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ORIGINAL ARTICLE

Effect of filler nature on mechanical performance and drying shrinkage of self-leveling mortars

Efeito de fílers minerais no desempenho mecânico e na retração de argamassas autonivelantes

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Abstract: Self-leveling mortars (SLM) have a high filler content in their composition to achieve fluidity, without component separation. These mortars are mainly used in floor systems and have a large area exposed to the external environment, which facilitates water loss and increases the risk of cracking due to drying shrinkage. This study, in order to verify the influence of filler on the production of SLMs, used for comparative effect, quartz filler (QF) and crushed basalt filler (BF), in addition to limestone filler (LF), in contents of 5%, 10% and 20% of the mass of total solid. For that purpose, the tests of spreading in the fresh state and flexural strength, compressive strength, dynamic modulus of elasticity, drying shrinkage and desorption isotherm in the hardened state were carried out. SLMs microstructure was evaluated through isothermal calorimetry. The results show that the use of LF provides higher compressive strengths (up to 18% and 14% higher than QF and BF, respectively, in the first ages, and up to 13% and 22% higher than QF and BF, respectively, at 28 days), probably due to the better physical effect of this filler. In formulations with 20% of filler, LF and QF SLMs showed lower shrinkage when compared to BF SLMs (shrinkage 44% higher than LF mixture and 64% higher than QF mixture). It is concluded that the ideal content of filler to be used in SLMs must be between 10% and 20% of the mass of total solid and varies according to the filler.

Keywords: self-leveling mortar, filler, mechanical behavior, hygroscopic, drying shrinkage.

Resumo: As argamassas autonivelantes apresentam elevado teor de fílers em sua composição para o alcance da fluidez, sem separação dos componentes. Essas argamassas são utilizadas principalmente em sistemas de pisos e apresentam uma grande área exposta ao ambiente externo, o que facilita a perda de água e aumenta o risco de fissuração devido a retração por secagem. O presente estudo, com o intuito de verificar a influência do fíler no comportamento mecânico e na retração por secagem de argamassas autonivelantes, utilizou para efeito comparativo, os fílers quartzoso (QF) e de britagem basáltico (BF), além do fíler calcário (LF), em teores que correspondem a 5%, 10% e 20% em massa. Para tanto, foram realizados os ensaios de espalhamento no estado fresco e resistência à tração na flexão, resistência à compressão, módulo de elasticidade dinâmico, retração por secagem e isoterma de dessorção no estado endurecido. A microestrutura das argamassas foi avaliada por meio de calorimetria isotérmica. Os resultados obtidos mostram que a utilização do LF proporciona resistências à compressão superiores (até 18% e 14% maior que QF e BF, respectivamente, nas primeiras idades, e até 13% e 22% maior que QF e BF, respectivamente, aos 28 dias), provavelmente devido ao melhor efeito físico desse fíler. Em formulações com 20% de fino, argamassas de LF e QF apresentaram menor retração, se comparadas com argamassas de BF (retração 44% maior que a formulação com LF e 64% maior que a com QF). Conclui-se que o teor ideal de fíler a ser empregado nessas argamassas deve estar entre 10% e 20% da massa de sólidos e varia conforme o fíler.

Palavras-chave: argamassa autonivelante, fíler, comportamento mecânico, higroscopia, retração por secagem.

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1 INTRODUCTION

Self-leveling mortar, commonly used in flooring systems, is characterized by a smooth and regular surface, high fluidity, high early strength and volumetric stability [1]. To achieve the necessary fluidity without component segregation, high levels of cement and fillers (<125μm), usually limestone filler, are added. The limestone filler has been widely used in cementitious materials, including self-leveling mortars, due to economic factors, its physical [2], [3] and chemical effects [4] and its good performance in mechanical strength [5].

Currently, in Portland cement compounds, the effect of different types of fillers has been compared [6], [7], [2], [3], [8]. The literature reports that the effect of limestone filler is more effective than quartz filler in accelerating the C3S hydration. According to Berodier and Scrivener [2], this is due to the dissolution of this phase and may be related to the fact that the limestone has a favorable surface structure to the C-S-H nucleation (in the end of the induction period, the limestone surface is completely covered with nuclei, which is not observed on the quartz surface at the same age). Kumar et al. [3] states that the limestone filler accelerates initial hydration due to its ability to induce ion exchange reactions with C-S-H, which increases the driving force of C-S-H growth.

The basaltic filler seems to act as intert material at early ages and latter its reactivity is increased due to pozzolanic reaction. In a comparative study, Nam and Chuong [6] reported that the addition of limestone filler in mortars benefits the initial strength development, while the use of basaltic filler contributes more prominently to later age strength, due to the slowness of the pozzolanic reaction. According to the study of Saraya [7], even in early ages, pastes with basalt showed better mechanical properties than those containing limestone as filler, as a result of the better packing caused by basalt, forming a more dense matrix, and a better dispersion of cement grains. The use of basaltic filler improves the strength of the concrete, mainly due to the better packing of particles. In addition, the particles act as nucleation sites for the precipitation of hydration products, accelerating the hydration of the cement grains and contributing to the increase of early age strength. Also, chemical interactions may occur on the surface of the basalt particles, through a gradual local exchange of ions between minerals on the basalt surface and C-S-H phase. Calcium ions diffuse from the pore solution and exchange magnesium, while magnesium ions diffuse into C-S-H phase. This local transformation of the minerals leads to enhancement of the interfacial transition zone [9].

Moreover, according to Liu et al. [10], the supplementary cementitious materials (SCMs) affect the compressive strength of pastes through three main effects: characteristics inherent to SCM, particle size refinement of the SCM and hydration of the SCM. The inherent characteristic effect of SCM is related with surface texture, charge properties, elastic modulus and others. The particle size refinement effect influences the paste compressive strength due to the increase of the interface area between SCM particles and hydration products, while the interface distributes more homogeneously (this effect account for 1% - 10% of the total compressive strength). The hydration effect of SCM leads to a densification of interface between SCM particles and the matrix, through the formation of hydration products and, consequently, mechanical properties of the interface can be improved. The high activity of fine particles is of great importance to early strength, while the 28 days compressive strength is significantly influenced by the activity of SCM.

However, it is still unclear whether the use of different types of filler influences the mechanical behavior and dimensional stability of SLMs. The role of limestone filler in the dimensional stability of cementitious systems has been studied and research shows that its incorporation may not change significantly [11] and in some cases may reduce drying shrinkage [12]. According to Itim et al. [12], the replacement of cement by this filler up to 15% may reduce drying shrinkage, because it provides a less porous structure, forming more refined pores, preventing desiccation. Benachour et al. [11] reported that the addition of up to 35% limestone filler (sand replacement) does not cause increases in drying shrinkage, despite inducing a finer pore size population. The higher specific surface of the filler, compared to sand, increases the water demand due to the adsorption phenomenon. On the other hand, filler particles fill the voids and the water has less pore space available.

However, the information available in the literature about the influence of mineral additions on drying shrinkage is still contradictory. Some authors claim that the use of these additions reduces drying shrinkage, possibly due to the matrix densification, preventing the evaporation of internal moisture, or because of the restriction to shrinkage deformation [12], [13]. Differently, other studies report that mixtures with incorporation of mineral additions show greater drying shrinkage. This could be due to the formation of smaller capillary voids. The shrinkage occurs mainly due to the loss of water from smaller voids [14]. Moreover, it is known that the drying shrinkage generally increases with the increase in the paste volume, since only the paste shrinks due to drying [15].

Self-leveling mortars have a large area exposed to the external environment, which facilitates water loss and increases the risk of cracking. Therefore, it is necessary to understand the impact of the filler nature on the mechanical properties and on the drying shrinkage of these mixtures to obtain durable products. This research used for comparative effect, in addition to limestone filler (LF), quartz filler (QF) and crushed basalt filler (BF) in contents of 5%, 10% and

20% of the mass of total solid in the production of these mortars. The study aimed to identify the impact of these fillers on the resulting mortar and the filler content that may be added to them without loss of performance.

2 MATERIALS AND METHODS

2.1 Materials

Portland cement CPV - ARI was used as a binder, according to NBR 16697 [16] (equivalent to Type III Portland cement specified by ASTM C150 [17]). This cement was chosen due to the low admixture content and because it presents a high initial mechanical strength, suitable for self-levelling systems. Three different fillers were evaluated: limestone (LF), quartz (QF) and crushed basalt (BF). The properties of cement and fillers are given in Table 1 and their particle size distributions are given in Figure 1. The LF is the filler that has the smallest particle size. The particle size distribution of QF and BF were similar, although the range of particle size distribution of BF is narrower. A quartz sand with fineness of 2.09, specific mass of 2.52g/cm^3 and 6.5% of powdery material was used as fine aggregate. A polycarboxylate superplasticizer with specific mass between 1.080 - 1.120g/cm^3, pH between 4,5 - 6,5 and solid content of 51%, was used.

Table 1. Chemical and physical properties of cement and fillers.

Composition (%)	Cement	Limestone filler	Quartz filler	Crushed basalt filler
SiO_2	17.297	3.210	99.457	68.599
Al_2O_3	-	-	-	12.775
Fe_2O_3	3.027	0.754	0.043	5.887
CaO	66.096	62.037	0.034	5.174
K_2O	1.376	0.202	0.031	3.781
SO_3	4.632	-	0.092	-
CO_2	6.930	33.550	0.280	2.670
Specific mass (g/cm^3)	3.16	2.85	2.74	2.52
Blaine specific surface (cm^2/g)	5,416.44	2,558.78	2,944.42	2,804.63

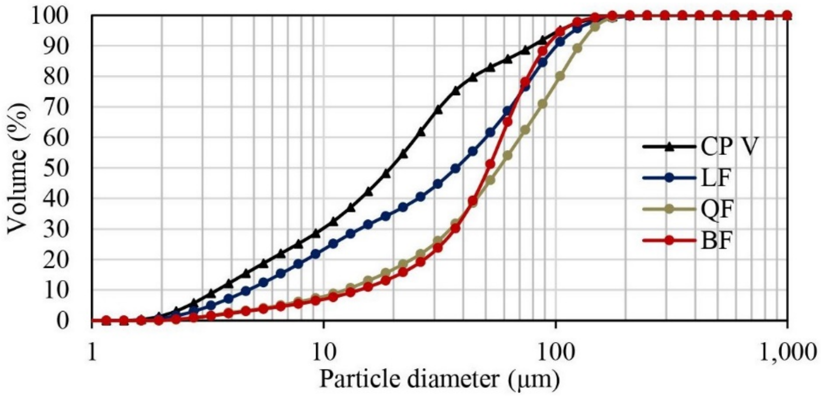

Figure 1. Particle size distributions of cement and fillers.

Figure 2 shows images of limestone, quartz and crushed basalt filler. It is possible to observe that the limestone and the crushed basalt filler surfaces are rougher than that of the quartz filler. The three fillers have particles with angular shape.

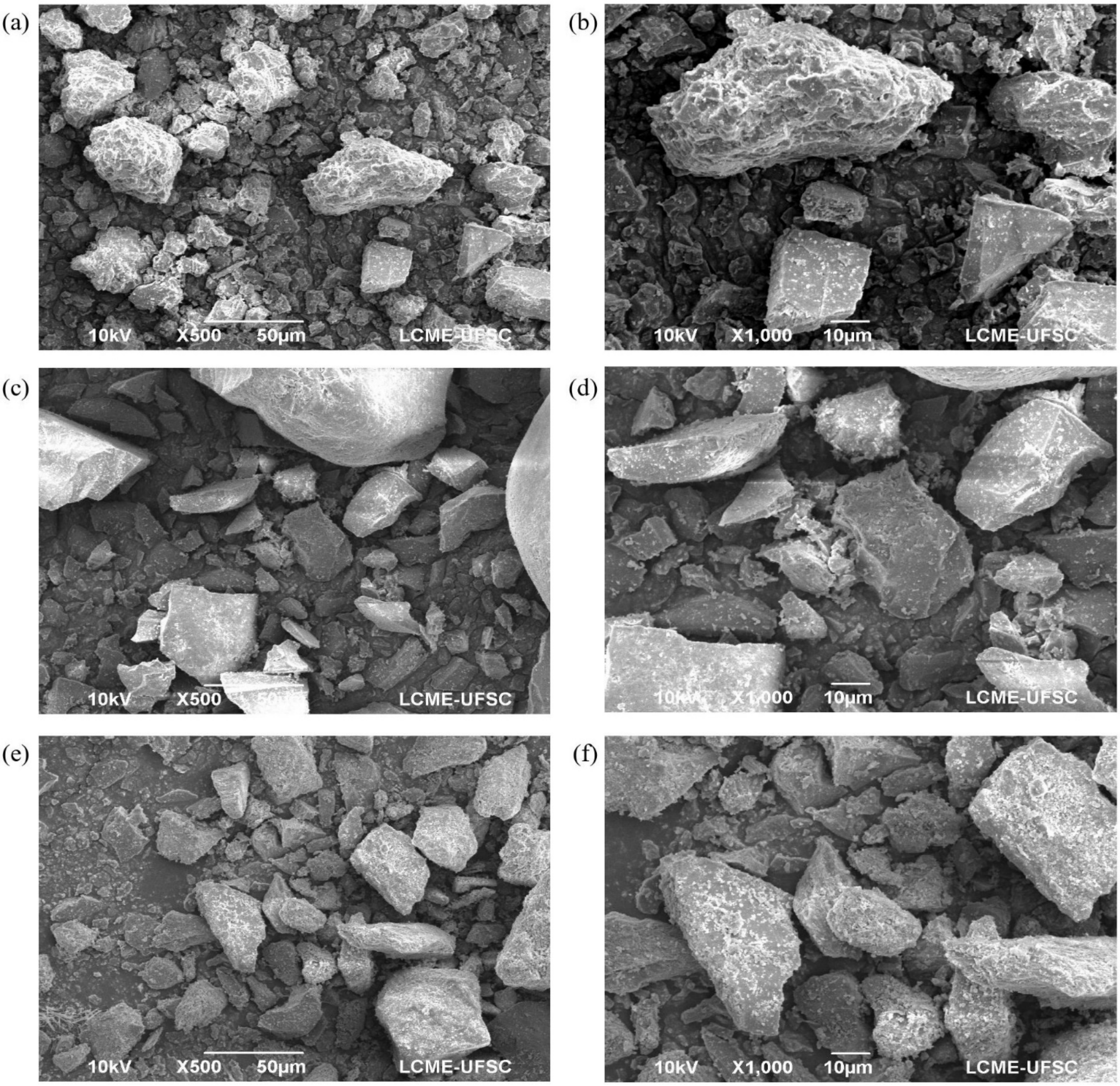

Figure 2. SEM micrographs of (a) limestone with 500x and (b) 1000x magnification, (c) quartz with 500x and (d) 1000x magnification and (e) crushed basalt filler with 500x and (f) 1000x magnification.

2.2 Mixture proportion and test program

In this study, SLMs were produced considering an agglomerate: aggregate (cement: filler + sand) ratio of 1:2 (mass) and water/cement ratio (w/c) of 0.5 (wt%). The mixture proportions were defined based on preliminary studies, aiming to verify the ability to incorporate aggregates in SLMs. These studies involved some visual analysis: leveling, edge quality, risks of segregation and bleeding. For mortars with a 1:2 ratio, the following requirements in the fresh state were: self-leveling, regular edges and absence of signs of segregation or bleeding. The limestone filler was used as a reference, due to its traditional use in self-leveling and self-compacting mortars [18]–[20] and it was used in contents of 5%, 10% and 20% of the mass of total solid. Limestone filler replacements by other fillers (quartz and crushed basalt) were performed with specific mass correction. Thus, the volume of filler used in each mixture with the same filler content was not changed.

Due to the correction of the specific mass of the fillers in relation to the LF in each filler contents, the percentage of mass of QF and BF show a small difference from the percentages initially defined (5%, 10% and 20%). However, to facilitate the designation of the samples, the nomenclatures QF5, QF10, QF20, BF5, BF10 and BF20 were maintained. The mixtures were prepared adding different amounts of superplasticizer in order to achieve spreading values between 25 and 30cm, values recommended by Barluenga and Hernández-Olivares [21] for SLMs, which need large flow ability. In view to ensure a pratical application, the required dosage of superptasticizer was establish to ensure a spreading diameter of 27,5 ± 2,5 cm, as listed in Table 2.

The mixing procedure was done according to NBR 16541 [22]: 1) mixing of anhydrous materials at low speed for 60s, 2) mixing at low speed for 30s with the addition of 75% of water in the initial 10s, 3) mixing at high speed for 60s, 4) stop the mixer for 90s for scraping the bowl and paddle, 5) mixing at low speed for 60s with the addition of the remaining 25% of water and superplasticizer in the initial 10s.

Table 2 provides the detailed composition of the SLMs.

Table 2. Mixture proportion of SLMs.

Mixture	Binder (kg/m^3)	Aggregate (kg/m^3)				Water (kg/m^3)	%SP[1]	Paste volume (%)[2]	Spreading (cm)
	Cement	Limestone filler	Quartz filler	Crushed basalt filler	Sand				
LF5	621.33	93.21	-	-	1149.63	310.66	0.45	54.38	28.5
LF10	624.00	187.22	-	-	1060.97	312.00	0.45	57.90	28.0
LF20	629.41	377.69	-	-	881.33	314.71	0.40	65.03	30.0
QF5	621.33	-	89.61	-	1149.63	310.66	0.40	54.38	25.0
QF10	624.00	-	179.99	-	1060.97	312.00	0.45	57.90	28.5
QF20	629.41	-	363.11	-	881.33	314.71	0.43	65.03	25.0
BF5	621.33	-	-	82.42	1149.63	310.66	0.45	54.38	28.5
BF10	624.00	-	-	165.54	1060.97	312.00	0.60	57.90	26.0
BF20	629.41	-	-	333.95	881.33	314.71	0.70	65.03	29.3

[1] By cement mass. [2] Paste = cement, filler and water.

2.2.1 Workability

Workability was measured by means of the spreading diameter of SLMs. For this purpose, a cone trunk was placed at the center of a glass plate and filled with mortar. After, the truncated cone was lifted and the mortar spreads. The average of two perpendicular measurements of diameter was taken. The cone trunk used has the following dimensions: ϕsup = 70mm, ϕinf = 100mm and h = 50mm. The spreading diameter of SLMs was carried out to determine the content of superplasticizer needed for each mixture to reach a spreading flow between 25 and 30 cm.

2.2.2 Flexural and compressive strength

Flexural strength was evaluated in three 40x40x160mm prismatic specimens and compressive strength was determined in each of the halves of the specimens resulting from flexural test, according to NBR 13279 [23]. Mixtures were poured into molds without any vibration and compaction. Specimens were demolded 24h after casting and were covered with plastic film until demolding. Specimens were tested at 1 and 28 days.

2.2.3 Dynamic modulus of elasticity

Dynamic modulus of elasticity of SLMs was determined in three 40x40x160mm prismatic specimens, at 28 days of age, according to NBR 15630 [24]. The test was carried out in a room with 60 ± 5% RH and the specimens remained until the 28th day in the same room. Specimens were demolded 24h after casting and were covered with plastic film until demolding.

2.2.4 Desorption isotherm

Water vapor isotherm test was conducted in accordance with C 1498-01 [25] in specimens with 10% e 20% of filler, obtained from the specimens that were evaluated for shrinkage up to 28 days. After shrinkage test, the specimens were sawn into pieces of approximately 5mm thick and section 25x25mm. Afterwards, specimens were dried at 50ºC for 48 hours. Later drying, the samples were placed in airtight containers with decreasing relative humidity to check the mass moisture content in each relative humidity (RH).

The different relative humidity of the environments were provided by six salts: potassium sulfate (K_2SO_4), sodium chloride (NaCl), sodium nitrite ($NaNO_2$), magnesium chloride ($MgC_{l2}.6H_2O$), potassium acetate (KCH_3CO_2) and hydroxide potassium (KOH), which provided the relative humidity (RH) of, respectively, 96%, 75%, 65%, 31%, 20% and 6% at 50ºC. The mass moisture content was calculated according to Equation 1.

$$W = \frac{m - m0}{m0} x100 \tag{1}$$

where W = mass moisture content (%); m = mass of the equilibrium specimen (g) and m0 = mass of the dry specimen (g).

The GAB method was used to obtain the adjustment of the experimental points using the nonlinear least squares method. The specific surface of SLMs (SGAB) was calculated according to Equation 2.

$$S = \frac{Wm\ x\ am\ x\ Navg}{M} \tag{2}$$

where S = specific area of the material (m^2/g); Wm = monolayer moisture content (g/g); am = area occupied by one water molecule ($10Å^2$ or $10 \times 10^{-20} m^2$); Navg = Avogadro's number (mol^{-1}) and M = molecular mass of water (g/mol).

2.2.5 Drying shrinkage

Drying shrinkage was measured in two 25x25x285mm prismatic specimens. According to C 1708 [26], specimens were demolded after 24 hours and then the initial length reading was done (with 1 day). The other readings were taken at 3, 7, 14 and 28 days from the moment of mixing the materials. Specimens were covered with plastic film in the first 24h and after demolding they were stored in an environment with 65 ± 10% RH. Shrinkage was calculated according to Equation 3.

$$\varepsilon i = (Li - L0)/250\ x\ 100\% \tag{3}$$

where ε i = value of shrinkage at age "i" (%); Li = length at certain age (mm); L0 = length after demolding (mm); i = age of measurement and 250 = effective length of mortar specimen (mm).

Mass loss was calculated according to Equation 4.

$$\triangle m = (mi - m0)/m0\ x\ 100\% \tag{4}$$

where $\triangle$m = mass loss (%); mi = mass at certain age (g) and m0 = mass after demolding (g).

2.2.6 Hydration kinetics by isothermal calorimetry

The test was carried out on pastes considering the same proportion between Portland cement and filler used in LF20, QF20 and BF20 SLMs and in a reference paste (REF) containing only cement. The ratio water/binder = 0.5 was used and, in this case, the fillers were considered as part of the binder. The cement mass was kept constant in all pastes and therefore the fillers were used as an addition to the pastes.

The hydration kinetics of the pastes were evaluated by isothermal calorimetry in a Thermometric AB calorimeter from TAM Air from TA Instruments. The powdered materials were previously homogenized for 1 minute. Then, water was added and, after manual external mixing for a period of 2 minutes, the samples were inserted into the calorimeter. The heat released was recorded at a temperature of 23ºC.

3 RESULTS AND DISCUSSIONS

3.1 Workability

In Table 2 it is possible to observe that, in general, with the increase in filler content, there is an increase of superplasticizer content to obtain the target spreading, since the w/c ratio was kept constant. The increase in the filler content requires a greater amount of water to wet the surface of the particles, due to the greater specific surface area [18].

Higher contents of superplasticizer were necessary for formulations with BF. Although this material has the specific surface slightly smaller than the specific surface of QF, the values are quite similar between these fillers. However, the BF particles have a rougher texture, which may result in a decrease in the mixture fluidity and an increase in the superplasticizer demand for the same spreading [19], [27].

All mixtures showed spreading between 25 and 30cm and without segregation or bleeding.

3.2 Flexural and compressive strength

Figure 3a shows the results of flexural strength and Figure 3b, the results of compressive strength of SLMs, at 1 and 28 days.

The flexural strength at 1 day increased with the increase in filler content. However, at 28 days, there was no noticeable influence of the use of different types and contents of filler. About compressive strength, the use of filler up to 20% resulted in higher early strength for all fillers. At 28 days, the compressive strength increased significantly with the increase of LF up to 10%, however, the use of QF and BF in 5%, 10% and 20% resulted in equivalent compressive strength.

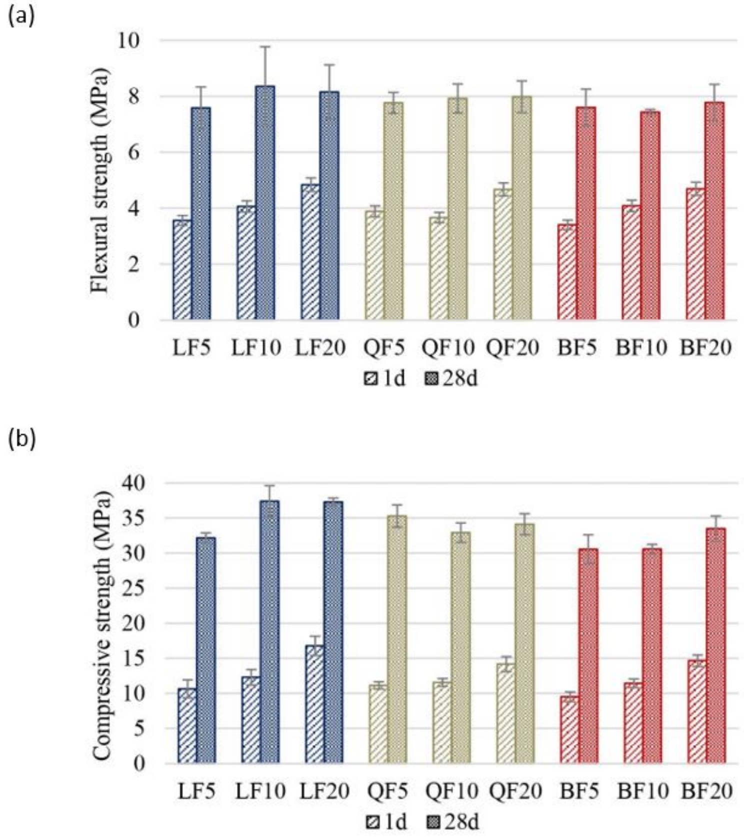

Figure 3. (a) Flexural and (b) compressive strength of self-leveling mortars at 1 and 28 days.

With the use of filler, the solid matrix may have been reinforced due to a more homogeneous distribution of smaller C-S-H crystals [11] and due to the filling of voids between the cement paste and the fine sand particles [18]. Filler acts mainly on the first day of hydration, when the microstructure is developing rapidly. Hydrates, such as C-S-H, form connections between cement grains, explaining the compressive strength increase at 1 day as the filler content increases [2].

Regarding the type of filler, it was observed that at 1 day, only SLMs with 20% of filler showed differences in the results of compressive strength. No influence of the filler nature in SLMs with 5% and 10% of filler was verified. SLMs containing LF showed higher early compressive strengths, LF20 showed compressive strength 18% and 14.4% greater than QF20 and BF20, respectively. At 28 days, in general, compressive strengths were also greater for formulations with LF, up to 13% and 22% greater than QF and BF, respectively.

It was observed that the limestone filler was more favorable to the development of compressive strength compared to quartz and crushed basalt filler. Assuming that limestone filler is an inert material, mixtures with LF show better compressive strength, probably due to the more effective behavior of LF in relation to the physical effects when compared to QF and BF. As the w/c ratio was fixed for all SLMs and the amount of cement was constant in formulations with the same filler content, it was excluded the possibility of dilution effect on the mixtures. So, the higher compressive strength values of SLMs mixes with LF are probably result of the inherent characteristic effect (such as surface texture, charge properties and elastic modulus) and the particle size refinement effect of LF (LF d50=37μm, QF d50=57μm and BF d50=51μm). A hypothesis is that the inherent characteristic may have affected the heterogeneous nucleation of C-S-H on the filler surface and the particle size may have reduced the distance between particles, due to a more homogeneous particle distribution in the cement paste, besides having contributed with nucleation.

From a practical purpose, the compressive strength at early ages may be a motivation for the use of LF, since early compressive strength is important in self-leveling systems. Moreover, similarly to the early strength, the use of LF influenced the compressive strength at 28 days.

In the SLMs of this study, sand was replaced by fillers in mass, which resulted in slight changes in cement consumption between the mixtures. Figure 4 shows the relationship between cement consumption and the compressive strengths achieved by mortars at 1 and 28 days.

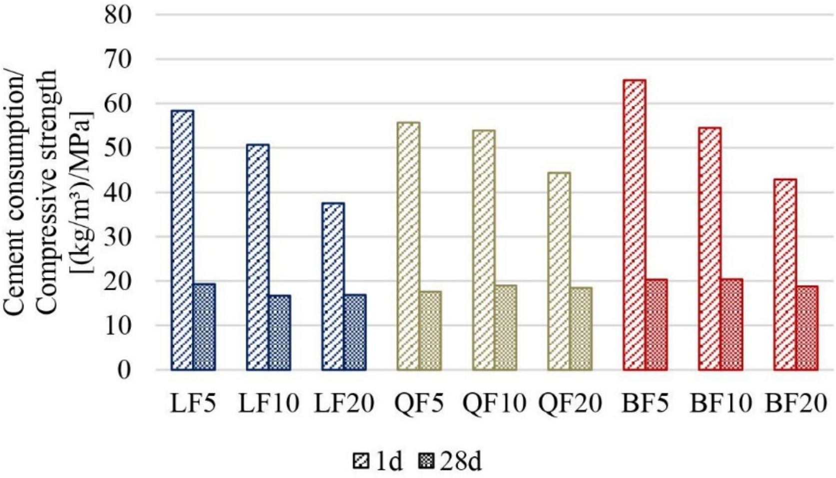

Figure 4. Relation between cement consumption and compressive strength of self-leveling mortars at 1 and 28 days.

It is possible to observe that, at the age of 1 day, the relationship between cement consumption and compressive strength is lower in SLMs with higher filler content. That is, for equivalent cement consumption, the results of compressive strength are higher in mortars with higher filler content, motivating the increase of filler content in these SLMs. At 28 days, the relationship between cement consumption and compressive strength was similar between the mixtures.

3.3 Dynamic modulus of elasticity

The dynamic modulus of elasticity values are reported in Figure 5.

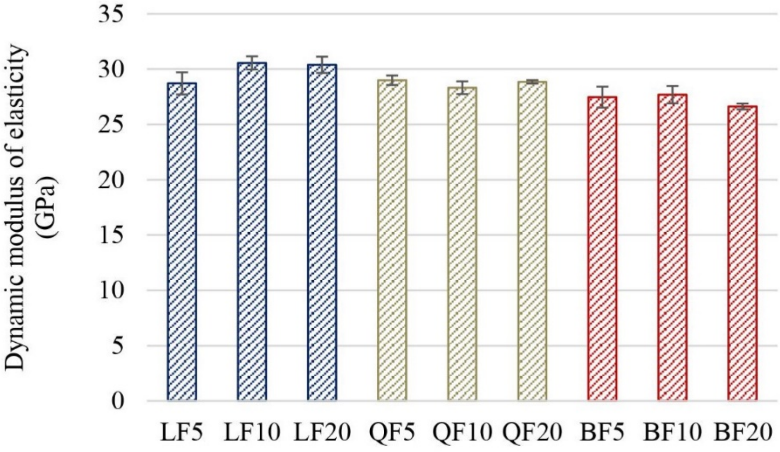

Figure 5. Dynamic modulus of elasticity of self-leveling mortars.

All the mixtures showed similar values. However, it is possible to observe that SLMs with LF showed dynamic modulus of elasticity slightly higher than other SLMs. This may be related to the higher compressive strengths of mixtures containing limestone filler.

3.4 Desorption isotherm

The desorption isotherms were developed for mortars with 10% and 20% of filler, since these mixtures showed better mechanical performance at early ages and allow higher incorporation of filler in the SLMs. Figure 6 shows the desorption isotherms of SLMs, where the points represent the points obtained experimentally and the lines represent the mathematical adjustment based on the GAB model.

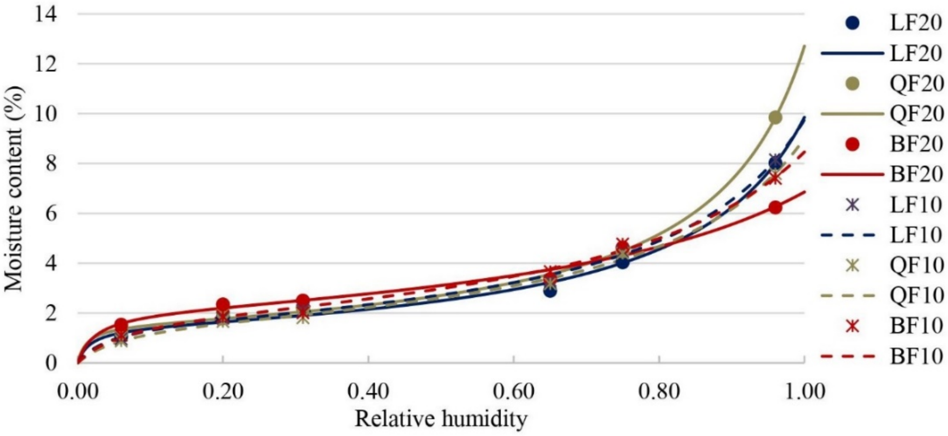

Figure 6. Desorption isotherms of self-leveling mortars with 10% and 20% of filler.

Figure 6 shows that the shape of the desorption curves was quite similar for all SLMs. The BF20 mixture showed higher moisture content in RH <65%, which may be seen by the curve shifted upwards in relation to the others. Table

3 shows the specific surface of the SLMs calculated from the GAB adjustment. It can be observed that the specific surface increases with the use of BF, with the sample BF20 having the highest SGAB.

Table 3. Monolayer moisture content (Wm) and specific surface area (S_{GAB}) calculated from the GAB model of self-leveling mortars with 10% and 20% of filler.

Mixture	Wm (%)	S_{GAB} (m^2/g)
LF20	1.46	48.85
QF20	1.54	51.46
BF20	2.09	69.77
LF10	1.68	56.18
QF10	1.66	55.49
BF10	1.98	66.19

According to Jennings et al. [28], water isotherms may be used to estimate the amount of water contained and removed from gel pores and capillary pores. In desorption, the size and quantity of the pores may be assessed by calculating the water lost in established RH intervals. At each interval, pores of a certain size are emptied and the amount of water lost represents the amount of pores of such dimension.

This is a simplified qualitative analysis to avoid calculating pore size distributions, which may be complicated and uncertain - the results vary depending on the assumed function for the thickness of the adsorbed layer (if the adsorption curve is used) and are underestimated due to the pore ink-bottle effect (desorption) [29], [30]. Based on Jennings et al. [28] and in Saeidpour and Wadsö [30], the amount of water desorbed (calculated from the mass moisture content measured) was divided into four intervals:

i. Between 100 and 85% RH, where capillary pores (width between ~8nm and ~10µm) are emptied;
ii. Between 85 and 50% RH, where large gel pores are emptied (~4nm);
iii. Between 50 and 25% RH, where small gel pores are emptied (~2nm);
iv. Between 25 and 0% RH, where the interlayer water is removed (spaces ≤ 2nm).

Figure 7 shows the amount of water desorbed in each described interval.

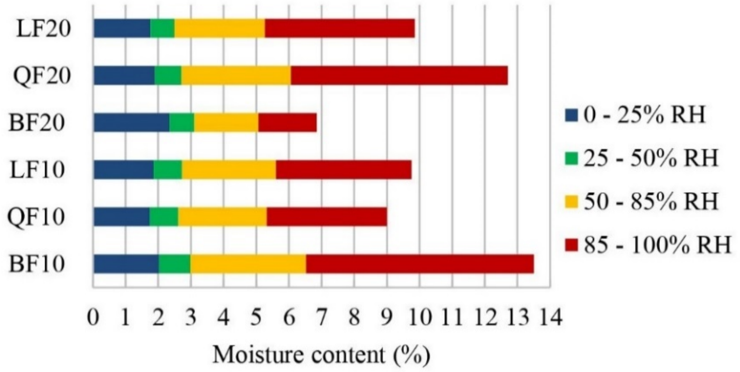

Figure 7. Amount of water desorbed in each RH interval for self-leveling mortars with 10% and 20% of filler.

At lower humidity intervals (50 - 25% RH and 25 - 0% RH), desorption occurs in the small gel pores (~2nm) and in the interlayer spaces of C-S-H (≤ 2nm). Based on Figure 7, it can be observed that between 50% and 0% RH mixtures containing BF have higher moisture content, when compared to mixtures with LF and QF, especially BF20. Therefore, these SLMs probably have a larger amount of smaller pores - small gel pores and interlayer spaces - consistent with the larger specific surfaces showed by these formulations.

The amount of large gel pores (85 - 50% RH) is greater in BF10 and QF20 mixtures. The amount of capillary pores is only an estimative (85 - 100% RH), since the complete filling of larger pores occurs only under conditions of immersion in water and immersion or saturation under vacuum and the larger pores are only covered by thin adsorbed layers [28]. However, it was observed that the BF20 mortar had significantly lower moisture content in this relative humidity range.

3.5 Drying shrinkage

The results of drying shrinkage and mass loss of SLMs at 3, 7, 14 and 28 days are shown in Figure 8. The shrinkage and mass loss values increased until 28 days and the increases were more evident in the first 14 days. There was no correlation between the mass loss and the drying shrinkage values. Shrinkage not only occurs due to the water loss to the external environment, but also depends on the size and type of voids that lose water (difficulty in removing water) and the mechanical properties of the material.

In SLMs developed with LF, the one with 10% of filler showed the lowest shrinkage value. In mixtures with QF, there is a decrease in shrinkage with increase of filler content up to 20%. Concerning mixtures with BF, the shrinkage decreases as the filler content increase up to 10%.

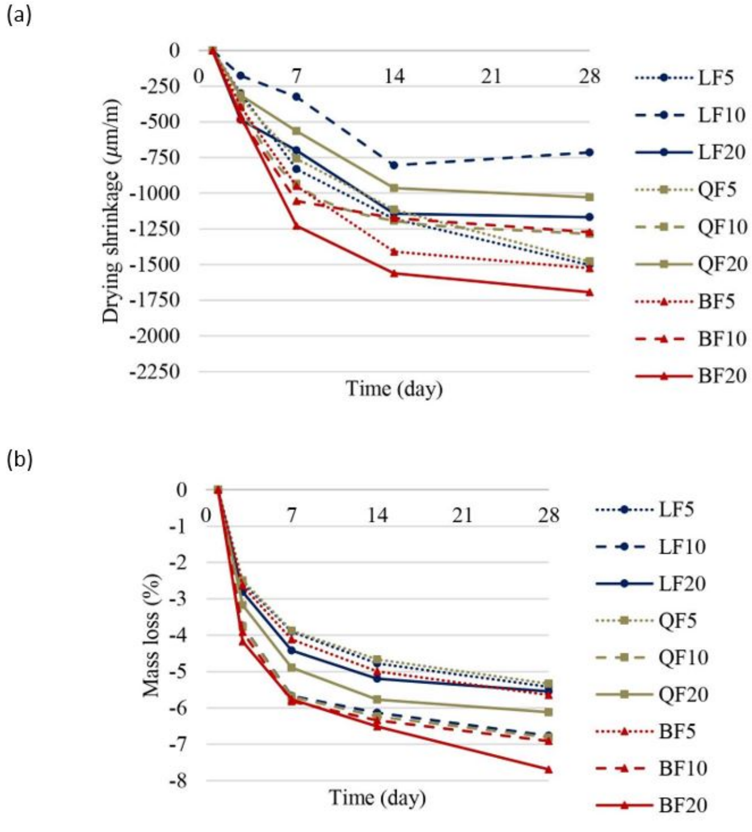

Figure 8. (a) Drying shrinkage and (b) mass loss of self-leveling mortars.

With the increase in filler content in the mixtures studied, the paste content (cement, filler and water) increases and the volume of sand decreases. However, the fillers seem to contribute to filling pores [12], being able to reduce the shrinkage of mortars, when used up to a certain content, which varies according to the filler. In addition, the decrease in shrinkage may also be due to the restraint in paste level provided by the fillers [13].

Regarding the type of filler, in mixtures developed with 5 and 10% of filler, there is no notable trend in the results. In general, the differences in shrinkage values of SLMs with the different fillers in these filler contents are not so prominent. In SLMs with 20% of filler, mixtures with BF showed greater shrinkage at 28 days (44% greater than LF and 64% greater than QF). Mixtures with the same filler content were developed with the same initial paste volume.

Therefore, the differences between shrinkage values may be related to the differences on the porous structure formed, to the deformation linked with filler nature and to the differences in the superplasticizer content.

Mortars with greater porosity tend to shrink more than those with less porosity, as long as they present similar pore size distributions. When the porosity is similar, the pore size distribution may still influence the shrinkage [13]. For drying above 50% RH, capillary stresses are the primary driving force for shrinkage [31]. Besides the lower moisture content verified in the capillary pores of BF20 mortar, compared to LF20 and QF20 (item 3.4), it showed the higher water loss. Moreover, it was found that the BF20 mortar (highest shrinkage) showed a lower mass loss/ drying shrinkage ratio at 28 days. This ratio was 47.5, 59.5 and 45.4, for LF20, QF20 and BF20, respectively. The lower this ratio, the higher the shrinkage for the same water loss, possibly due to the difficulty in removing water from the pores.

In addition, drying shrinkage may be related to the elastic deformation of the mortar (affected by the modulus of elasticity of the aggregates). In this context, mixtures produced with aggregate with low modulus of elasticity, result in higher shrinkage. Furthermore, it is believed that the higher shrinkage values observed when 20% of BF is used may also be associated with the higher superplasticizer content used (about twice the amount of SLMs with 20% of LF or QF). According to Ma et al. [32], the increase in polycarboxylate-based superplasticizer content may increase drying shrinkage of mortars. This occurs possible due to the increase in fine capillary pores.

3.6 Hydration kinetics by isothermal calorimetry

Figure 9a shows the heat flow curves and Figure 9b shows the cumulative heat curves during the first hours of pastes hydration, obtained from isothermal calorimetry. The peak of heat shown in Figure 9a occurs after the period of induction and corresponds to the period of acceleration, controlled by nucleation and the growth of hydration products.

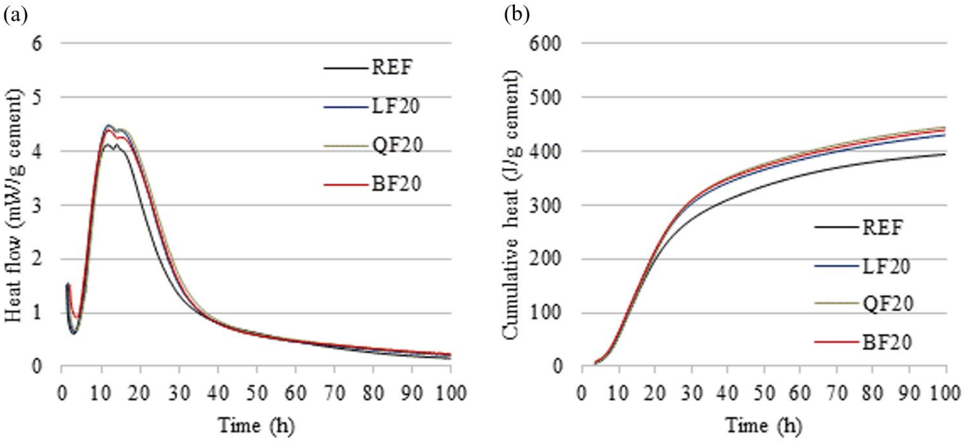

Figure 9. (a) Heat flow and (b) cumulative heat of LF20, QF20 and BF20 pastes.

From Figure 9a, it is possible to notice that pastes with filler show an increase in the heat flow when compared with pure cement paste (REF), probably by facilitating hydration reactions due to nucleation. In the LF paste was verified a slight increase in the maximum value of this peak, when compared with the QF and BF pastes. However, the curves of LF20, QF20 and BF20 pastes showed very close maximum values of heat flow, 4.48, 4.46 and 4.38mW/g cement, respectively. In addition, comparing the stretch that leads to the main peak between REF paste and the pastes with filler, there were no differences in the slope intensity, which indicates that there was no chemical effect of the fillers [33].

Figure 9b shows that the LF20, QF20 and BF20 pastes presented greater cumulative heat compared to the REF paste. At the end of the analyzed period, it is observed that the amount of heat released by the LF20 paste was lower than the QF20 and BF20 pastes (despite the greater peak presented by LF20, verified in Figure 5a). Thus, there is a decrease in the hydration kinetics of the paste with LF. However, pastes with filler did not show noticeable variations between them with relation to cumulative heat. After a hundred hours, the cumulative heat was 429.74, 444.42 and 439.07J/g cement for LF20, QF20 and BF20 pastes, respectively.

It is believed that the LF used may be considered as an inert filler [10]. Hence, it is assumed that the use of LF contributed to the development of the early strength due to its better physical effect caused by its inherent characteristics, providing stiffness to the mixture, and its particle size distribution in the mixture [34]. When the filler particle size decreases, filler particles are distributed more homogeneously in the cement paste and the whole mix tend to be more homogeneous, resulting in higher compressive strength [10].

5 CONCLUSIONS

Based on the results obtained, the following conclusions can be drawn:

- It is possible to incorporate up to 20% of limestone, quartz or crushed basalt filler in SLMs, achieving the desired fluidity;
- The use of limestone filler provided better mechanical performance at early ages and better response of dimensional stability when a maximum amount of 10% is added to SLM;
- Regarding SLMs produced with quartz filler, the compressive strength values were similar to those showed by SLMs containing limestone filler. Moreover, SLMs with quartz filler promoted the lowest shrinkage values when used 20% of filler;
- In SLMs with crushed basalt filler, it was verified a higher superplasticizer demand to achieve the target fluidity, especially when used 20% of filler. Despite of this, it was possible to achieve strength levels similar to those of SLMs with limestone and quartz filler. However, mortars containing crushed basalt filler exhibited the hightest shrinkage values (at 20%) and this can indicate a limitation of its use in practice;
- It was verified that limestone, quartz and basalt filler have potential for use in the development of self-leveling mortars and, in general, perform better when used in contents of 20%, 20% e 10%, respectively.

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