of both alternative and conventional materials occurs as they are exposed to environmental conditions. Such conditions trigger an aging process that may lead to changes in the microstructure and, consequently, in their properties, like the mechanical features and porosity, since this interaction depends on humidity and temperature variations and climate (SCHACKOW et al., 2015; JOHN et al., 2005). According to Farias Filho et al., (2011) and John et al. (2005), in order to determine the degree of deterioration of mortar blended with residues, one must execute natural aging tests having the mechanical behavior as parameter, evaluated by the determination of the SCS, and the evolution of the microstructure by means of the characterization by X-ray diffraction (XRD) and thermogravimetric analysis (TG/DTG). XRD and TG/DTG are considered complementary techniques in the process of evaluation of the durability of alternative materials, since they allow the visualization of changes occurring in the microstructure and the identification of reactions that occurred in the aging process (RAMOS et al., 2019).

There are no reports in the worldwide literature on the durability of coating mortar blended with quartzite residues, and hence the present work has the purpose of verifying if the reactions that characterize the behavior of mortar blended with quartzite residues are the same that occur in conventional mortar and compare their durability with that of reference samples using the natural aging test and the XRD and TG/DTG techniques.

2 Materials And Method

2.1. Materials

The binding elements used in this research were: Portland Cement CP IV 32RS (Nassau@, Cimento Portland Sociedade Brasileira) and hydrated calcitic lime CH-I. This type of cement was chosen for having a pozzolane content of 25%, which minimizes the

carbonation of the lime produced at the hydration the Portland cement. The pozzolane content was determined by selective dissolution by the Voinovitch method. The hydrated lime used came from Indústria Carbomil, located in Limoeiro do Norte – CE, Brazil. The quartzite residues came from the Tecquímica do Brasil Company, located in the town of Várzea – PB. The natural sand used in the reference proportions came from the Paraíba River, dried in oven at a temperature of 110 °C, sieved in an ABNT sieve 4 (4.8 mm) in order to reduce the influence of the transition zone between the aggregate and the paste. Barros et al. (2016) determined the chemical composition of the cement, of the lime and of the quartzite residues used in this research (Table 1).

Table 1 - Chemical composition (% in mass) of the feedstock

Material	CaO	SiO₂	MgO	Al₂O₃	Fe₂O₃	SO₃	K₂O	TiO₂	P₂O₅	OO^a	LI^b
Cement (%)	45.1	28.2	2.2	8.1	3.1	5.4	2.1	0.3	1.3	0.2	4.0
Hydrated lime (%)	72.32	1.64	1.69	0.64	0.22	-	0.31	-	-	0.04	23.14
QP (%)	0.83	77.90	0.93	11.91	1.22	0.28	4.83	-	-	0.49	1.61
QS (%)	1.20	67.50	1.62	17.28	2.20	0.36	7.22	-	-	0.1	2.0

^aOO: other oxides; ^bLI: Loss on Ignition

The residues used as aggregate were labeled as: QP (quartzite powder), resulting from the sawing of quartzite plates; QS (quartzite sand), coming from chips resulting from the cutting of quartzite plates. The chips were subjected to the processes of crushing, grinding and classification in ABNT sieve 4 (4.8mm) in order to become quartzite sand. The physical properties (Table 2) of these residues were determined by Barros et al. (2016).

Table 2 - Physical properties of the quartzite sand (QS) and of the quartzite powder (QP)

Test		QS	QP
Unitary or apparent mass (g/cm ³)		1.33	1.79
Powdery material content (%)		1.06	100
Granulometry	Maximum diameter (mm)	2.4	Not applicable
	Thickness modulus (%)	2.90	Not applicable

According to the maximum diameter, the QS are classified as a normal aggregate of average granulometry. According to the NBR 5251 (ABNT, 2012) and NBR 5752 (ABNT, 2014) standards, the QP is classified as filler, since it presented pozzolanic activity neither with the lime nor with the cement. The pozzolanic activity index with the lime was of 1.06MPa (should be at least 6MPa) and with the cement it was of 53% (should be at least 75%). This behavior of the QP was expected due to its chemical and mineralogical composition, which essentially formed by crystalline silica. The QP was used as filler to improve the packing of the system.

2.2 Method

The analysis of the durability of the mortar samples under study consisted in a natural aging test, in which the test specimens were exposed to environmental and climatic conditions (sunlight and rain) of the town of Campina Grande-PB, Brazil for 28, 60, 120, 180 and 360 days. During the period of exposure of the test specimens (October 2015 to October 2016) to the environmental conditions, according to Instituto Nacional de Meteorologia data (INMET, 2016), the maximum monthly average temperature of 31.51°C and the minimum precipitation of 3mm occurred in November 2015 whereas the minimum monthly temperature of 17.92°C occurred in August 2016 and the maximum precipitation of 127.5mm occurred in January 2016. The relative humidity of the air varied from 71.37% to 83.29%. The average wind speed was 3.6m/s. After each exposure period, we determined the SCS of the mixture blended with the quartzite residues according to the NBR 7215 (ABNT, 1996) and compared the results with the respective conventional mixture, which were subjected to the same testing conditions.

The formulations of the mortar mixture blended with QS and QP residues to study the durability were determined in the experimental planning by means of the mixture delineation technique, according to the procedure described by Barros et al. (2016), which results in mixture with improved mechanical strength for application in construction, namely: 1:3 (cement: 100% QS and 0% QP); 1:3 (cement: 85% QS and 15% QP); 1:1:6 (cement: lime: 100% QS and 0% QP) and 1:1:6 (cement: lime: 85% QS and 15% QP). The amount of water for each formulation defined in the mixture delineation was determined by the flow table test, according to the NBR 13276 standard (ABNT, 2005), in order to guarantee the proper workability to the sample, being adopted the standard spreading of 260 ± 10 mm (Table 3). After determining the mixture and the amount of water, we molded 30 cylindrical test specimens sizing 50 mm X 100 mm for each proportion, which were cured for 28 days in humid chamber. After the cure, they were exposed to environmental conditions for 28, 60, 90, 120, 180 and 360 days and, at the end of each aging period, we determined their SCS. The SCS tests were executed in a SHIMADZU AG-IS 100KN universal machine, with loading speed of 0.25 ± 0.05 MPa/s.

Table 3 - Consistency of prepared mortars

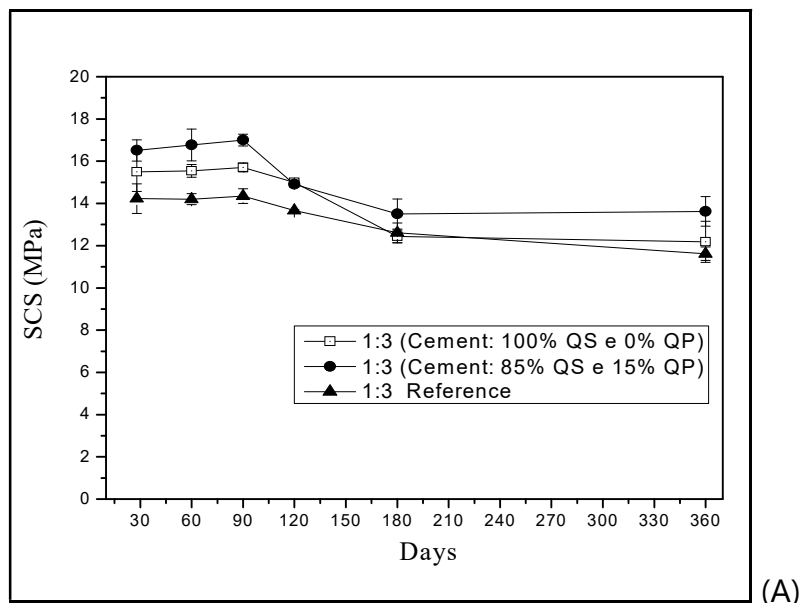
Mixture	Consistency				Water (ml)	a/c
1:1:6 (0%RFQ)	265	270	265	267	520	0.74
1:1:6 (15%RFQ)	250	255	248	251	490	0.71
1:3 (0%RFQ)	260	258	253	257	460	0.34
1:3 (15%RFQ)	255	260	252	256	450	0.34

In the present study, as complementary tests for the analysis of the durability, we used X-ray diffraction (XRD) (Shimadzu XRD 6000 device with $\text{CuK}\alpha$, radiation, voltage of 40kV, current of 30 mA in "fixed tim" mode and with angular step of 0.02° and time step of 0.6 s, running the angle 2θ from 10 to 60°), thermogravimetric analysis (TG) and its derivative (DTG) (BP Engenharia Indústria e Comércio's RB-3000 thermal analysis device, with heating speed of $12.5^\circ\text{C}/\text{min}$ from room temperature up to $1000^\circ\text{C}/\text{min}$). These tests had the purpose of evaluating the losses of mass corresponding to the mineralogical

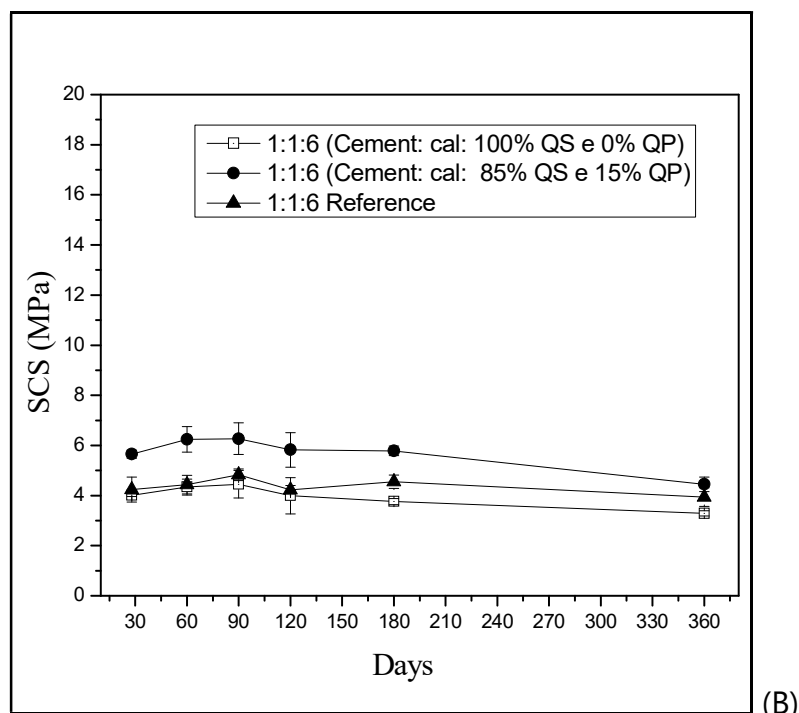
transformations after each exposure period. The preparation of the samples for these tests occurred by smashing the test specimens ruptured in the SCS test, then sieving the material in an ABNT sieve 200 (0.074 mm) and keeping the resulting material in closed containers until the due date for each test.

3 Results and discussion

Figures 1a and 1b illustrate the one-year evolution of the simple compressive strength of mortar for the mixture of 1:3 and 1:1:6.



(A)



(B)

Verified that the maximum SCS value occurs around 90 days, with a 1.29% increase rate of the SCS between 28 and 90 days, which corresponds to a raise of 0.20MPa for the proportion 1:3 with 0% QP; of 2.97%, which represents a raise of 0.49MPa for the mixture 1:3 with 15 % of QP; of 3.65% for the reference proportion of 1:3, which corresponds to a raise of 0.52MPa; of 10.97% which corresponds to a raise of 0.44MPa for the proportion 1:1:6 with 0% of QP; of 10.95% which corresponds to a raise of 0.62MPa for the mixture 1:1:6 with 15% of QP and of 13.92% for the reference mixture of 1:1:6, which corresponds to a raise of 0.59MPa.

The raises in the SCS of the mortar samples under study can be ascribed to the pozzolanic reactions that take place during the hydration process of the cement and to the carbonation resulting from the reaction between the air and the calcium hydroxide present in the mortar. These factors contribute with the refining of the microstructure and increase of the mechanical strength. According to Mota et al. (2016), the carbonation only affects the SCS in the case of reinforced mortar, since it may reach the reinforcement zone, putting the reinforcement steel at immediate risk of corrosion. It also causes a volume increase, starting a deterioration process.

After 90 days of natural aging, the SCS started to reduce over time, with decrease rates of: 22.42% between 90 and 360 days of exposure to environmental conditions, which corresponds to a decrease of 3.52MPa for the mixture 1:3 with 0% of QP; of 19.88% which corresponds to a decrease of 3.38MPa for the mixture 1:3 with 15% of QP; of 21.22% for the reference mixture of 1:3 which corresponds to a decrease of 3.13MPa; of 26.1% which corresponds to a decrease of 1.16MPa for the mixture 1:1:6 with 0% of QP; of 29% which corresponds to a decrease of 1.82MPa for the mixture 1:1:6 with 15% of QP and of 18.43% for the reference mixture of 1:1:6 which corresponds to a decrease of 0.89MPa.

The reduction of the SCS of the mortar under study is probably due to the variations in temperature, relative humidity of the air and to the action of the wind. These factors may cause thermal retraction, which in turn causes internal disaggregation of the components and, consequently, the reduction of the SCS. On the other hand, after 90 and 360 days of aging, the test specimens did not present external fissures before being subjected to the test for determination of the SCS.

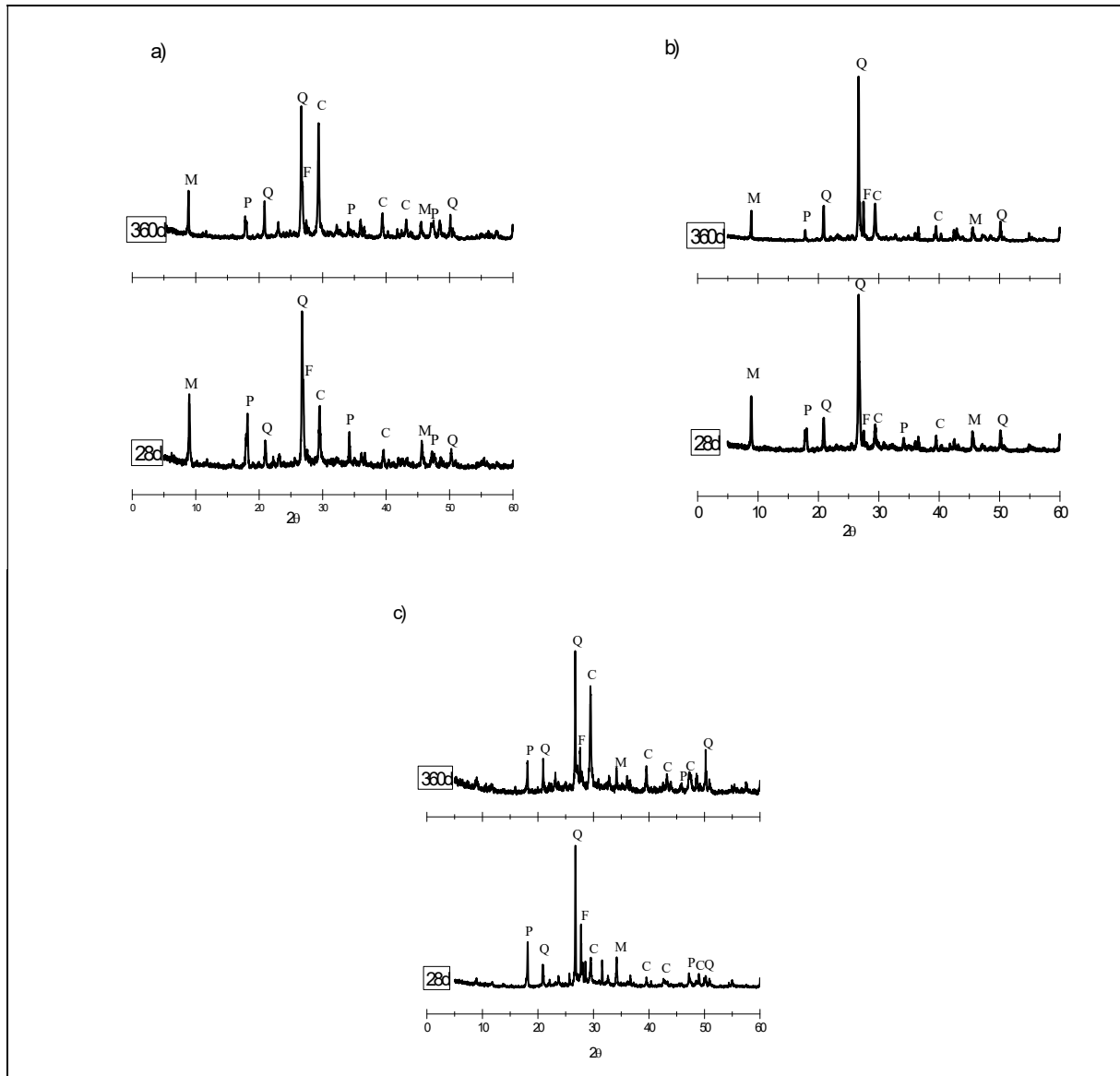
The results for the SCS of the test specimens subjected to natural aging (Figure 1) show that the mortar samples blended with quartzite residues had mechanical behavior similar to that of the reference samples over the aging process. Punctually at each rupture age, we noticed that the SCS values obtained for the alternative mortar compositions were higher than those of the reference samples, except for the mixture 1:1:6 with 100% QS and 0% QP. Similarly, the mixture 1:3 and 1:1:6 with 85% QS and 15% QP had better SCS values than the mixture 1:3 and 1:1:6 with 100% QS and 0% QP. This proves that the addition of 15% of QP to mortar leads to a better mechanical behavior due to an improvement in the system packing.

The mechanical performance of the mortar samples blended with quartzite residues (Figures 1) did not compromise the replacement of the natural sand with quartzite residue because, even after the decreases that occurred during the aging process for the mixture 1:3 with 0% and 15% of QP and for the mixture 1:1:6 with 0% and 15% of QP, their SCS values met the recommendations of the NBR 13281 (ABNT, 2005), standard for binding and plastering mortar, thus belonging to the classes P6 and P4, respectively.

The XRD results (Figures 2a, 2b, 2c, 3a, 3b and 3c) match the mechanical behavior achieved by these mixtures, due to the presence of characteristic peaks of portlandite, which are related to occurrence of pozzolanic reactions and of calcite, which in turn highlighted the occurrence of carbonation. Similar mineralogical and mechanical behaviors were found by Farias Filho et al. (2011).

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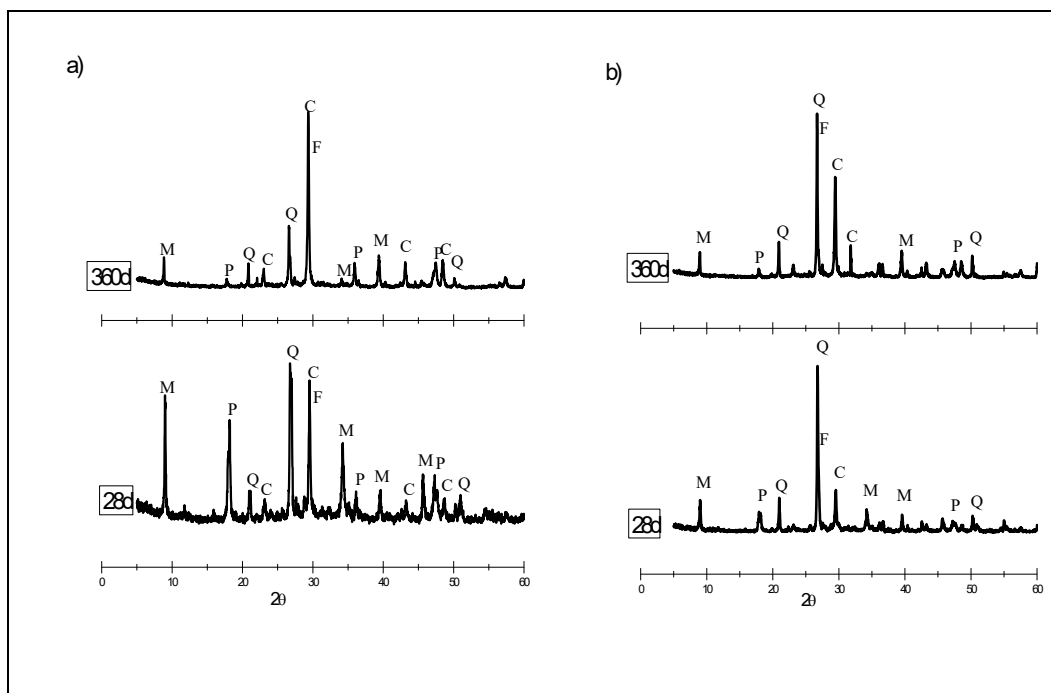
Figure 2 - a) XRD of the mixture 1:3 with 0% of QP b) XRD of the mixture 1:3 with 15% of QP and c) XRD of the reference mixture of 1:3. C-calcite, F-feldspar, M-mica, P-portlandite, Q-quartz

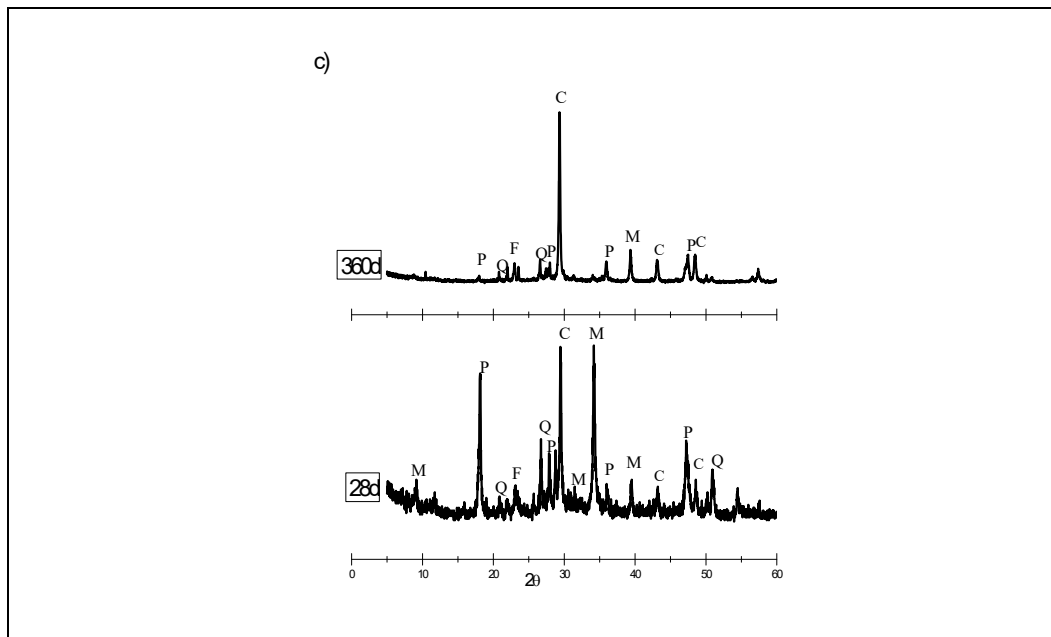


Figures 3a, 3b and 3c present the XRD of the test specimens after 28 days of curing and 360 days of aging for the mixture 1:1:6 with 0% of QP, 1:1:6 with 15% of QP and 1:1:6 of reference. We observed the following mineralogical phases (Figures 2a, 2b, 2c, 3a, 3b and 3c): calcite (JCPDS file: 72-1937, CaCO_3); feldspar (JCPDS file:84-0710, $\text{K}_5\text{Na}_5\text{AlSi}_3\text{O}_8$); quartz (JCPDS file: 46-1045, SiO_2); mica (JCPDS file:88-0791, $\text{K}(\text{Mg}_{2.665}\text{LiO}_{.225}\text{NaO}_{.110})(\text{Si}_{3.312}\text{FeO})$) and portlandite (JCPDS file: 72-0156, $\text{Ca}(\text{OH})_2$). These results are similar to those found by Balasubramanian et al. (2019) and Marmol et al. (2010) in their studies about

ornamental rock residues applied to mortar. Comparing the XRD curves of Figures 3a, 3b, 3c, 4a, 4b and 4c, we verified that the mortar samples blended with quartzite residues presented the same mineralogical phases as their respective reference samples along the aging process. Furthermore, the evolution of the mineralogical behavior of the alternative mortar samples between 28 days of curing and 360 days of exposure to environmental conditions was similar to that of the reference sample, being characterized by more intense portlandite peaks at 28 days and weaker peaks at 360 days, while the opposite occurred for the calcite peaks, demonstrating that the pozzolanic reactions occurred with higher intensity in the starting periods whereas the carbonation reactions had higher intensity in the most advanced stages of the aging.

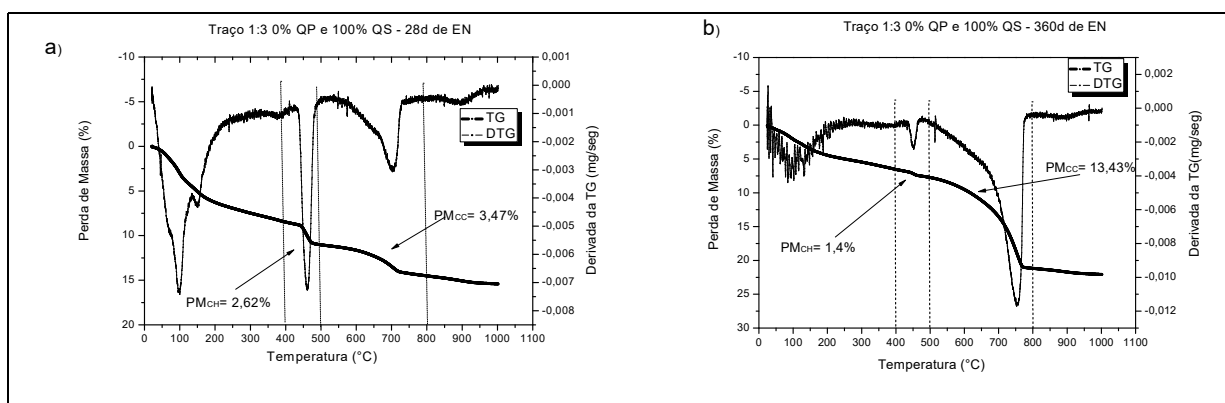
Figure 3 - a) XRD of the mixture 1:1:6 with 0% of QP b) XRD of the mixture 1:1:6 with 15% of QP and c) XRD of the reference mixture of 1:1:6. C-calcite, F-feldspar, M-mica, P-portlandite, Q-quartz

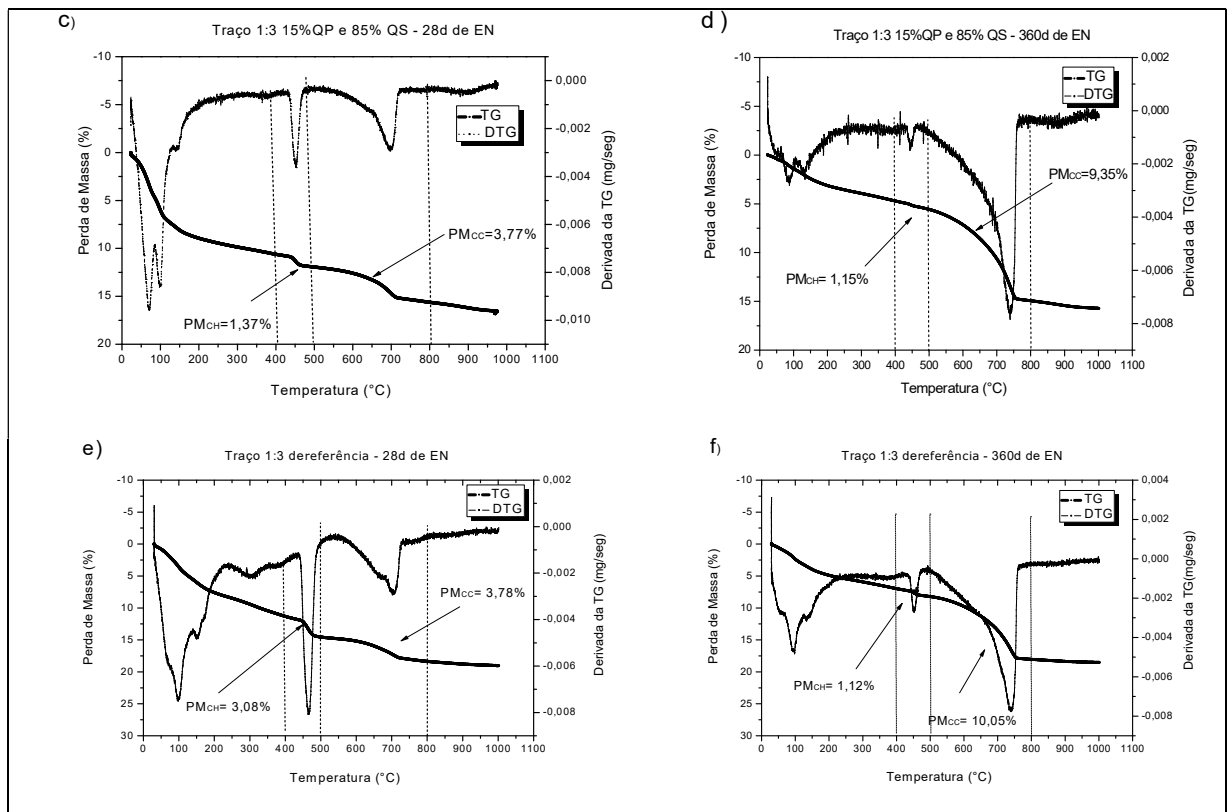




Figures 4a, 4b, 4c, 4d, 4e and 4f illustrate the TG and DTG curves after 28 days of curing and 360 days of aging for the mixture 1:3 with 0% of QP, with 15% of QP and for the reference mixture, respectively.

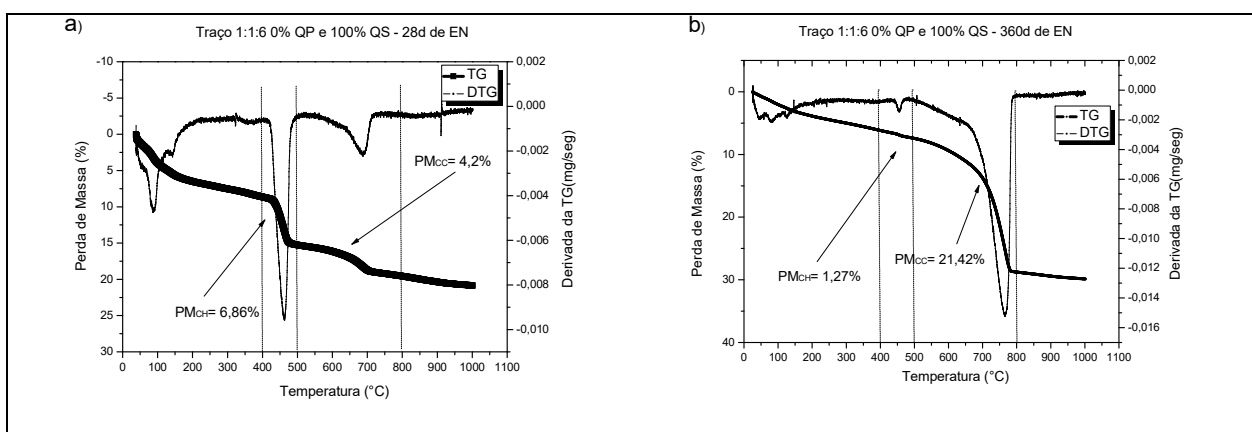
Figure 4 - a) TG and DTG of the mixture 1:3 with 0% of QP – 28 days of curing b) TG and DTG of the mixture 1:3 with 0% of QP - 360 days of aging c) TG and DTG of the mixture 1:3 with 15% of QP – 28 days of curing d) TG and DTG of the mixture 1:3 with 15% of QP – 360 days of aging e) TG and DTG of the reference mixture of 1:3 - 28 days of curing and f) TG and DTG of the reference mixture of 1:3 - 360 days of aging

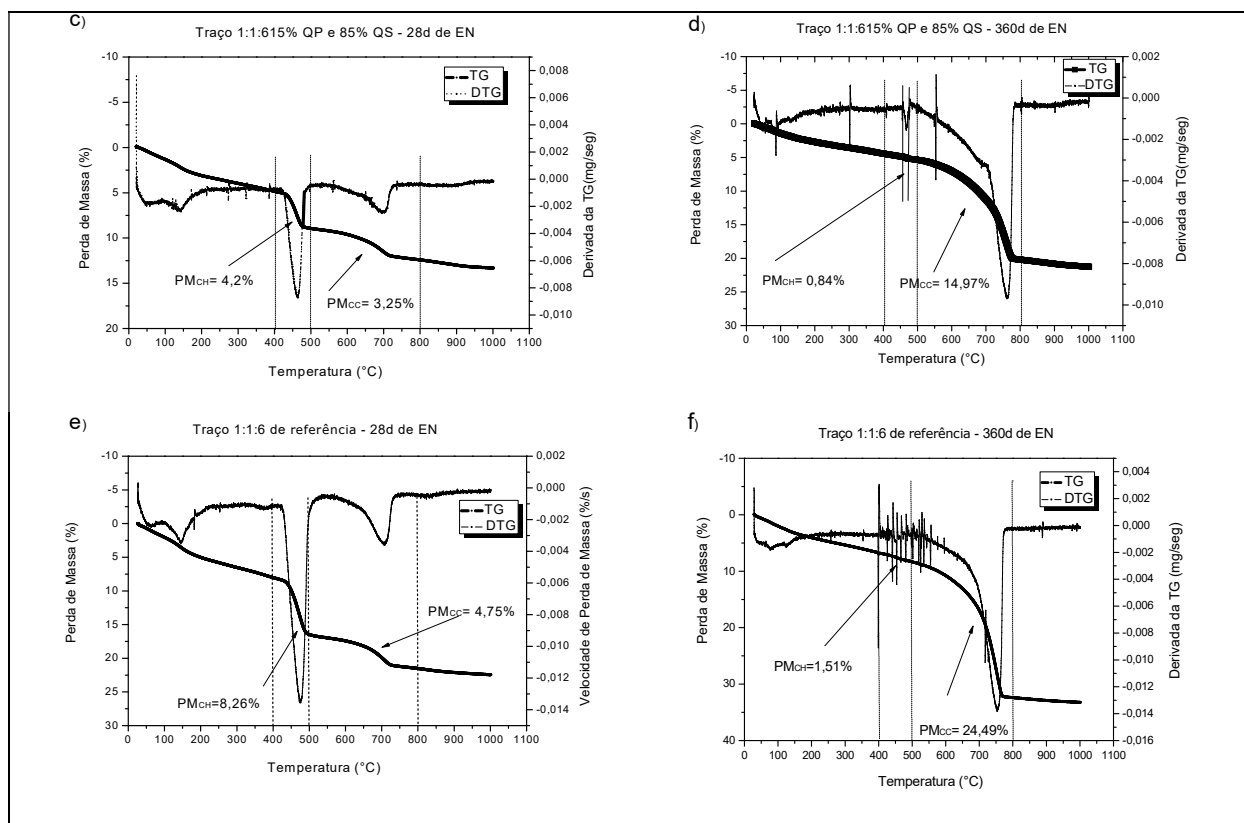




Figures 5a, 5b, 5c, 5d, 5e and 6f illustrate the TG and DTG curves after 28 days of curing and 360 days of aging for the mixture 1:1:6 with 0% of QP, with 15% of QP and for the reference mixture, respectively.

Figure 5 - a) TG and DTG of the mixture 1:1:6 with 0% of QP - 28 days of curing b) TG and DTG of the mixture 1:1:6 with 0% of QP - 360 days of aging c) TG and DTG of the mixture 1:1:6 with 15% of QP - 28 days of curing d) TG and DTG of the mixture 1:1:6 with 15% of QP - 360 days of aging e) TG and DTG of the reference mixture of 1:1:6 - 28 days of curing and f) TG and DTG of the reference mixture of 1:1:6 - 360 days of aging





Analyzing the TG and DTG curves (Figures 4 and 5), found a similar behavior between the mixture of the mortar mixed with the quartzite residues and the respective reference mixture. We also observed the occurrence of three well-defined zones according to the temperature ranges, and as the time of exposure to environmental conditions passed, the endothermic peaks revealed that the pozzolanic reactions reduced (ended) and the carbonation reactions started, reinforcing the results found in the XRD of these samples. The first zone comprised the range from 90 °C and 200 °C and presented endothermic peaks characteristic of the elimination of the free water and partial dehydration of the C-S-H. The second zone was between 400 °C and 500 °C and corresponded to the typical endothermic peak of the dehydroxylation of portlandite (CH). Finally, the third zone between 500 °C and 800 °C is characteristic of the decarbonation of calcite (CC). The identification of the endothermic peaks in the respective temperature ranges occurred according to Gameiro et al. (2014), Rocha et al. (2013) and Gameiro et al. (2012).

The quantification of the consumption of portlandite ($\text{Ca}(\text{OH})_2$) and the formation of calcite (CaCO_3) at the ages of 28 and 360 days followed the methodology developed by

Gameiro et al. (2014). Table 4 presents the percentage of the consumption of portlandite and of the formation of calcite according to the equation they developed.

Table 4 - Consumption of portlandite and formation of the calcite after 28 days of cure and 360 days of aging of the alternative and conventional mortar samples

Mixture	Age	CH poz (%)	CC carb (%)
1:3(cement:0%QP and 100%QS)	28	10.7	7.5
	360	5.7	30.1
1:3(cement:15%QP and 85%QS)	28	5.5	8.2
	360	4.6	20.9
Reference 1:3 (cement:natural sand)	28	12.6	8.6
	360	4.5	22.5
1:1:6 (cement: lime: 0%QP and 100%QS)	28	16.1	8.3
	360	0.1	29.9
1:1:6 (cement:lime : 15%QP and 85%QS)	28	12.1	6.2
	360	1.7	32.8
Reference 1:1:6 (cement: lime: natural sand)	28	28.8	9.6
	360	1.1	36.7

^aCH poz – percentage of portlandite consumed in the pozzolanic reactions and ^bCC carb – percentage of calcite formed in the carbonation reactions.

During the natural aging process of the mortar samples blended with quartzite residues and of their respective reference samples, we noticed (Table 4) the occurrence of pozzolanic reactions (revealed by the consumption of $\text{Ca}(\text{OH})_2$ portlandite or calcium hydroxide) and of carbonation (revealed by CaCO_3 or calcite or calcium carbonate). These results match those found by XRD, TG, and DTG and agree with the mechanical behavior found for the mortar samples under study.

Multivariate statistics was performed using Principal Component Analysis (PCA), and all results were based on the correlation matrix of the data. Table 5 shows that two components (1 and 2) with eigenvalues greater than one were extracted, component 1 with eigenvalue of 2.45 which represents 61.34% of the total variance and component 2 with eigenvalue of 1,107 which represents 27.69% of the total variance, totaling a total explained variance of approximately 89.03%.

Table 5 - Principal components extracted from samples

Components	Eigenvalue	% Total variance	Cumulative Eigenvalue	Cumulative %
1	2.45	61.34	2.45	61.34
2	1.11	27.68	3.56	89.03

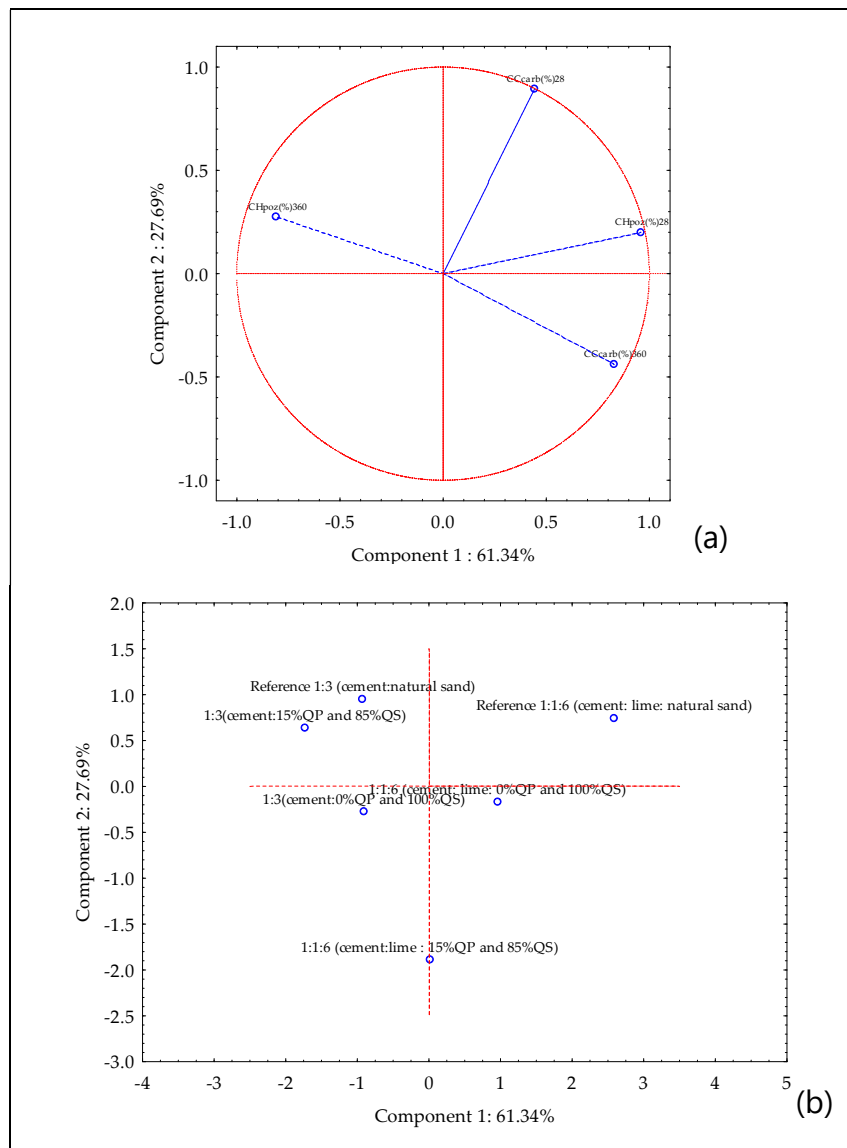
In Figure 6a is the PCA considering the variables: CHpoz(%)28, CCcarb(%)28, CHpoz(%) 360 e CC carb (%) 360. It is observed that the variables CHpoz(%)28, CHpoz(%)360 e CCcarb(%)360 are more closely linked to component 1, indicating that these have greater explanatory power for the variance of the mixtures used, especially the variable CHpoz (%) 28 with greater explanatory power. In component 2 we have the variable CCcarb(%)28 which was responsible for most of the variation in this component, but has less relevance than those that configure mostly in component 1.

Figure 6b shows the mixtures classified according to their relationship with the four variables studied. The mixture 1:3(cement:15%QP and 85%QS) got close to the mixture Reference 1:3 (cement:natural sand), this shows a strong relationship with the variable CHpoz(%)360. For mixture Reference 1:1:6 (cement: lime: natural sand) was linked to the variables CHpoz(%)28, CCcarb(%)28,

This indicates that this mixture is influenced by these variables. The remaining mixtures stood out by not being influenced by the variables or by having low values for the variables.

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Figure 6 - a) Principal Component Analysis of the variables CHpoz (%) 28, CCcarb (%) 28, CHpoz (%) 360 and CCcarb (%) 360 b) mixtures classification analysis



4 Conclusion

After the studies on the durability by natural aging of the mortar samples blended with QS and QP residues and of reference samples, we can conclude that:

- The SCS values reached maximum at 90 days of exposure to environmental conditions, when the pozzolanic reactions ended and the carbonation reactions started, according to the results of the mineralogical analysis by XRD, TG/DTG, both for the sample blended with quartzite residues and for the reference sample.

- The mixture with 15% of QP presented a better mechanical behavior in the aging process, showing that their use as filler materials improves the system packing.

- The mechanical behavior of the mortar sample blended with quartzite residues during the aging process was similar to that of the reference sample.

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