Influence of Experimental Parameters of the Squeeze-flow Test on the Rheological Behaviour and Phase Segregation of Cement Mortars

Fábio A. Cardoso[1], Fábio C. Lofrano[1], Vanderley M. John[1] and Rafael G. Pileggi[1]

[1] Dept. of Construction Engineering, Escola Politécnica - University of São Paulo, Brazil

ABSTRACT

The present work evaluates the influence of experimental parameters of the squeeze-flow test on the rheological behaviour and phase segregation of a cement mortar. Under slow squeezing speed, segregation was high due to the percolation of the liquid phase throughout the structure formed by the particles. The increase of plate roughness caused the strain-hardening stage to occur at smaller deformations and enhanced phase segregation, suggesting that shear flow and phase segregation were directly related. Squeeze curves of the configuration CA100 shifted to higher stress values in all roughness and velocities, owing to its higher D/h ratio, which induced more shear.

INTRODUCTION

The squeeze flow method has been successfully applied for the rheological evaluation of diverse fluids, suspensions and pastes, either to simulate processing and application conditions or to overcome difficulties of the rotational techniques as: slip at material-shear element interfaces, measuring very thick or fiber-containing materials[1]. In civil engineering, the technique has been used as an alternative/complementary method for characterizing the rheological behaviour of pastes[2,3], plasters[4], extruded building materials[5] and different types of mortars[6,7].

The test's geometry change during gap reduction makes the technique particularly interesting, as it creates flow conditions similar to those involved in processing and application of mortars (e.g. pumping and spraying; spreading and finishing; squeezing between bricks). As a result, a testing method for rendering and masonry mortars was recently standardised in Brazil (ABNT NBR 15839:2010).

However, due to its inherent change in geometry, the squeeze test can induce heterogeneous flows and, for suspensions, phase segregation[3,6-8]. Elongational strain is predominant if a slip condition at interfaces is attained in the test, whereas shear occurs under a no-slip situation[1,9]. In practice, both types of flows may take place and their intensity depends on slip condition, geometry (D/h) and material's characteristics[1,6,9]. In this context, plate roughness is a key parameter to induce flow type and, for mortars, to simulate application on substrates with different surface types.

Therefore, the present work evaluates the influence of experimental parameters of the squeeze-flow test on the rheological behaviour and phase segregation of a cement mortar.

EXPERIMENTAL

Mortar

A Brazilian factory-produced rendering mortar was used in the research. The mortar was composed of Portland cement as the binder, hydrated lime and crushed sand with maximum particle size of 2mm. The mean particle size of the composition was 220µm and the fresh characteristics of the mortar after mixing are listed in Tab. 1.

Table 1. Characteristics of the fresh mortar.

% Volume				Density
Aggregates	Fines	Water	Air	(g/cm^3)
45.2	21.5	27.1	6.2	2.0

Squeeze-flow setup

The tests were conducted with three different geometric setups as illustrated in Fig.1. (CA50) Constant area 50mm - the sample area under the 50.8mm diameter top plate was constant but the volume of the sample was reduced during squeezing; (CA100) Constant area with a 101mm top plate; (CV) Constant volume - the volume of the squeezed sample under the top plate (101mm) remained constant but its area increased (initial sample diameter = 50.8mm). Initial sample height (or gap) in all cases was 10mm.

Smooth polished metallic plates (MP) were employed as a reference condition and to change the roughness, water-proof SiC-emery papers (EP) with a grit size of 524µm (Trizact 3M, P36) were glued to the plates.

Squeeze-flow tests were conducted on a universal testing machine (INSTRON 5569) with a 1kN load cell. The bottom plate (200mm in diameter) was mounted over the fixed compression base of the equipment and the top plate (50.8 and 101mm) fixed to the load cell at the crosshead. The samples were squeezed at speeds of 0.1, 1 and 3mm/s, to a maximum displacement of 9mm or maximum load of 1kN (whichever was reached first).

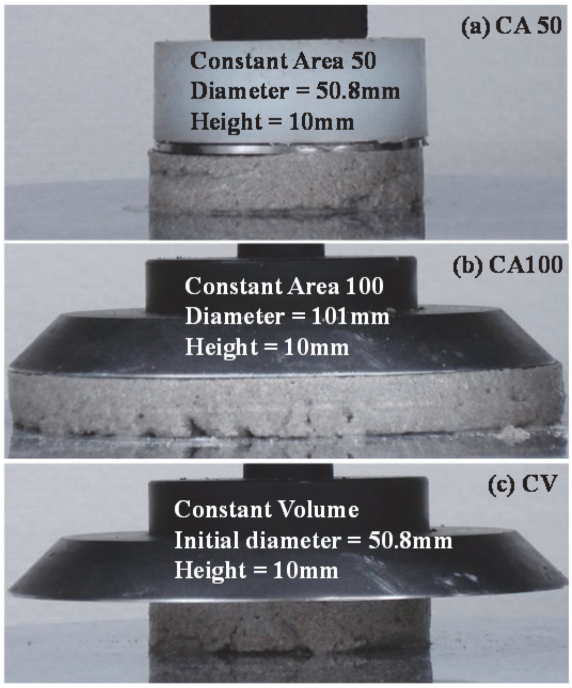

Figure 1. Squeeze-flow setups: (a) Constant area with a 50.8mm top plate; (b) Constant area with a 101mm top plate; (c) Constant volume with a 101mm top plate.

Phase segregation

Liquid-solid segregation induced by the tests was determined by assessing the water contents of the centre and the border of the deformed samples by weighing each portion before and after microwave drying for 15min (Electrolux MEX55 - 1500W). The diameters of the centre portions were 25.4mm for CA50 and VC configurations and 56mm for CA100.

RESULTS AND DISCUSSION

Fig.2 shows the plots of the normal stress vs. the displacement of the top plate for the configuration CA50 with smooth metallic plates (graph a) and emery paper (graph b). In the first case, it can be seen that at higher speeds the viscous flow or plastic deformation stage (a linear increase in stress with the deformation) took place up to 4-4.5mm of displacement and, then, was followed by the transition to the strain hardening. This last stage is characterized by a significant increase of stress (or load) with a small increase in deformation (gap reduction). At 0.1mm/s, this transition occurred at a much smaller displacement (2.5mm) and the plastic deformation stage was short.

The strain hardening behaviour is associated with the friction between particles due to geometry restriction or increase of solids concentration in the central region between the plates[3,5,6,8,10]. The first occurs when the gap reaches a critical value near the maximum particle size present in the material being tested (which was not the case, since no test approached a final displacement around 8mm), while the latter is related to liquid-solid segregation.

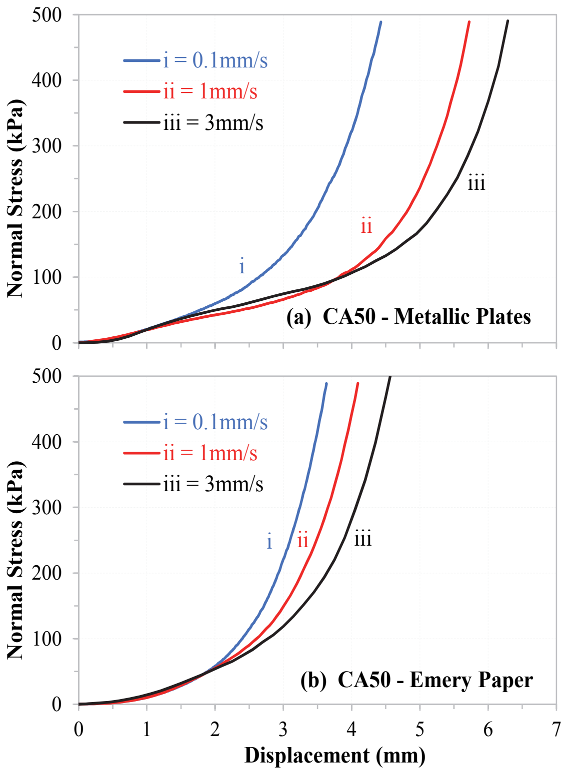

Figure 2. Squeeze-flow results of configuration CA50 at different speeds with (a) metallic plates and (b) emery paper.

The observed behaviour of the squeezing force (or stress) with the displacement rate is caused by the occurrence of liquid-solid segregation, which takes place more intensely at slower speeds. When the sample is squeezed at slow speeds the liquid has a longer time to flow outwards (when compared to faster speeds) throughout the porous structure of packed particles (fines and aggregates), and thus the likelihood of phase segregation is high[3,5,6,8,10]. Conversely, when the sample is tested at high displacement rates, the liquid may not have enough time to flow separately (thus, the material tends to flow homogeneously) and the chance of segregation is lower.

For granular suspensions, radial migration of liquid causes an increase of solids concentration in the central region. As the strain hardening behaviour is associated with the friction between particles, it can take place at smaller displacements and more intensely when phase segregation occurs.

In the condition of high roughness due to the use of the emery paper, the same trend was observed, as depicted in graph b of Fig.2: the slower the displacement rate, the higher the stress required to deform the mortar sample. Additionally, in this case, the strain hardening took place at smaller deformations in all speeds and the squeeze curves were closer to each other. The increase of roughness tend to prevent or minimize slip at the plates-mortar interfaces, thus creating shear flow in the material. According to the results, the introduction of shear made the squeeze-flow of the mortar more difficult when compared to the flow between smooth plates and this difference was more significant at higher speeds.

The same general behaviour can be observed in Fig. 3 and Fig. 4 for the configurations CA100 and CV respectively. The last, presented a very similar behaviour to the CA50, showing a considerable plastic deformation stage and reaching large displacements with the smooth plates and fast speeds. In the condition with the emery paper, strain hardening took place around 2mm and the final displacements were between 3 and 5mm as for the CA50.

However, for the CA100, differences between the curves at different displacement rates were smaller than those observed for CA50 and for CV. Moreover, the maximum displacement of all curves was reduced significantly when compared to the other configurations.

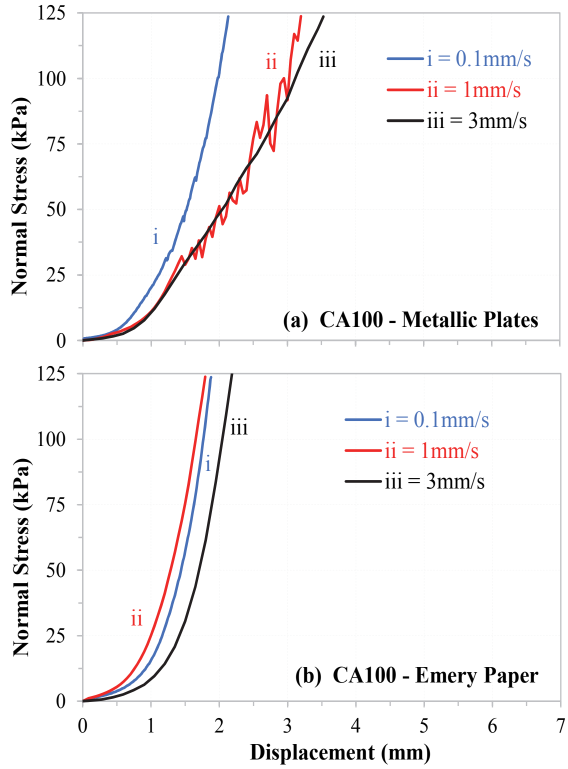

Figure 3. Squeeze-flow results of configuration CA100 at different speeds with (a) metallic plates and (b) emery paper.

The behaviour observed regarding the effect of the displacement rate on the squeeze-flow results of the studied is in accordance with previous studies in cement-based materials[3,5,6], ceramic suspensions[9] and suspensions of spheres in different types of fluids[10]. Nevertheless, in order to enhance the knowledge regarding the occurrence of phase segregation of mortars and how it is affected by the experimental parameters of the test, a method was developed to directly measure the liquid phase migration induced by the squeeze test.

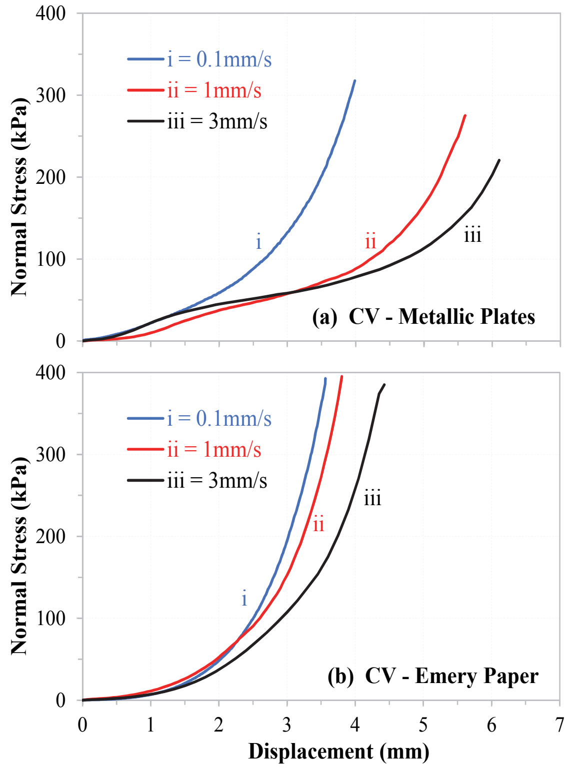

Figure 4. Squeeze-flow results of configuration VC at different speeds with (a) metallic plates and (b) emery paper.

The radial migration of the liquid phase was assessed by weighing the centre and border portions of each mortar sample after deformation. The relative difference of water content between the portions is defined as the relative segregation ($S_{Relative}$):

$$S_{Relative}\ [\%] = (W_{Border} - W_{Centre}) \ / \ W_{Total} \quad (1)$$

Where: W_{Border} = water content in the border; W_{Centre} = water content in the centre; W_{Total} = total water content considering centre + border.

However, as the segregation is generated by the deformation and each sample can reach a different final displacement (or gap), the parameter $S_{Relative}$ is then divided by the final displacement of the corresponding test for a more appropriate comparison of the results, yielding the specific segregation ($S_{Specific}$), as follows:

$S_{Specific}$ [%/mm] = $S_{Relative}$/FD (2)

Where: FD = final displacement of the squeeze test [mm].

Fig. 5 shows the results of both relative and specific segregations (graphs a and b, respectively) as a function of the displacement rate for the diverse combinations of configuration and roughness studied. The analysis using both segregation parameters is important to provide information about the amount of segregation of each test condition as well as how to compare these values.

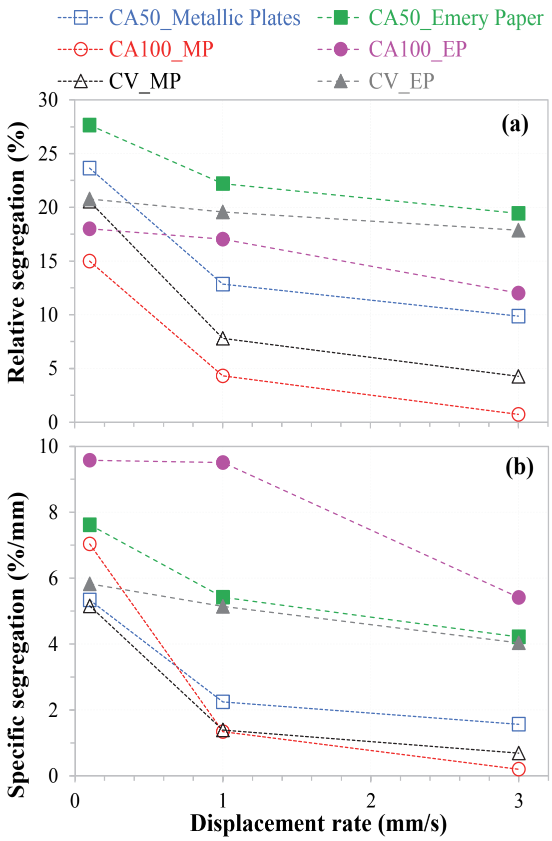

Figure 5. Results of (a) relative segregation and (b) specific segregation as a function of the squeezing speed for the different configurations and roughness used.

It is important to point out that the difference of water content from the centre and border reached values from 15 to 30% at 0.1mm/s, evidencing how heterogeneous the material can be after the rheological test.

For the tests with the metallic plates, there is a considerable reduction of segregation when the speed passed from 0.1 to 1mm/s and the reduction continued from 1 to 3mm/s, in a lesser extent though. The relative segregation is different for each configuration, but when the final displacement is considered, the specific segregation is more similar between the setups. A higher segregation can be observed for CA100 at 0.1mm/s and slightly higher values for CA50, though.

The analysis of Fig. 6 (in addition to Figs. 2, 3 and 4) demonstrates with more detail the shift of normal stress to higher values owing to the use of the emery paper in all three configurations. This was also previously observed for cement suspension[2] and for spread cheese[11] with the use rough surfaces, although for ceramic paste the results reported were the opposite, supposedly due to an increase in segregation with the bare metal[9]. In the present study, not only the normal stress increased, but also the strain hardening occurred at smaller deformations and phase segregation increased substantially, as observed in Fig. 5.

The reduction of segregation due to the increase of speed was not as intense as the reduction that took place with the configurations with the metallic plates. These, results suggest that the shear flow promoted by squeeze with no-slip or near no-slip condition in the cement mortar tested was accompanied by the induction of a considerable amount of phase segregation, even at the highest speeds applied.

Fig. 6 also demonstrates the comparison between the squeeze-flow curves of the different configurations. CA50 and CV show very similar results in both roughness conditions. Still, a lower stress could be observed at high speeds and smooth surface for CV, possibly because of the increase of its area, since the material can flow with no barriers.

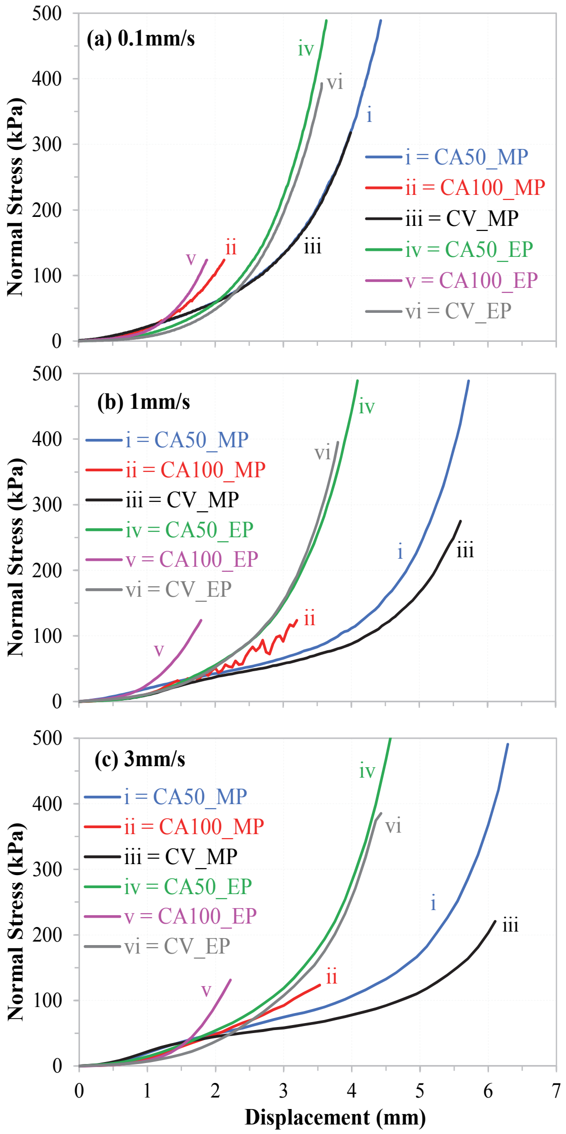

Figure 6. Comparison of squeeze-flow results of the different test configurations (CA50, CA100 and CV) and roughness (MP and EP) used. Squeezing speed: (a) 0.1mm/s; (b) 1mm/s; (c) 3mm/s.

On the other hand, in the CA50 condition the portions of the mortars that were squeezed out could provide some resistance to the flow, possibly causing this kind of difference between the results of the two configurations. The phase segregation of the CA50 is also slightly higher than that of CV, since the latter had a more homogenous pressure distribution (with no edge effects) - since all material was below the top plate -, while the former probably had a considerable pressure variation between the portions of the sample below the plate and squeezed out.

The configuration CA100 required higher stresses to deform the mortar samples than did the other configurations in both roughness conditions applied. Its greater diameter / height ratio (D/h = 10) tends to promote the occurrence of shear flow[1,9] and probably caused the increase of stress observed. With the metallic plates, only at low speed (0.1mm/s) its phase segregation was higher than that of the other configurations, but with the use of the emery paper, its amount of segregation in relation to displacement was much greater than those of CA50 and CV.

For the determination of the viscosity values, one of the conditions is that the material must remain homogeneous for the application of rheological models. In the present work, it was proved that in the majority most of the experimental situations applied – configuration, roughness and displacement rate – the mortar did not remain homogeneous. In this sense, phase segregation should be taken into account when performing and analysing squeeze flow rheometry of concentrated suspensions.

CONCLUSIONS

This paper reported an investigation on the experimental parameters of the squeeze-flow test applied to cement mortars. An experimental method was developed to quantify the phase segregation induced by the test and showed that liquid-solid segregation plays an important role on the squeeze-flow behaviour of concentrated suspensions, as the cement mortar analysed. Normal stress decreased with the increase of squeezing speed, due to the reduction of phase segregation in all configurations and roughness conditions applied.

Constant area (CA50) and constant volume (CV) configurations with initial diameter of 50mm showed similar results, with small influence of edge effects especially at higher speeds. The higher diameter/height ratio of the CA100 configuration increased the force required to deform the samples, probably owing to the generation/intensification of shear flow during the experiments.

The no-slip or near no-slip condition induced by the use of emery paper at the plates caused a significant increase of force to squeeze the mortar samples along with a very considerable growth of the phase segregation levels.

ACKNOWLEDGMENTS

The authors would like to thank Brazilian research funding agencies CNPq, CAPES and FAPESP for funding.

REFERENCES

1. Engmann, J., Servais, C. and Burbidge, A.S. (2005), "Squeeze flow theory and applications to rheometry: A review", *J. Non-Newtonian Fluid Mech*., **130**, 149-175.

2. Min, B.H., Erwin, L. and Jennings, H.M. (1994), "Rheological behavior of fresh cement paste as measured by squeeze flow", *J. Mater. Sci.*, **29**, 1374-1381.

3. Phan, P.H., and Chaouche, M. (2005), "Rheology and stability of self-compacting concrete cement pastes", *Appl. Rheol*., **15**, 336.

4. Cardoso, F.A., Agopyan, A.K., Carbone, C., John, V.M. and Pileggi, R.G. (2009), "Squeeze flow as a tool for developing optimized gypsum plasters", *Constr. Build. Mater*. **23**, 1349.

5. Toutou, Z., Roussel, N., and 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.

6. Cardoso, F.A., John, V.M. and Pileggi, R.G. (2009), "Rheological behavior of mortars under different squeezing rates", *Cem. Concr. Res*., **39**, 748-753.

7. Cardoso, F.A., John, V.M., Pileggi, R.G. and Banfill, P.F.G., (2014), "Characterisation of rendering mortars by squeeze-flow and rotational rheometry", *Cem. Concr. Res*., **57**, 79-87.

8. Poitou A. and Racineux G. (2001), "A squeezing experiment showing binder migration in concentrated suspensions", *J. Rheol.* **45**, 609-625.

9. Meeten, G.H. (2004), "Squeeze flow of soft solids between rough surfaces", *Rheol. Acta*, **43**, 6-16.

10. Collomb, J., Chaari, F. and Chaouche, M. (2004), "Squeeze flow of concentrated suspensions of spheres in Newtonian and shear-thinning fluids", *J. Rheol.* **48**, 405-416.

11. Steffe, J.F. (1996), "Rheological methods in food process engineering", Freeman Press, USA, pp. 255-293.