



ORIGINAL ARTICLE

Characterization of fresh and hardened state properties of grouts for use in structural masonry

Caracterização das propriedades do estado fresco e endurecido de grautes utilizados em alvenaria estrutural

José Augusto Ferreira Sales de Mesquita^a Mércia Maria Semensato Bottura de Barros^a Luiz Sérgio Franco^a Rafael Giuliano Pileggi^a Roberto Cesar de Oliveira Romano^a ^aUniversidade de São Paulo – USP, Escola Politécnica, São Paulo, SP, Brasil

Received 16 January 2023

Revised 19 April 2023

Accepted 24 April 2023

Corrected 27 March 2024

Abstract: Grout is a material usually applied to enhance the mechanical properties of structural masonry. As such, the demand for grout has been growing parallel to the demand for structural masonry. Without guidelines for grout mixing design, these products are often prepared on-site based on the same concepts used for preparing ordinary Portland cement-based concretes. Several grout properties still need to be investigated and understood due to this mixing-design approach, and consequently, the production of ecoefficient grouts is a secondary priority. Due to these obstacles, there is a demand for technical-scientific studies to aid with developing grout design guidelines. This work evaluates the rheological and hardened properties of grout compositions that simulate in-field conditions to show the inadequacy of the mixing design method and a way to understand this lack in the technology. The research investigates how the changes in the water-to-cement ratio, to increase the strength, would affect the fresh and hardened state properties of grouts for structural masonry. The grout's mixing was done in a rheometer which made it possible to determine the rheological behaviour and parameters during mixing and under different shear conditions. Compressive strength, porosity, and air-permeability were evaluated in the hardened state. The results showed that compositions with more cement content does not necessarily reflect enhanced mechanical properties since the differences were not statistically significant, because other variables were also changed. The research findings suggest that the design method used in practice may not be appropriate and potentially result in the waste of cementitious materials and no-eco-friendly compositions.

Keywords: grouts, structural masonry, rheology, compressive strength, hardened properties.

Resumo: O graute é um material normalmente aplicado para melhorar as propriedades mecânicas da alvenaria estrutural. Assim, a demanda pela utilização do grautes vem crescendo paralelamente à demanda por alvenaria estrutural. Sem diretrizes específicas para dosagem, esses produtos geralmente são preparados no local com base nos mesmos conceitos para preparo de concretos comuns. Várias propriedades do graute ainda precisam ser investigadas e compreendidas como ineficiência dos parâmetros de dosagem, resultando na produção de grautes não ecoeficientes. Desta forma, existe uma demanda por estudos técnico-científicos que auxiliem no desenvolvimento de diretrizes para dosagens de grautes. Este trabalho avalia as propriedades reológicas e do endurecidas de composições de grautes que simulam condições de campo para mostrar a inadequação do método de dosagem, demonstrando uma maneira de entender essa lacuna na tecnologia. A pesquisa investiga como as mudanças na relação água/cimento, para aumentar trabalhabilidade, afetariam as propriedades de estado fresco e endurecido de grautes para alvenaria estrutural. A mistura dos grautes foi realizada em um reômetro que permitiu determinar o comportamento e os parâmetros reológicos durante a mistura e em diferentes condições de cisalhamento. A resistência à compressão, a porosidade e a permeabilidade ao ar foram as propriedades avaliadas no estado endurecido. Os resultados mostraram que composições com maior

Corresponding author: José Augusto Ferreira Sales de Mesquita. E-mail: joseaugusto_mesquita@hotmail.com

Financial support: None.

Conflict of interest: Nothing to declare.

Data Availability: the data that support the findings of this study are available from the corresponding author, [JAFSM], upon reasonable request.

This document has an erratum: <https://doi.org/10.1590/S1983-41952024000200011>



This is an Open Access article distributed under the terms of the Creative Commons Attribution License, which permits unrestricted use, distribution, and reproduction in any medium, provided the original work is properly cited.

teor de cimento não necessariamente refletiram propriedades mecânicas melhores, uma vez que não houve alterações estatística, pois outras variáveis também foram alteradas. Os resultados da pesquisa sugerem que o método de projeto usado em obra pode não ser apropriado o que pode resultar no desperdício de materiais cimentícios e composições não ecoeficientes.

Palavras-chave: graute, alvenaria estrutural, reologia, estado endurecido, propriedades mecânicas.

How to cite: J. A. F. S. Mesquita, M. M. S. B. Barros, L. S. Franco, R. G. Pileggi, and R. C. O. Romano, "Characterization of fresh and hardened state properties of grouts for use in structural masonry," *Rev. IBRACON Estrut. Mater.*, vol. 17, no. 2, e17205, 2024, <https://doi.org/10.1590/S1983-41952024000200005>

1. INTRODUCTION

Grout is a kind of construction material used for many applications in civil engineering and can be defined as a fluid mortar or a low-viscosity micro-concrete used to fill voids, with maximum size aggregates (MSA) of 9.5 mm [1]–[4]. In addition to structurally enhanced masonry, other relevant grout applications include its use for stabilization of soils and rocks [5], for injection to fill cracks in concrete structures [6]–[9], for coating cables in precast constructions, to fill post-tensioning cable ducts, and soil stabilization near the tunnels.

Grout's employment is defined in the structural project, and its primary role, when used in structural masonry, is to solidify steel rebars within the masonry. Besides being associated with the bars, the grout may enhance masonry's structural resistance and provide stability to the ensemble [10]–[12].

Despite improper mixing design, grout use is considered an adequate solution to improve the mechanical properties of the structural masonry. To meet this goal successfully, structural masonry ensue needs to present some properties, as described [1], [2], [4], [13]–[15]:

- compatibility between the materials and the components (grout↔block↔steel);
- adequate adhesion between the concrete blocks and steel bars;
- controlled effects of shrinkage;
- slow rate of water absorption by the blocks;
- adequate flowability to fill in the block without any additional imposition of vibration;
- adequate mechanical strength (minimum of 15 MPa), depending on the place where it needs reinforcement.

However, the lack of Brazilian standards excludes some grout applications and products available in the marketplace, which vary according to the purpose of use, creating a deadlock for classifying this kind of cementitious component [16].

Even though there has been an increase in the number of publications on grout-like materials in recent years, that there is an attempt to make these components more eco-efficient and durable [3], [17]–[22], Tula and Oliveira [2] emphasized that studies on grout are still limited. Among typical grout constituent materials are chemical and mineral admixtures, most of them being pre-made mixtures available on the market.

In the field production of grout is still being done using the same concepts applied for ordinary concrete mixtures [23]: its mix design is based on increasing the cement content and reducing the water-to-cement ratio (w/c) when it is desired to increase the characteristic mechanical properties.

Therefore, there is a knowledge gap that needs to be overcome with scientific research for the development of more eco-efficient grout materials since changes in cement consumption and w/c, whether to increase strength or mitigate its adverse impacts on the environment, directly affect rheological behaviour and, consequently, the hardened properties of the product.

In this context, this work aims to evaluate the rheological and hardened properties of grout compositions proportioned on site (that is, without intervention from the concepts of dispersion or particle packing, or even using supplementary cementitious materials) as a function of cement. The concepts applied in this work can be extended for the development of eco-friendly compositions of cement, because the mix-design of raw material and the water content to produce concretes with good workability, needs to be in accordance with the rheological properties considering the kind of application. So, the strategy applied in this work can be replicable for other components.

2. RESEARCH METHOD

The methodology adopted in this research is intended to assess the quality of grouts proportioned on-site for application in different construction segments and to propose a new alternative for grout mixture proportion.

It should be noted that this is a case study referring to the common grout mixing practice of a specific construction company in the city of São Paulo. But it is well known that the method is a common practice used by several other construction companies to produce grout on-site.

The company provided the grout compositions to obtain of grouts for structural masonry with compressive strength of 15, 20, 25, and 30 MPa, primarily used in multi-floor buildings. The proportioning of composition provided by the company wasn't modify, and the raw materials were collected on-site and characterized as detailed below. The dosages were reproduced in the laboratory, with the monitoring of the rheological state properties and cast specimens for the hardened properties evaluation.

The results were discussed as a function of the binder consumption in the compositions and the mobility parameters of the particles. The mobility parameters were calculated from the particle size distribution, the volumetric surface area of the particles, and the volume of solids.

3. MATERIALS

3.1. Characterization of raw materials

The materials used in the compositions of grouts consisted of Brazilian Portland cement CII-Z (cement with up to 14% of pozzolan addition), natural quartz sand, and ground granite gravel. Before mixing, the aggregates were dried for 24 hours at 110 °C. A representative portion was then selected for characterization and mixing.

The density of aggregates and cement was determined by gas He pycnometry in Quantachrome equipment. The specific surface area was obtained using the BET method in Belsorp Max equipment, and unit mass was obtained using Brazilian standard (NBR 7810). The results obtained are shown in Table 1.

As expected, the specific surface area (SSA) of cement is greater than that of other raw materials due to the greater fineness of the binder. The SSA of crushed granite gravel was greater than that of natural sand due to the mineralogical composition of the rock and its high amount of fines from the grinding procedure. The measurement of SSA is essential for the mix design since it affects the water demand for adequate flow.

Table 1. Characterization of raw materials

Characterization of raw materials	Materials		
	Cement	Sand	Coarse aggregate
Specific gravity (g/cm ³)	2.99	2.65	2.65
Specific surface area (m ² /g)	1.14	0.60	0.90
Unit mass (g/cm ³)	1.04	1.32	1.41
D ₁₀ (µm)	2.36	300	6300
D ₅₀ (µm)	18.9	850	9500
D ₉₀ (µm)	63.0	2360	12500
Fineness modulus (%)	-	-	3.50

A Sympatec Qicpic laser granulometer was used to determine the particle size distribution of the binder, while for the aggregates, a Sympatec Helos with a dynamic image analyser was used. The particle size distribution of the grout constituents is shown in Figure 1. The results indicate that the cement presents ordinary particle size distribution with d50 of 20 µm.

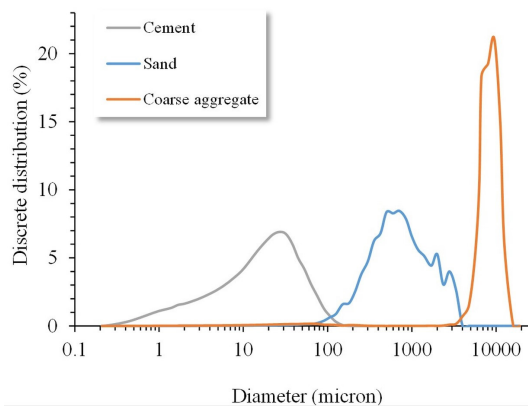


Figure 1. Particle size distribution on raw materials.

The quartz sand presents about 5% of its particles smaller than 106 μm and a maximum particle diameter of 2.7 mm. In contrast, the granite gravel presents a maximum particle diameter larger than 10 mm and an average diameter of around 7 mm.

3.2. Compositions evaluated.

Four grout compositions were evaluated with the proportions of raw materials shown in Table 2. A partner company provided the evaluated compositions: they were produced in the laboratory with no intervention by us. The only change, comparing with production in field, was the equipment used to mix. However, is common sense that the equipment can change from site to site. Each dosage corresponds to a mix-design done with the target of compressive strength class, estimated at 15, 20, 25, and 30 MPa.

Table 2. Compositions evaluated, in %-weight.

Composition	(MPa)	Cement consumption (kg/m ³)	Cement (%)	Sand (%)	Coarse aggregate (%)	Water (%)	w/c (weight)
G15	15	381	18.8	36.2	45.0	10.9	0.58
G20	20	410	20.2	34.8	45.0	10.9	0.54
G25	25	444	21.6	32.4	46.0	10.8	0.50
G30	30	495	24.0	30.0	46.0	10.8	0.45

All compositions were produced maintaining the same water-to-solids ratio. The particle size distribution for each composition is illustrated in Figure 2. As presented previously, for the target of increasing the mechanical strength, the strategy was to reduce the water-to-cement ratio; that is: to increase the strength, there is an increase in the amount of cement and a reduction in the amount of natural quartz sand. It deserves to be mentioned one more time that we tried to reproduce the in-field practice.

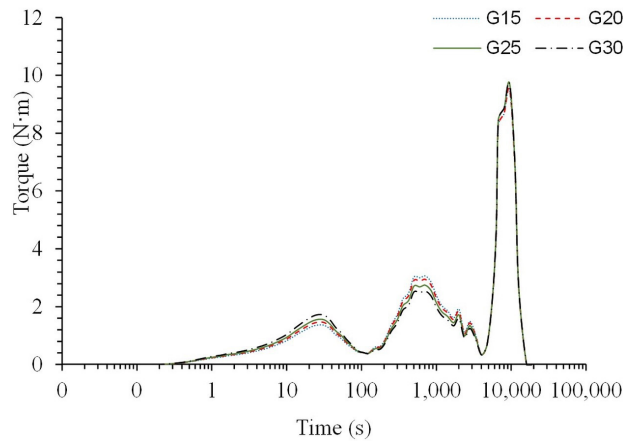


Figure 2. Particle size distribution of the different grout compositions.

The changes caused in particle mobility can be better explained applying the concepts of IPS (interparticle separation distance) and MPT (maximum paste thickness), as reported by Oliveira *et al.* [24], considering the VSA (*volumetric surface area*, i.e. the product between specific gravity and specific surface area), the packing porosity (P_{of} – calculated according to the Westman and Hugill concept [25]) and the solid volume (V_s). Equation 1 illustrates the calculation purposed and can be a way to summarize both physical contribution of particles only in one variable.

$$IPS/MPT = \frac{2}{VSA} \times \left[\frac{1}{V_s} - \left(\frac{1}{1-P_{of}} \right) \right] \tag{1}$$

These parameters (IPS/MPT) are particularly interesting for rheological evaluations, but can also be correlated with some properties in the hardened state [26]–[28]. The compilation of IPS, and MPT values is present in Table 3. While the first indicates the average separation distance between the particles that make up the cementitious paste (estimated with particles up to 106 μm), the MPT suggests the thickness of the paste layer that separates the aggregates. It is highlighted that both the IPS and the MPT shown in the table are average values, which can be changed depending on the function of particle size distribution.

Table 3. Mobility parameters of the compositions of grouts.

Parameters	Results for each composition			
	G15	G20	G25	G30
Interparticle separation distance – IPS (micra)*	0.70	0.66	0.61	0.56
Maximum paste thickness – MPT (micra)*	0.41	0.43	0.44	0.48

*Average value.

4. TEST METHODS

4.1. Fresh state properties: mixing and shear cycle

To evaluate the fresh state properties, all the dry powder was placed in the rheometer (Pheso – Calmetrix), the water was added with flow control of 90 g/s. The rheological evaluation was performed during the mixing stage maintaining the rotational speed of 125 rpm for 4 minutes. After this time, the shear cycle step was started to determine the torque profiles as a function of the changes in the shear condition. The protocol of the test used in both steps is shown in Figure 3. For the first step, we have information about mixing and with the results obtained in the shear cycle, we can understand the rheological behaviour and to have indicative of thixotropy, yield stress and viscosity of compositions [24].

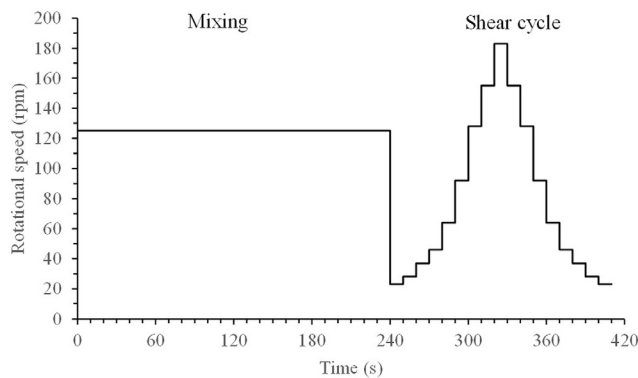


Figure 3. Mixing procedure used for the rheometry and shear cycle tests.

The products are commonly mechanically mixed in the field (concrete or mortar mixer machine) with no change in the applied energy. However, depending on the production demand, the same product is mixed with different equipment that provides different energies. Given the possibility of such variation, the evaluation of the mixing step is essential, and it is expected that the formulations need to be designed to be mixed with low energy to decrease the chance of variability of the applied product since the mixing step should homogenize and disperse the particles properly [10]. Additionally, mixtures that require less energy can increase productivity since they allow an increase in workability and, therefore, can supply the different work fronts more quickly.

4.2. Casting and curing of samples

For each composition of grout were produced 9 cylindrical specimens, with diameter of 50 mm and height of 100 mm, as established by ABNT NBR 7215:1996 [29]. The moulds were laid on the vibration table (MJ2 mol, brand MVL) and then filled 1/3 at a volume of each. Mechanical vibration was applied for 1 minute with 60% of the maximum

energy. This process of filling was repeated two more times. After the casting, the exposed surface was levelled with a metal spatula.

The moulds were maintained for 24 hours in a chamber with temperature control of $25 \pm 2^\circ\text{C}$ and relative humidity 50%, and after this period, the specimens were removed. Specimens were placed in a humid chamber with a temperature control of $25 \pm 2^\circ\text{C}$ and a relative humidity of 100% for 28 days.

After this period dure, the specimens were dried at $60 \pm 2^\circ\text{C}$ for 2 days to ensure that all the tests in the hardened state were carried out with no humidity. This strategy was chosen mainly due to the porosity and permeability tests.

4.3. Hardened state evaluation

The hardened state properties evaluated include the characterization of porosity, compressive strength, and air permeability, detailed follow. The porosity was quantified by the Archimedes immersion test [28]. The specimens were dried, weighed (m_s), and sequentially immersed in water for 24 hours. The vacuum was applied in the first 2 hours, keeping the pressure constant at 2 bars. After this time, the weight of each specimen was determined, both with the immersion (m_i) and wet samples (m_u - dried superficially with a damp cloth after being removed from the immersion). Open porosity (PA) was calculated according to the Equation 2, while the total porosity was calculated according to the Equation 3.

$$PA (\%) = \frac{m_u - m_i}{m_u - m_s} \times 100\% \quad (2)$$

$$PT (\%) = (1 - \rho_{REL}) \times 100\% \quad (3)$$

where PA is the apparent porosity, PT is the total porosity and ρ_{REL} is the relative density of dry powders composing the formulations. The compressive strength was determined on EMIC equipment (DL 10.000). The tests were carried out with loading control until rupture following the ABNT NBR 7215 [29] after the specimen surface grinding.

The air-permeability was quantified using the vacuum-decay technique [30], [31]. The specimens were coated with PVC film on the longitudinal cylindrical surface, leaving the upper and lower flat surfaces exposed to ensure unidirectional airflow. A suction cup with an opening diameter of 20 mm was positioned on the upper surface and sealed to prevent air loss during the test. The vacuum pump was activated until reaching the negative stable pressure, which was maintained for 30 seconds¹. Then, the pump was turned off, and the vacuum decay was monitored over time. The permeability constant (Darcian – k_1) was determined from Forchheimer equation (illustrated in Equation 4 [26]: the faster the pressure drop, the more permeable the material is, considering negligible air-compressibility and only taking into account the linear contribution ($\mu \cdot v_s / k_1$)).

$$\frac{\Delta P}{L} = \frac{\mu}{k_1} v_s + \frac{\rho}{k_2} v_s^2 \quad (4)$$

where L is the specimen thickness, ΔP is the pressure variation, μ is the fluid viscosity, ρ is the fluid density, v_s is the speed of air-percolation, k_1 and k_2 is the Darcian and non-Darcian permeability, respectively. It is worth noting that the first term ($\mu \cdot v_s / k_1$) represents the viscous effects of the fluid-solid interaction, while the second term ($\rho \cdot v_s / k_2$) accounts for the inertial effects (values of k_2 were not used in this paper).

¹Details about the equipment were reported by Sentone [31].

5. RESULTS AND DISCUSSION

5.1 Fresh state properties

5.1.1. Mixing stage

After contact with the water with the dry mix (powder), several surface phenomena are observed, resulting in a change in the consistency of the grains during mixing. Figure 4 is presented the graph that correlates the changes in torque with the shear time for each grout.

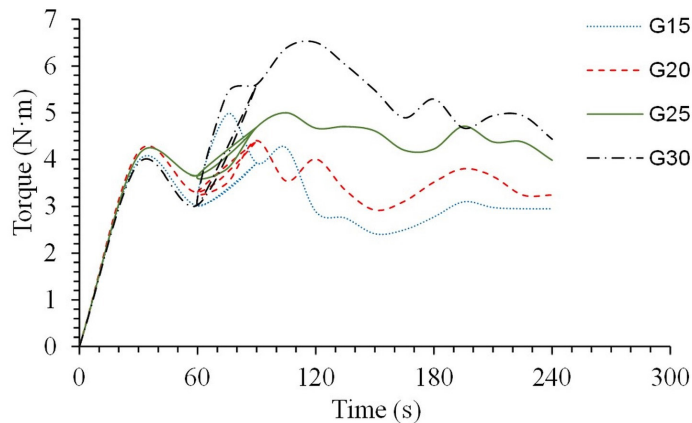


Figure 4. Torque variation as a function of time during the grout mixing.

The mixing torque shown follows the same trend for all strength ranges. There is an increase in torque after adding water caused by the rapid agglomeration of the particles due to the action of capillary and van der Waals attractions forces, trapping liquid inside.

With continuous shearing, the agglomerates are broken, and the water inside the agglomerates is released and used to recover the other neighbouring particles, separating them, and reducing the mixing torque. The final torque of mixing can give us an indicative of consistency of each grout.

The more significant the amount of cement, the longer the time needed to reach the turning point [32], and the greater torque at the end of the mixing, indicating the change in the flowability of each grout: the higher the amount of cement, the lower the flowability. This result was expected due to the dosage strategy based on increasing the number of fine particles to replace sand and the amount of fine material with greater SSA.

The calculated area under the curves of Figure 4 indicates an estimative of the energy needed for the adequate mixing of each grout. The results are shown in Figure 5, with the graph produced as the function of the consumption of binder in each composition [33]. This comparative evaluation is valid only for the same mixing conditions applied in this work.

It indicates that the dosage strategy employed resulted in considerable changes in the processing of the grouts: the highest the binder consumption, the highest the energy needed to mix, or in other words, the high-performance grout demands high energy to be mixed, and according to the results of Figure 4, the consistency is not so fluid like for the compositions with lower strength.

This is an essential information because the flowability of grout is qualitatively evaluated in the field and represents a stage where the decisions are taken. In practice, in the area is common to add more water to obtain similar flowability in the field, which can cause a decrease in the performance after hardening. As our target here is to reproduce what was performed in field, we did not apply any intervention to obtain the same workability for application.

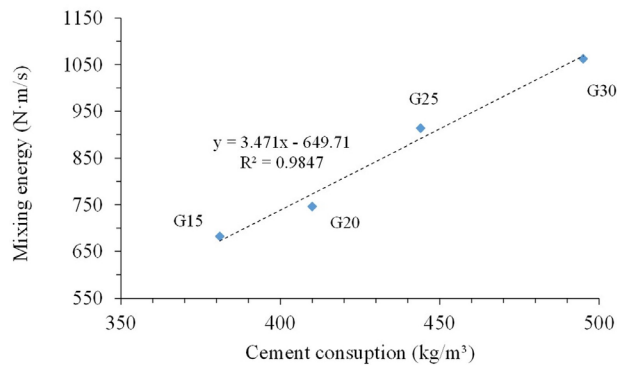


Figure 5. Mixing energy for grout processing as a function of the cement consumption.

The relationship with the mobility parameters of the particles shown in Figure 6 makes it possible to explain the changes in mixing energy: cement was the primary fine raw material used in the formulation and caused a considerable decrease in the IPS (the portion of sand with particle size below 106 μm also used for the IPS calculation).

The particle of binder tends to agglomerate from the moment that is in contact with water. Small changes in the mean separation distance between the particles result in an exponential increase in the van der Waals attractive forces [24]. As the dosage strategy to increase strength led to a decrease in the water-to-cement ratio (with an increase in binder consumption), there was a decrease in the IPS value. The use of IPS makes it possible to assess in a single variable the impact of changes in density, SSA, and particle size distribution of fine particles, in addition to the volume of solids in the composition.

In the case of the evaluated grouts, the SSA and the reduction of the w/c ratio governed the rheological properties from mixing by intensifying the agglomeration potential of the finer particles.

On the other hand, a different trend was observed considering average MPT; that is, the greater the average separation distance of the aggregates, the higher the mixing energy (Figure 6b). As is explained in literature, the friction forces affect considerably the flowability of concretes. While the IPS value decreases due to the reduction of water-to-cement ratio in the composition with the highest fgk, G30, the strategy of removing sand from the composition (a coarser material with lower VSA) and increasing the binder content (a finer material with higher VSA), results in high value of MPT using a paste with high tendency of agglomeration of particle. So, the mixability was governed by the IPS changes mainly to the higher interference between the finer particles and the high viscosity of paste [34].

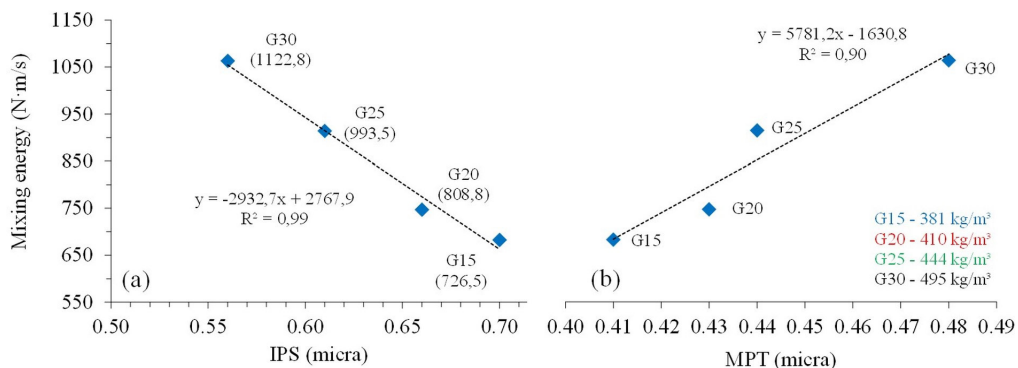


Figure 6. Relationship between mixing energy (a) with IPS and (b) with the MPT. The values displayed in parentheses in (a) represent the energy required to mix and the equivalent grout class.

5.1.2. Correlation between the torque and shear condition

During the rotational rheometry test, it was obtained the torque for each shear condition imposed. This makes it possible to quantify the yield torque (which can be associated with the resistance of the grout at the beginning of the flow, or yield stress), the hysteresis loop (which means the thixotropic potential of the composition) and the rheological behaviour. In the latter case, as seen in Figure 7, all grouts exhibited the shear thinning behaviour [17], suitable for applying the products

by pumping or simple casting. The lower yield torque level confirmed that it is possible to be used as a castable material with good flowability to fill the mould adequately. However, there was a trend toward increasing the absolute value of torque as a function of the increase in cement consumption, illustrating the impact of the dosage strategy.

As reported by Cunha [1] and Tula and Oliveira [2], the grouts must present, in addition to the compressive strength specified by the project, an adequate rheology property from the filling capacity of the block without being mechanically compacted. Therefore, the yield torque and viscosity of the compositions are important rheological parameters. Self-compacting compositions require low flow torque to facilitate fluidity and allow surface levelling without requiring other compaction techniques. It is necessary to have adequate viscosity to avoid phase separation during the flow (bleeding and segregation).

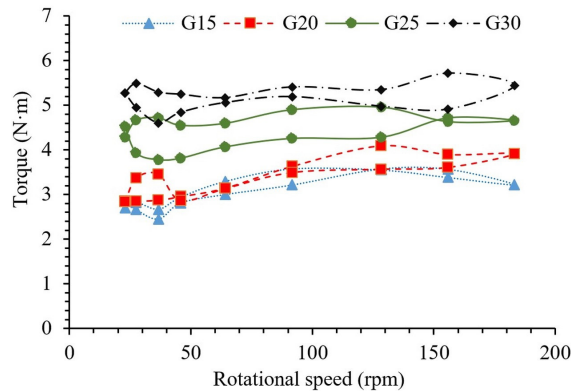


Figure 7. Shear profile (flow): relationship between torque and rotational speed for each grout.

As noted in Table 4, the greater the amount of cement in the composition (from G15 to G30), the greater the yield torque, resulting from the smaller separation distance between the fine particles and the consequent intensification of the surface forces. Compositions designed for high strength resulted in less fluid mixtures, which could result in filling failures while applying the grouts, but this is not the case of these materials, because even with the differences in the yield torque, the flowability was adequate.

Table 4. Rheological parameters

Sample	G15	G20	G25	G30
Cement consumption (kg/m ³)	381	410	444	495
Maximum torque (N·m)	4.97	5.09	5.78	6.51
Final mixing torque (N·m)	2.95	3.24	3.98	4.43
Mixing energy (N·m)	726	809	994	1123
Hysteresis loop	48.1	54.3	124.5	119.7
Yield torque (N·m)	2.86	2.84	4.52	5.28

On the other hand, the hysteresis loop indicates that the finer particles and the surface areas tend to cause agglomeration in the cementitious system, which, with the increase of shear condition, were broken during an acceleration step imposed in the test. However, with deceleration, a re-agglomeration rate is not as pronounced as the disruption of the agglomerates [33].

In this paper, the amount of water used for the mixture changed according to what had been indicated by the company responsible for the building. Thus, the changes observed in rheological properties were responses to the infield production method.

However, there is a search for the maintenance of workability, and it is practical to change the amount of water for this purpose, mainly when water-reducing admixtures such as superplasticizers or polyfunctional are not used. Therefore, the results obtained for the characteristics in the hardened state, despite serving as a parameter for comparison, to an ideal scenario, the variability in the field can be even more significant, including a decrease in the compressive strength due to a potential increase in water to maintain workability.

5.2. Hardening state properties

Figure 8 shows the results of porosity obtained, total and open. At the same time, Table 5 illustrates the statistical analysis of variance (one-way Anova) performed to define the significance of the comparative results.

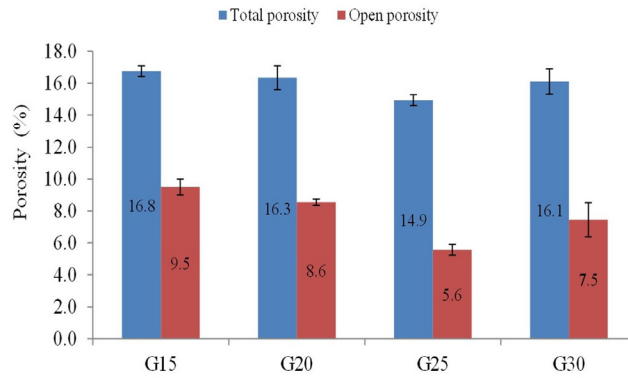


Figure 8. Total porosity and open porosity in the groups.

The p-value of the statistical evaluation concerns the proof value and indicates whether the variation in porosity is significant due to the change in cement consumption in the composition (or the variation in mechanical strength): the hypothesis of similarity must be accepted if the value of proof is greater than the established error of 5% (0.05). In the evaluated cases, the value was lower, indicating significant differences between the samples evaluated.

To confirm this, another way is to compare the value of F with F-critical: the F-critical limits the rejection region and means that for higher values of F, the similarity hypothesis must be rejected. Therefore, based on this concept, it can be confirmed that there are considerable differences in the porosity obtained in the compositions.

Table 5. Statistical analysis of the relationship between Fgk (cement consumption) and total (above) or open (below) porosity.

Total porosity				
Group	Score	Count	Average	Variance
15 MPa	4	66.99	16.75	0.12
20 MPa	4	65.36	16.34	0.56
25 MPa	4	59.75	14.94	0.08
30 MPa	4	64.41	16.10	0.43

ANOVA						
Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	7.24	3	2.41	8.15	0.0032	3.49
Within Groups	3.55	12	0.30			
Total	10.79	15				

Open porosity				
Group	Score	Count	Average	Variance
15 MPa	4	37.99	9.50	0.24
20 MPa	4	34.19	8.55	0.04
25 MPa	4	22.24	5.56	0.08
30 MPa	4	29.79	7.45	0.76

ANOVA						
Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	34.42	3	11.44	40.91	0.0000014	3.49
Within Groups	3.36	12	0.28			
Total	37.68	15				

When comparing cementitious products of the same strength class, porosity is the variable with the most significant impact on the results: the greater the number of pores, the lower the strength. However, when comparing compositions of different classes of strength only by increase of different cement consumptions, this fact isn't always true, as discussed below.

Table 6 shows the uniaxial compressive strengths for each class of mechanical strength (obtained for the grouts at 28 days of cure, in MPa), with deviations, amount of cement (kg/m³) and, as indicated by Damineli et al. [35], the binder index (BI, in kg/m³/MPa) for an illustrative evaluation of the eco-efficiency index of the grouts produced in work.

The results obtained were not related to the strength purposed by the project or to the total porosity of the specimens. Still, the coefficient of variation was considered low (up to 12%).

Table 6. Relation of the design compressive strength with that obtained in the laboratory.

Sample	f _{g28} (MPa)	Standard derivation	Cement consumption	BI*	ΔBI
			(kg/m ³)	(kg/m ³ /MPa)	(kg/m ³ /MPa)
G15	27.8	2.86	381	13.7	12.5-15.3
G20	34.2	3.23	410	12.0	10.9-13.2
G25	31.9	3.84	444	13.9	12.2-15.9
G30	37.8	3.00	495	13.1	12.0-15.4

*Binder index

The same statistical evaluation (using one-way Anova) was applied for the mechanical strength, indicating no significant differences between the compositions formulated with cement consumption between 381 kg/m³ (G15) and 495 kg/m³ (G30): F = 1.52 and F-critical = 3.49.

The eco-efficiency index of the evaluated grouts is considered inappropriate, illustrating that the dosage strategy is ineffective for that. Furthermore, although the average BI of the G20 composition was lower than in the other cases, it cannot be said that it is a grout with less environmental impact since the variation range was high, as in the other subjects. Likewise, there was no relationship between the BI and cement consumption.

As can be observed in Figure 9, the BI of grouts follows the same trend of ordinary Portland cement concretes as presented by Damineli et al. [35]: they were produced with Bis considerable high. The current common practice is below 7 kg/m³/MPa.

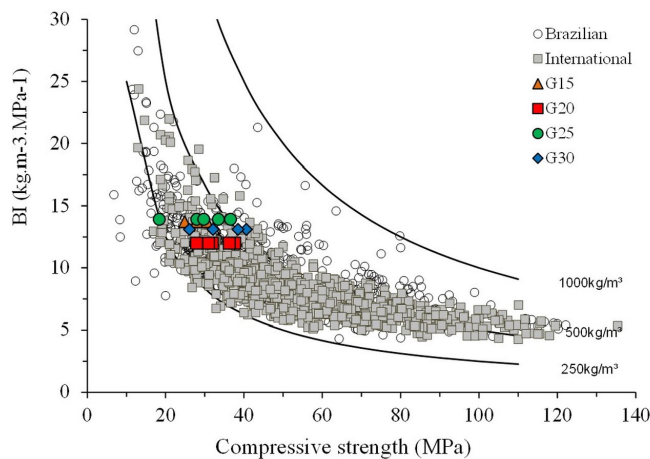


Figure 9. Relationship between compressive strength and binder consumption (BI) of grouts compared to ordinary concrete manufactured in Brazil and worldwide (adapted from Damineli et al. [35]).

This information illustrates the limited efficiency of the applied dosage method. However, the inefficiency of grouts was obtained because there were not used dispersing agents to reduce the amount of kneading water [27], [28], [34], [36], [37].

Compressive strength is a property related to the performance requirements of the material in use, but it does not illustrate any durability parameter. Therefore, the evaluation of air-permeability was used in this work: the more

susceptible to penetration of degrading agents, the shorter the useful life of the cementitious component, especially in cases exposed to aggressive agents. This parameter is less important when dealing with grouts for structural masonry usually confined in masonry septa. Even so, the results obtained were correlated with cement consumption and thickness of the paste layer that separates the aggregates (MPT), as illustrated in Figure 10a and 10b, respectively.

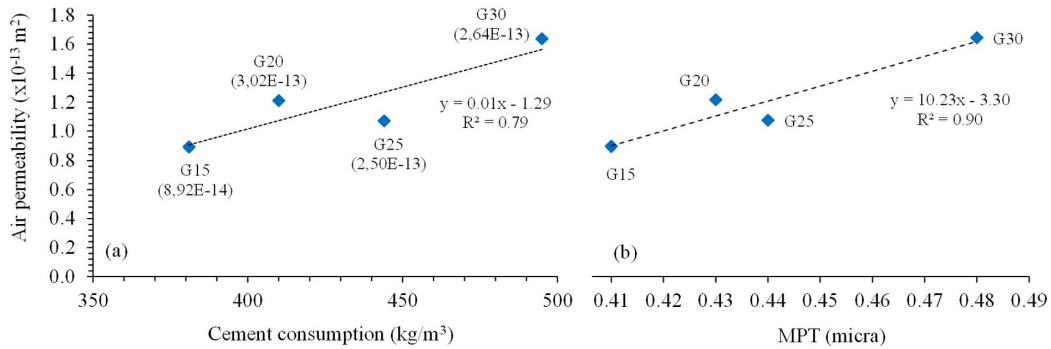


Figure 10. Air permeability as a function of (a) cement consumption and (b) thickness of the layer of paste separating the aggregates (MPT). The values showed in parentheses in (a) express the Dacyan's permeability and the fgk of each composition.

With the increase in cement consumption, an increase in the air-permeability was obtained, which is divergent from what is frequently found in the literature [38]. However, in this work, the comparison was made between grouts of varying class of strength, keeping the dosing strategy applied in field.

Therefore, it can be stated that this strategy did not result in statistical changes in the compressive strength; the use of a high amount of cement also did not result in a decrease in permeability and, consequently, an increase in the durability of the richer material is not expected: the increase in the binder consumption also increased the water content.

So, the correlation with the MPT result may be the correct explanation for this apparent inconsistency: the paste thickness increased with the increase in cement content, resulting in a more significant amount of permeable channels (connected pores) and, consequently, the permeability increases, with a greater probability of percolation of air or degrading agents of the cementitious product [39].

6. CONCLUSIONS

The results obtained with the experimental program allowed us to state that the strategy of dosage used to produce grouts in field had negative impacts on all properties evaluated.

The rheological properties had a good correlation with the amount of cement: the high the class of strength, the higher the yield torque. This is associated with the attraction forces between the particles and the formation of agglomerates.

The results also showed that it is required more energy to mix the grouts with a high amount of cement. This is a negative aspect because although this stage is carried out in-field using mixers, the lower the energy required, the shorter the time needed for mixing; that is, the grout more quickly combines from desired homogeneity and consistency, increasing, with this, the productivity of this activity.

For the compressive strength, the strategy of dosage did not make possible to differentiate the specified class of strength statistically; that is, the increase in binder consumption did not change the compressive strength in the same proportion.

The most significant amount of cement did not lead to a decrease in air permeability either; on the contrary, it has increased, and this can affect the durability of components, especially in situations where they are exposed to the environmental conditions of using. In this case, it was explained by the volume of paste that separates the aggregates and the creation of permeable channels of pores.

Thus, it can be said that the strategy of dosage used can cause negative social, environmental, and economic impacts since the grouts are obtained with a high amount of binder; that is, without and an adequate (scientific) development of the proportion between the materials, including the water. There was, therefore, a waste of essential and scarce resources.

The results leave no doubt that there is a need for scientific/technological development for this kind of material, that is being consumed more and more: it is necessary to invest in research that allows to purpose using of correct mix design and adequate dispersion of particles. This can also allow us to produce the same components with less variability, less cement consumption and eco-friendly.

In summary, the amount of cement used was high for the class mechanical strength purposed in the field, illustrating the poor efficiency of method of dosage applied in this construction. An alternative to reduce amount of cement and improve the eco-efficiency of this kind of product could be adopt the concepts of packing and dispersion of particles. The choice of adequate raw materials to partially replace Portland cement in the compositions, associated to the correct chemical stabilization, allow us to obtain better performance in the fresh and hardened state even with reduced amount of binder. So, the strategy of dosage evaluated in this case study can be improved.

6. ACKNOWLEDGEMENTS

The authors are thankful to Capes – Coordenação de Aperfeiçoamento de Pessoal de Nível Superior, FDTE – Fundação para o Desenvolvimento Tecnológico da Engenharia, and LME – Laboratório de Microestrutura e Ecoeficiência de Materiais, for their financial support during the work.

7. REFERENCES

- [1] E. H. Cunha, “Análise experimental do comportamento de prismas grauteados em alvenaria estrutural,” Master thesis, Univ. Fed. Goiás, Goiânia, GO, Brazil, 2001.
- [2] L. Tula, P. S. F. Oliveira, and P. Helene, “Grautes para reparo”, in *VII Congreso Latinoamericano de Patología de la Construcción*, 2003, pp. 139–152.
- [3] M. Vasumithran, K. B. Anand, and D. Sathyan, “Effects of fillers on the properties of cement grouts,” *Constr. Build. Mater.*, vol. 246, pp. 118346, Jun. 2020, <http://dx.doi.org/10.1016/j.conbuildmat.2020.118346>.
- [4] J. S. Camacho, B. G. Logullo, G. A. Parsekian, and P. R. N. Soudais, “The influence of grouting and reinforcement ratio in the concrete block masonry compressive behavior,” *Rev. IBRACON Estrut. Mater.*, vol. 8, pp. 341–364, Jun. 2015, <http://dx.doi.org/10.1590/S1983-41952015000300006>.
- [5] U. Müller, L. Miccoli, and P. Fontana, “Development of a lime based grout for cracks repair in earthen constructions,” *Constr. Build. Mater.*, vol. 110, pp. 323–332, May 2016, <http://dx.doi.org/10.1016/j.conbuildmat.2016.02.030>.
- [6] M. M. S. Lacerda, T. J. Silva, G. M. S. Alva, and M. C. V. Lima, “Influence of the vertical grouting in the interface between corbel and beam in beam-to-column connections of precast concrete structures – an experimental analysis,” *Eng. Struct.*, vol. 172, pp. 201–213, Oct. 2018, <http://dx.doi.org/10.1016/j.engstruct.2018.05.113>.
- [7] N. Tullini and F. Minghini, “Grouted sleeve connections used in precast reinforced concrete construction – experimental investigation of a column-to-column joint,” *Eng. Struct.*, vol. 127, pp. 784–803, Nov. 2016, <http://dx.doi.org/10.1016/j.engstruct.2016.09.021>.
- [8] G. Sipp and G. A. Parsekian, “Investigation of the adherence between clay blocks and grouts,” *Rev. IBRACON Estrut. Mater.*, vol. 15, pp. e15112, Aug. 2021, <http://dx.doi.org/10.1590/S1983-41952022000100012>.
- [9] O. S. Izquierdo, M. R. S. Corrêa, and I. I. Soto, “Study of the block/grout interface in concrete and clay block masonry structures,” *Rev. IBRACON Estrut. Mater.*, vol. 10, pp. 924–936, Aug. 2017, <http://dx.doi.org/10.1590/S1983-41952017000400009>.
- [10] Associação Brasileira de Normas Técnicas, *Alvenaria Estrutural - Parte 1: Projeto*, ABNT NBR 16868-1, 2020.
- [11] S. Calderón, L. Vargas, C. Sandoval, and G. Araya-Letelier, “Behavior of partially grouted concrete masonry walls under quasi-static cyclic lateral loading,” *Mater.*, vol. 13, pp. 2424, May 2020, <http://dx.doi.org/10.3390/ma13102424>.
- [12] M. Bolhassani, A. A. Hamid, and F. L. Moon, “Enhancement of lateral in-plane capacity of partially grouted concrete masonry shear walls,” *Eng. Struct.*, vol. 108, pp. 59–76, Feb. 2016, <http://dx.doi.org/10.1016/j.engstruct.2015.11.017>.
- [13] G. A. Parsekian, “Tecnologia de produção de alvenaria estrutural protendida,” Doctoral dissertation, Esc. Polit., Univ. São Paulo, 2002.
- [14] K. S. Alotaibi, A. B. M. S. Islam, and K. Galal, “Axial performance of grouted C-shaped concrete block masonry columns jacketed by carbon and glass FRP,” *Eng. Struct.*, vol. 267, pp. 114698, Sep. 2022, <http://dx.doi.org/10.1016/j.engstruct.2022.114698>.
- [15] S. Noor-E-Khuda and D. P. Thambiratnam, “In-plane and out-of-plane structural performance of fully grouted reinforced masonry walls with varying reinforcement ratio – a numerical study,” *Eng. Struct.*, vol. 248, pp. 113288, Dec. 2021, <http://dx.doi.org/10.1016/j.engstruct.2021.113288>.
- [16] N. Dirceu, “Graute microconcreto ou argamassa,” *Rev. Tecnol. Constr.*, vol. 240, pp. 16–23, Mar 2017.
- [17] A. Bras, R. Gião, V. Lúcio, and C. Chastre, “Development of an injectable grout for concrete repair and strengthening,” *Cement Concr. Compos.*, vol. 37, pp. 185–195, Mar. 2013.
- [18] M. K. Mohan, R. G. Pillai, M. Santhanam, and R. Gettu, “High-performance cementitious grout with fly ash for corrosion protection of post-tensioned concrete structures,” *Constr. Build. Mater.*, vol. 281, pp. 122612, Apr. 2021, <http://dx.doi.org/10.1016/j.conbuildmat.2021.122612>.
- [19] C. A. Anagnostopoulos, “Effect of different superplasticisers on the physical and mechanical properties of cement grouts,” *Constr. Build. Mater.*, vol. 50, pp. 162–168, Jan. 2014, <http://dx.doi.org/10.1016/j.conbuildmat.2013.09.050>.

- [20] T. S. Krishnamoorthy, S. Gopalakrishnan, K. Balasubramanian, B. H. Bharatkumar, and P. R. M. Rao, "Investigations on the cementitious grouts containing supplementary cementitious materials," *Cement Concr. Res.*, vol. 32, no. 9, pp. 1395–1405, Sep. 2002, [http://dx.doi.org/10.1016/S0008-8846\(02\)00799-8](http://dx.doi.org/10.1016/S0008-8846(02)00799-8).
- [21] M. Shamsuddoha, G. Hüsken, W. Schmidt, H.-C. Kühne, and M. Baeßler, "Ternary mix design of grout material for structural repair using statistical tools," *Constr. Build. Mater.*, vol. 189, pp. 170–180, Nov. 2018, <http://dx.doi.org/10.1016/j.conbuildmat.2018.08.156>.
- [22] A. Teymen, "Effect of mineral admixture types on the grout strength of fully-grouted rockbolts," *Constr. Build. Mater.*, vol. 145, pp. 376–382, Aug. 2017, <http://dx.doi.org/10.1016/j.conbuildmat.2017.04.046>.
- [23] M. M. M. Soares, "Especificação, execução e controle de alvenaria estrutural em blocos cerâmicos de acordo com a NBR 15812," Master thesis, Univ. Fed. São Carlos, São Carlos, SP, Brazil, 2012.
- [24] I. R. Oliveira, A. R. Studart, R. G. Pileggi, and V. C. Pandolfelli, *Dispersão e Empacotamento de Partículas: Princípios e Aplicações em Processamento Cerâmico*. São Paulo, Brazil: Fazendo Arte, 2000.
- [25] A. E. R. Westman and H. R. Hugill, "The packing of particles," *J. Am. Ceram. Soc.*, vol. 13, pp. 767–779, Oct. 1930, <http://dx.doi.org/10.1111/j.1151-2916.1930.tb16222.x>.
- [26] R. C. O. Romano, "Incorporação de ar em materiais cimentícios aplicados em construção civil," Doctoral dissertation, Univ. São Paulo, São Paulo, SP, Brazil, 2013, <https://doi.org/10.11606/T.3.2013.tde-27122013-113747>.
- [27] B. L. Daminieli, "Conceitos para formulação de concretos com baixo consumo de ligantes: controle reológico, empacotamento e dispersão de partículas," Doctoral dissertation, Univ. São Paulo, São Paulo, SP, Brazil, 2013, <https://doi.org/10.11606/T.3.2013.tde-19092014-103459>.
- [28] R. C. O. Romano et al., "Impact of using bauxite residue in microconcrete and comparison with other kind of supplementary cementitious material," in *Proc. 35th Int. ICSOBA Conf.*, Hamburg, Germany, 2017, pp. 505–518.
- [29] Associação Brasileira de Normas Técnicas, *Cimento Portland - Determinação da Resistência à Compressão de Corpos de Prova Cilíndricos*, ABNT NBR 7215, 1996.
- [30] R. J. Torrent and G. Frenzer, *Methods for Measuring and Assessing the Characteristics of Concrete Cover on Site*. Zürich, Switzerland: Office Fédéral des Routes Zürich, 1995. Rep. 516.
- [31] D. T. Sentone, "Desenvolvimento de método para medida de permeabilidade superficial de revestimentos de argamassa," Master thesis, Univ. São Paulo, São Paulo, SP, Brazil, 2011, <https://doi.org/10.11606/D.3.2011.tde-11082011-123629>.
- [32] M. H. Maciel, H. M. Bernardo, G. S. Soares, R. C. O. Romano, M. A. Cincotto, and R. G. Pileggi, "Effect of cement consumption on rendering mortars produced according the concepts of packing particles and mobility," *Ambient. Constr.*, vol. 18, pp. 245–259, Jan./Mar. 2018, <http://dx.doi.org/10.1590/s1678-86212018000100219>.
- [33] D. R. Torres, R. C. O. Romano, R. G. Pileggi, D. R. Torres, R. C. O. Romano, and R. G. Pileggi, "Influência da variação da velocidade de rotação e do tipo de cimento nas propriedades de argamassas de revestimento nos estados fresco e endurecido," *Cerâmica*, vol. 63, pp. 508–516, Oct./Dec. 2017, <http://dx.doi.org/10.1590/0366-69132017633682193>.
- [34] V. Morin, F. C. Tenoudji, A. Feylessoufi, and P. Richard, "Superplasticizer effects on setting and structuration mechanisms of ultrahigh-performance concrete," *Cement Concr. Res.*, vol. 31, pp. 63–71, Jan. 2001, [http://dx.doi.org/10.1016/S0008-8846\(00\)00428-2](http://dx.doi.org/10.1016/S0008-8846(00)00428-2).
- [35] B. L. Daminieli, F. M. Kemeid, P. S. Aguiar, and V. M. John, "Measuring the eco-efficiency of cement use," *Cement Concr. Compos.*, vol. 32, pp. 555–562, Sep. 2010, <http://dx.doi.org/10.1016/j.cemconcomp.2010.07.009>.
- [36] C. Varhen, I. Dilonardo, R. C. O. Romano, R. G. Pileggi, and A. D. Figueiredo, "Effect of the substitution of cement by limestone filler on the rheological behaviour and shrinkage of microconcretes," *Constr. Build. Mater.*, vol. 125, pp. 375–386, Oct. 2016, <http://dx.doi.org/10.1016/j.conbuildmat.2016.08.062>.
- [37] M. S. Rebmann, "Robustez de concretos com baixo consumo de cimento Portland: desvios no proporcionamento e variabilidade granulométrica e morfológica dos agregados," Doctoral dissertation, Univ. São Paulo, São Paulo, SP, Brazil, 2017, <https://doi.org/10.11606/T.3.2017.tde-20012017-090400>.
- [38] M. H. Maciel, G. S. Soares, R. C. O. Romano, and M. A. Cincotto, "Monitoring of Portland cement chemical reaction and quantification of the hydrated products by XRD and TG in function of the stoppage hydration technique," *J. Therm. Anal. Calorim.*, vol. 136, pp. 1269–1284, Sep. 2019, <http://dx.doi.org/10.1007/s10973-018-7734-5>.
- [39] R. C. O. Romano, D. R. Torres, and R. G. Pileggi, "Impact of aggregate grading and air-entrainment on the properties of fresh and hardened mortars," *Constr. Build. Mater.*, vol. 82, pp. 219–226, May 2015, <http://dx.doi.org/10.1016/j.conbuildmat.2015.02.067>.

Author contributions: JAFSM: development of practical activities, treatment and discussion of results and writing of the paper; MMSBB: discussion of results and review of the paper; LSF: paper review; RGP: discussion of results and review of the paper; RCOR: discussion of the experimental procedure, support in the treatment and discussion of results and review of the paper.

Editors: Vladimir Haach, Guilherme Aris Parsekian.