Experimental Developments of the Squeeze Flow Test for Mortars

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Abstract. Squeeze flow rheological technique is based on the compression of a cylindrical sample between parallel plates and is used to determine the flow properties of food, pharmaceuticals, composites, suspensions, including cementitious materials. This work summarizes the main experimental developments on squeeze-flow for the evaluation of mortars that have been performed in Brazil for the last 15 years and supported the creation of the Brazilian standard test method applied to rendering and masonry mortars (ABNT NBR 15839:2010). The paper exemplifies possible test setups (configuration, geometry, velocity, roughness) for different types of mortars and situations, the use of porous substrate (ceramic or concrete blocks) as the bottom plate, and a method to measure phase separation induced by the. A complementary instrumentation (interfacial pressure mapping) for the assessment of pressure evolution during flow is also presented – forming the pressure mapped squeeze flow method (PMSF) – which allows for identification of transitions in flow type and localized pressure peaks resulting from microstructural changes like particle jamming. The technique was also employed to study and develop laboratory mixing methods for mortars, as the resulting flow curves are very sensitive to the material's agglomeration state. Finally, rheological parameters of mortars by squeeze-flow and rotational rheometry were compared showing that yield stress have some degree of agreement, whereas viscosity values from these techniques are complex to be related.

Keywords: Mortar · Squeeze flow · Phase separation · Pressure distribution · Mixing

1 Introduction

Inorganic mortars, besides being a fundamental portion of concretes, are also products widely used for many functions. The rheological requirements associated with the various uses and processing combinations are diverse as well [1]. In this sense, squeeze-flow testing has been applied as an alternative/complementary technique to assess the flow behavior of building materials. Besides its ease of implementation, the geometry change during gap reduction makes the method particularly interesting, as it creates flow conditions similar to those involved in processing and application of

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mortars like pumping, spreading, finishing, squeezing between bricks, extrusion and more recently 3D printing processes [1–4]. The method is very versatile allowing various geometric configurations, plate roughness, gaps and displacement rates to better simulate diverse technological situations [2, 5]. During squeeze, suspensions may undergo phase separation depending on material's susceptibility and flow conditions [1, 2]. This phenomenon is crucial as it affects localized solid content and consequently the rheological behavior of mortars even in a more complex way, since they are multiphasic heterogeneous materials with micro and macroscopic particles. Therefore, phase separation should be considered for a better interpretation of squeeze flow behavior [1, 2], preferably with complementary techniques that can provide information regarding this associated effect. The present paper aims to highlight a group of developments about the test for mortars that improved experimental possibilities and comprehension of some phenomena closely related to this type of flow; plus, a practical use of the test for evaluation and development of mixing methods. Most results present in this work are of factory-produced rendering mortars (Brazilian or European) [1, 5–8] and a few are of mortars formulated in the university's laboratory [2, 6]. Further details of mortars' features and methods can be found in the references. All squeeze flow tests were displacement-controlled with constant velocity (0.1 mm/s or 3 mm/s).

2 Squeeze Flow Configurations and Plate Roughness

Figure 1 shows the two main geometric configurations employed: (a) constant area and (b) constant volume. The first has the advantage of maintaining a constant surface area of the material in contact with the moving top plate, facilitating stress determination, but susceptible to edge effects when the material is extruded outwards. On the other hand, in the constant volume configuration the material is always in between the plates with no edge effects other than the boundary of the spreading sample. However, determination of stress must always be performed, which for mortars may not only be a matter of volume conservation since entrained air is compressible and can even collapse; in addition diameter/height ratio changes more significantly throughout the test (when compared to CA), which can induce transition of flow type – from elongational to shear – because this ratio is one the main parameters that influences the flow [1, 5, 7, 8]. Nevertheless, both configurations are useful and have some degree of similarity with processing and application conditions of mortars and other cement-based materials.

In Fig. 2 the influence of the geometric configuration can be seen both with smooth and rough plates. For the same displacement, normal stress tends to be higher for CA than for CV, since for the first, diameter/height ratio is higher which induces more shear and overall resistance to flow. Most curves of this particular mortar are characterized by a strain-hardening behavior at small displacements, with absence of a more pronounced viscous or plastic behavior stage [1]. At 3 mm/s (graphic b), final displacements achieved (test with 1000 N) are larger than those at 0.1 mm/s because liquid-solid phase separation (filtration phenomenon) is more intense at slower rates. Therefore, CV_smooth curve displays some viscous or plastic stage. The other main

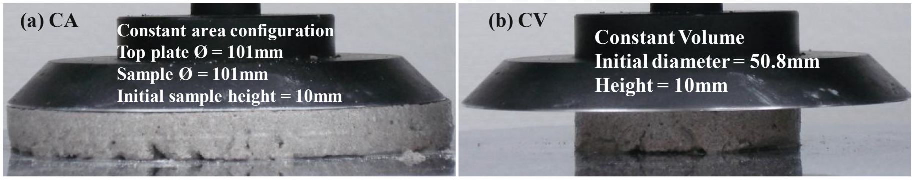

Fig. 1. Squeeze-flow configurations: (a) Constant area a 101 mm top plate; (b) Constant volume with a 101 mm top plate and sample initial diameter of 50.8 mm. In both cases, bottom plate was 200 mm in diameter (adapted from [5]).

parameter that affects the predominant flow type is plate roughness: the higher the friction at the interface, more shear flow; whereas, more slip results in more elongation. This is the reason why squeeze flow curves with rough plates (SiC emery paper) have higher stresses and smaller final displacements [5]. The manifestation of strain-hardening at such large gaps (small displacements) when compared to the maximum particle size of the mortar (2 mm) is caused by phase separation that increases solid content in the central region [1, 2, 5, 6]. These results from the variations of configuration and interfacial roughness, indicate that shear induced during squeeze flow of mortars was associated to phase separation in this case. Confirmation was obtained by phase separation experimental data from [5] measured as described in Fig. 4.

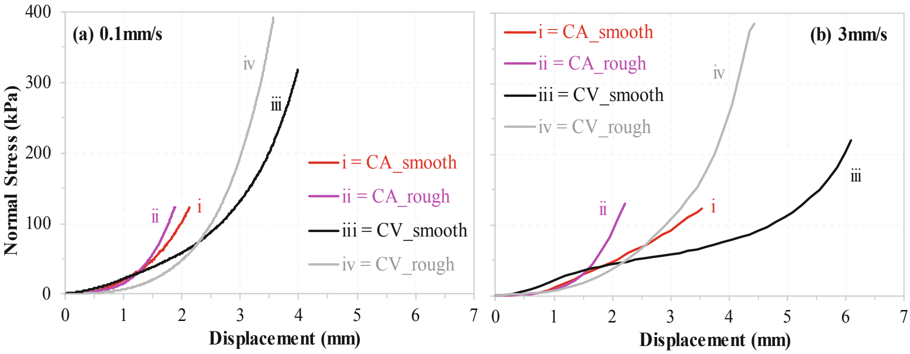

Fig. 2. Squeeze-flow results of mortar Alfa with constant area (CA) and constant volume (CV) using smooth metallic plates and emery paper. (a) 0.1 mm/s; (b) 3 mm/s (adapted from [5]). For CV stress, nominal constant volume of the samples was considered.

3 Assessment of Pressure Distribution and Phase Separation

Pressure distribution at mortar-plate interfaces was evaluated by a dynamic pressure mapping system (I-Scan, Tekscan Inc.) [2]. Figure 3 shows the evolution of pressure distribution of two similar mortars, except for the use of MHEC (methyl hydroxyethyl cellulose ether admixture - Tylose 10012P6) in mortar coded as CE. This technology provided evidence of phase separation effects on the squeeze flow behavior, proving

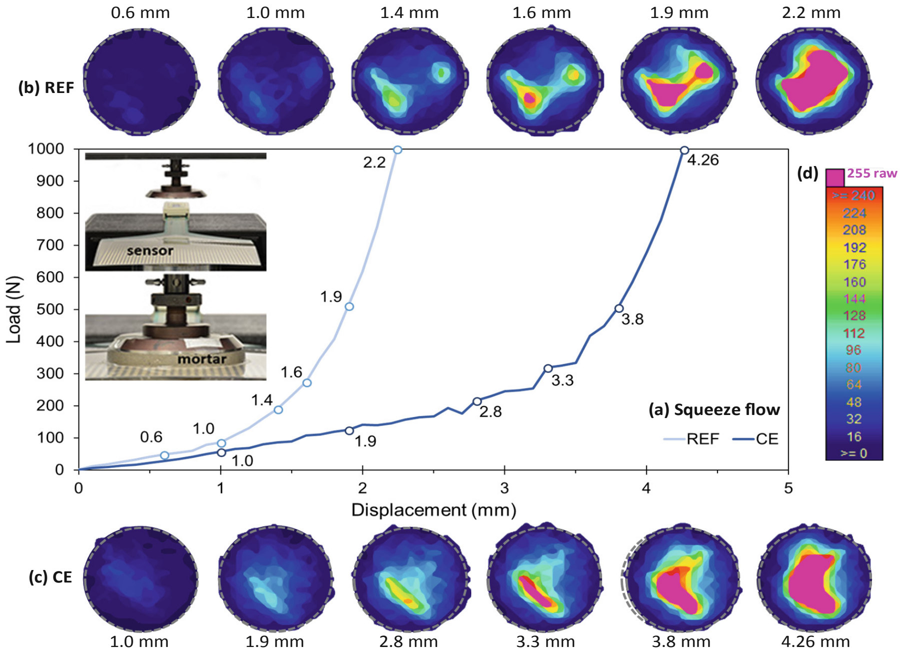

Fig. 3. Squeeze-flow and pressure distribution results of mortars REF (without MHEC cellulose ether) and CE (with MHEC) tested at 0.1 mm/s (adapted from [2]).

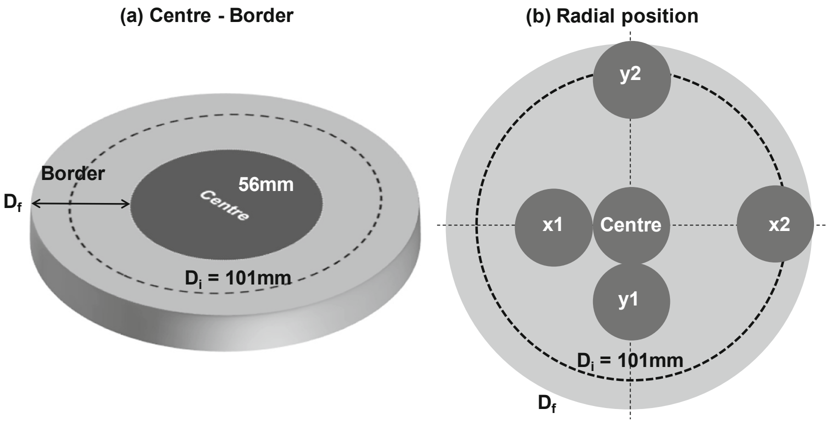

Fig. 4. Geometric configurations used to divide fresh mortar samples for the phase separation evaluation after squeeze flow tests: (a) Centre – Border, (b) Radial position. Each portion was microwave dried for 20 min (unpublished results from [6]).

that strain-hardening at gaps much larger than the maximum particle size is caused by formation of drier microstructures, when the liquid percolates radially the packing of particles [1, 2]. Hence, localized pressure peaks are formed due to particle jamming, and at the end of this microstructural change, the solid content of the center (and pressure) are much higher than in the border, as proved by phase separation tests [2]. The use of viscosity enhancing admixtures, like cellulose ethers, increases the viscosity of water thus improving its drag capacity, which tends to reduce (or eliminate) the relative flow between liquid and particles [2]. The consequences of this effect can be seen by the occurrence of strain-hardening and localized pressure increase only at higher displacements for mortar CE. This is the reason for the employment of such admixtures in compositions submitted to pumping and spraying, situations where phase separation is undesired. The other important contribution of the PMSF is the determination of profiles in radial position (not shown in this work), which allows the comparison with models and the identification of flow type (elongation, shear, plastic or frictional) and possible transitions that can occur [2]. Knowing the actual flow type is crucial to apply the proper rheological model.

As mentioned, a method for quantification of phase separation was developed based on division of the squeezed mortar in different portions and quantifying their water content by microwave drying [2, 5–7]. Figure 5 exemplifies results combining squeeze flow with pressure mapping and phase separation quantification in order to illustrate how the mortar flow occurs when liquid migration is intense. Mortar K is a composition very keen to this phenomenon since it has no admixture whatsoever and the sand's particle size distribution is very narrow. With a very thin paste and a very permeable particle packing this composition is highly susceptible to filtration rather than flowing as a homogeneous fluid, especially when tested at slow rate [1, 2, 6]. As a result, pressure concentration and strain-hardening occur early in the test and the resulting microstructure is a central part with solid content considerably higher than the nominal and even higher than the border. Yet possible and probable, paste-aggregate phase separation is still to be experimentally proved.

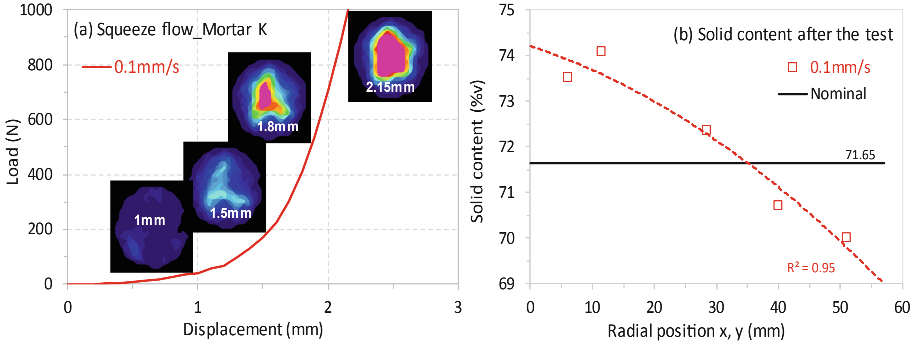

Fig. 5. (a) Squeeze-flow and pressure distribution results of mortar K tested at 0.1 mm/s. (b) Radial solid content (%v) distribution of mortar K after the test assessed by microwave drying [6].

4 Squeeze Flow Over Porous Substrates

In practical applications mortars are applied over different substrates with varying characteristics such as roughness, porosity and water absorption capacity. As demonstrated local variations of water content are important and can adversely affect the flow behavior and homogeneity of the material. Nevertheless, when applied to a wall for example a rendering mortar interacts with the substrate and the increase in consistency within an appropriate time frame is required to allow cutting, leveling and finishing procedures. Therefore, an experimental apparatus was developed, and mortars tested over a ceramic block after different resting times and compared with the standard test, as illustrated in Fig. 6 [6]. Results show that the mortar changes much faster when it loses water to the porous block, with strain-hardening occurring for very small displacements. Such adaptations of the squeeze flow method can be very useful for evaluation of different substrates and development of mix-designs tailored for various environmental conditions, which can be simulated in the laboratory before going to pilot field tests.

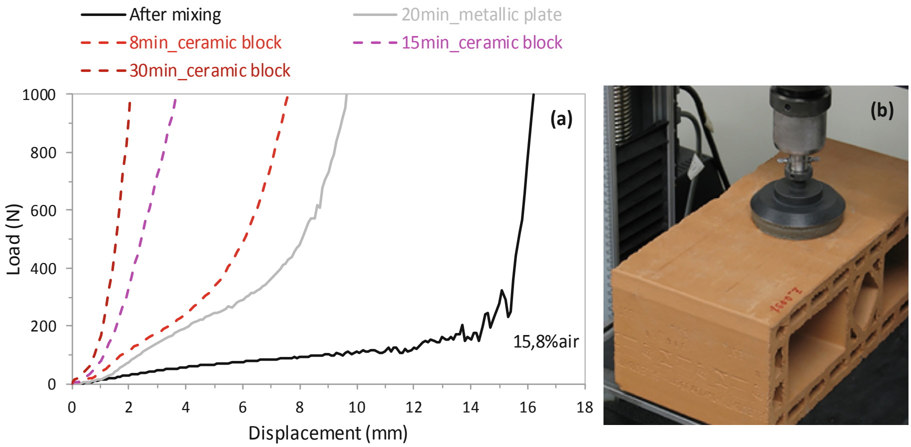

Fig. 6. (a) Squeeze-flow results of mortar Zeta tested at 0.1 mm/s and initial height of 20 mm: solid lines - immediately after mixing and after 20 min resting over the metallic bottom plate; dashed lines - after different resting times over ceramic blocks. (b) Picture of the setup used with the ceramic blocks. (unpublished results from [6]).

5 Evaluation and Development of Mixing Procedures

Due to the geometric restriction, compressive displacement and induction of phase separation that are inherent to squeeze flow, the test is highly sensitive to the agglomeration and homogenization states of the suspensions [2, 4, 8]. These characteristics make the technique very interesting to evaluate the mixing condition of mortars and, hence, the efficiency of mixing procedures and equipment. Using the test, differences in flow behavior have been identified between mortars produced by manual procedure, mechanical mixing according to ABNT 13276 (valid standard procedure until 2016) and a mechanical two-step water addition procedure, as shown in Fig. 7(a). More efficient procedure (Two-step > ABNT 13276 > Manual) produced more flowable mortars and provided smaller variation between batches, due to the higher energy provided to disperse agglomerates and homogenize the constituents [7]. Further research resulted in the creation of a more efficient new Brazilian standard procedure for mortars (ABNT 16541/2016), as evidenced by Fig. 7(b) [8].

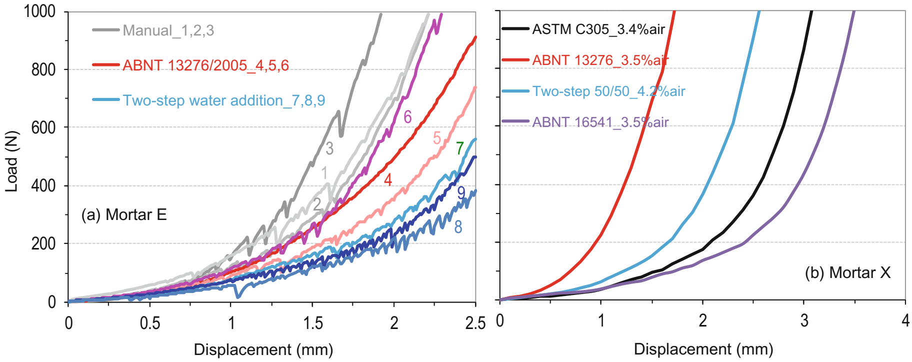

Fig. 7. (a) Squeeze flow results for Mortar E prepared by different mixing procedures: manual mixing with a spatula, mechanical mixing according to standard ABNT 13276/2005 and two-step 50/50% water addition. The batches were prepared 3 times each and the mortar tested at 0.1 mm/s (adapted from [7]). (b) Mortar X prepared following 4 different procedures, including the one described by the new Brazilian standard ABNT 16541/2016, which is more efficient as it is based on two-step water addition (75% + 25%) concept with one minute at high rotation speed in between the steps [8].

6 Rheological Parameters from Rotational Rheometry and Squeeze Flow

A systematic comparison between rotational shear and squeeze-flow for evaluating the rheological behavior of several mortars (Fig. 8) showed encouraging agreement in ranking the mortars' yield stress with a good linear correlation, with some differences depending if whether the material behavior was more visco-plastic or granular [1].

7 Conclusions

Squeeze flow technique has been showing great potential for the rheological evaluation of cement-based and other building materials due its ease of implementation and, especially, because it fills a gap of flow conditions and association with relevant phase separation phenomenon that other methods are not able to provide. For simple and controlled shear evaluation with no geometry change, rotational rheometry is more appropriate, but squeeze flow can be an important complementary test for specific

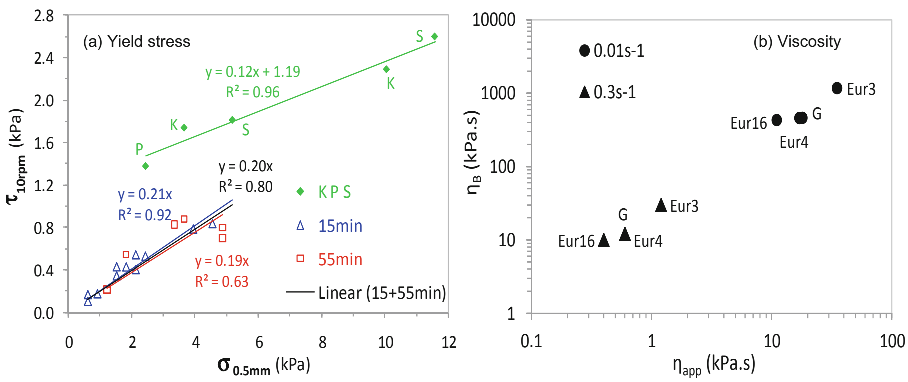

Fig. 8. (a) Comparison of yield stresses of several mortars: rotational shear stress at using Viskomat rheometer vs. squeeze (normal) stress at 0.5 mm. (b) Biaxial extensional viscosity (η_B) from squeeze flow testes of mortars with no phase separation vs. shear apparent viscosity (η_{app}) from Viskomat shear cycles (adapted from [1]).

situations, including 3D printing related processes. The reported developments and associated experimental techniques are important tools for complete rheological evaluation of such complex materials as cement-based mortars and other similar suspensions, fiber-containing and composites.

For some mortars with no segregation it was possible to compare shear and extensional viscosities with a good linear correlation and Trouton ratios between 20 and 40. But for most mortars, phase separation during squeeze flow and also in rotational tests makes this correlation more complex to be performed since different flow conditions are obtained.

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References

1. Cardoso FA, John VM, Pileggi RG, Banfill PFG (2014) Characterisation of rendering mortars by squeeze-flow and rotational rheometry. Cem Concr Res 57:79–87
2. Grandes FA, Sakano VK, Rego ACA, Cardoso FA, Pileggi RG (2018) Squeeze flow coupled with dynamic pressure mapping for the rheological evaluation of cement-based mortars. Cem Concr Compos 92:18–35
3. Toutou Z, Roussel N, Lanos C (2005) The squeezing test: a tool to identify firm cement-based material's rheological behaviour and evaluate their extrusion ability. Cem Concr Res 35:1891–1899. https://doi.org/10.1016/j.cemconres.2004.09.007
4. Nair SAO, Alghamdi H, Arora A, Mehdipour I, Sant G, Neithalath N (2019) Linking fresh paste microstructure, rheology and extrusion characteristics of cementitious binders for 3D printing. J Am Ceram Soc 102:3951–3964. https://doi.org/10.1111/jace.16305

5. Cardoso FA, Lofrano FC, John VM, Pileggi RG (2014) Influence of experimental parameters of the squeeze-flow test on the rheological behaviour and phase segregation of cement mortars. Trans Nordic Rheol Soc 22:79–85
6. Cardoso FA, Pileggi RG (2013) Phase separation in mortars: development of measuring method and influence on rheological behavior. FAPESP Post-Doc Scientific Report 2011/00948-9, no. 1, São Paulo (Unpublished results from internal document)
7. Cardoso FA, Campora FL, Pileggi RG, John VM (2018) Evaluation of mortar mixing procedures using squeeze flow test. In: Proceedings of the International RILEM workshop on rheological measurements of cement-based materials, Arras, pp 18–22
8. Cardoso FA, de França MS, Campora FL, Pileggi RG (2019) Development of the new Brazilian standard mixing procedure for mortar preparation in laboratory. In: 15th International Congress on the Chemistry of Cement, Prague (accepted)