Influence of Squeezing Rate on Yield Stress and Viscosity of Fresh Mortar

Van Tien Phan
Faculty of Construction
Vinh University
Vinh City, Vietnam
vantienkxd@vinhuni.edu.vn

Duy Duan Nguyen
Faculty of Construction
Vinh University
Vinh City, Vietnam
duyduankxd@vinhuni.edu.vn

***Abstract*-In the present work, squeeze flow techniques were used to investigate the influence of squeezing rates on the yield stress of mortars in fresh state. The tested samples were prepared under similar conditions of room temperature and atmospheric pressure. The fresh mortars were tested at three squeezing rates (20 and 200mm/s) 15 minutes after mixing. The results show that the material's yield stress increases with the increasing of the squeeze velocity. This increase is evident at low tensile speeds and is not obvious at high tack velocity. Elongational viscosity values increased as a result of the gap reduction for all the tested samples. However, when the squeeze speed was high, the strain rate increased because of the high displacement rates, a significant reduction in the mortar's elongational viscosity was observed compared with those obtained when the squeeze speed was low. Despite that this behavior is associated with fluid-solid phase separation, which occurs for low displacement rates, these viscosity values actually represent the behavior of the material in practical situations when submitted to different velocities. The increase in the displacement rate of one order of magnitude caused a reduction in the viscosity of one order of magnitude.**

Keywords-squeeze flow technique; fresh mortar; yield stress; viscosity

I. INTRODUCTION

The fresh state of a cement-based material, including mortar, corresponds to only a minor part of its lifetime, nevertheless, the behavior of the material within this frame has major consequences on its hardened properties. The currently applied methods for testing this kind of materials during the fresh state are simple but limited. The flow table (ASTM-C1437, EN-1015-3) and dropping ball (BS-4551) methods investigate fresh mortar by using single point measurements [1]. These methods are unable to dissociate the contributions of the yield stress or of the viscosity on the resulting measurements. The material's behavior is also undetermined, since at least two points are needed to describe simple rheological behavior [1, 2]. In order to overcome the limitations of the traditional methods, rotational rheometers have been used, in which the mortar's rheological behavior and parameters such as yield stress and viscosity were determined in either shear stress or shear rate-controlled procedures [3, 4]. The technique is an important tool for controlling and developing cementitious material formulations, including mortar, especially for the simulation of mixing and pumping situations.

However, during applications, the mortar is spread over a substrate and then squeezed between bricks (masonry and tile adhesive mortar) or projected and spread over a surface for internal and external rendering purposes. The mortar fraction of a concrete mix is also squeezed locally between coarse aggregates during fresh concrete flow [5]. Therefore, the rotational rheometers are not suitable in these cases. The behavior of the material under different squeezing rates provides important information for controlling its rheological performance. Considering this scenario, the main objective of this research is to investigate the influence of the squeezing rates on the yield value and the viscosity of fresh mortar.

II. SQUEEZE TEST

The squeeze test has been widely used to determine the flow properties of highly viscous pastes (food, cosmetic, polymers, composites, ceramic pastes and others) [6-10], as it overcomes some of the common problems of conventional rheometers such as slip, disruption of plastic materials and the difficulty to load very thick and fiber-containing fluids in rotational devices [5]. Squeezing technique can characterize cementitious materials by compressing a cylindrical specimen between parallel surfaces by controlled force or displacement rate. This method has been previously used for characterizing the rheological behavior of cement pastes [11], Herschel Bulkley fluid [12], Bingham plastic [13], etc. The typical load vs. displacement profile of a constant velocity squeeze flow experiment was determined in [11] and it is used to determine the rheological parameters of testing materials, including yield stress and viscosity. Direct measurements of yield stress are uniquely performed by stress-controlled rheometers [3, 4]. Squeeze flow tests carried out with constant displacement velocity do not allow such direct measurement [5] since the material flow occurs regardless of the existence of the material's yield stress, unless the force required to overcome this value exceeds the load limit of the testing device.

In this study, the testing material is firstly compressed/squeezed between two parallel surfaces continuously with relaxation periods of 1.5min until reaching the predefined thickness and is finally separated with predefined tack velocity (Figure 1). A typical curve of squeeze-tack experiment is presented in Figure 2, in which three periods can be observed, including compression, relaxation and traction.

Corresponding author: Van Tien Phan

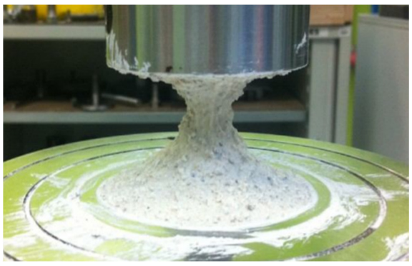

Fig. 1. A squeezing experiment of mortar in process.

It is possible to conduct indirect yield stress determination by the extrapolation of the flow curves in the squeeze stage of the experiment. The yield stress is calculated by dividing the maximum force recorded by the area at that time. The formula is as follows:

$$\sigma_t = \frac{F_S^{\max}}{A_s} \quad (1)$$

in which A_s is the average area of the testing sample at the moment that the recorded load is max:

$$A_t = \frac{V}{h'} = \frac{\pi R^2 h}{h'} \quad (2)$$

where h is the predefine thickness of the test mortar, R is the radius of the sample and h' is the mortar's thickness at the moment of the maximum squeeze force.

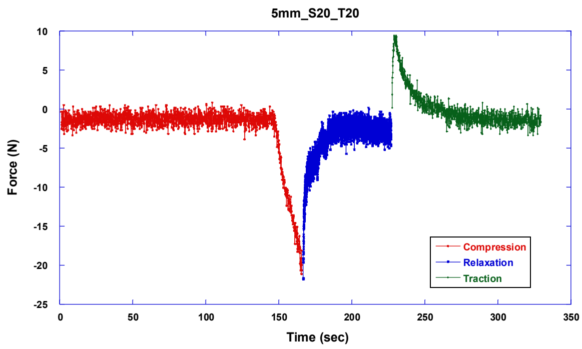

Fig. 2. A typical curve obtaines in a squeeze tack test.

During the squeeze flow experiments conducted with a constant downward velocity of the upper plate, the sample height decreased linearly, as: $h=h_0 - vt$, where h is the momentary height of the sample, h_0 is the initial sample height, v is the displacement velocity of the upper plate and t is the time elapsed after the beginning of the test [6]. The biaxial extensional strain rate or elongational strain rate is equal to half the vertical Hencky strain rate [6]:

$$\dot{\varepsilon}_B = \frac{\dot{\varepsilon}_H}{2} = \frac{v}{(2h)} \quad (3)$$

The extensional viscosity (η_B) is defined as the ratio between the biaxial extensional stress (σ_B), which is the squeezing load divided by the top plate area, and the extensional strain rate:

$$\eta_B = \frac{\sigma_B}{\dot{\varepsilon}_B} = 2L\left[\frac{h-(vt)}{v\pi R^2}\right] \quad (4)$$

where L is the load and R is the radius of the sample [6].

III. EXPERIMENTAL PROCEDURE

The mortar used in this investigation was referenced from the research of Dr. Rafael Pileggi from the University of Sao Paolo, Brazil [5, 14] and Dr. Mohend Chaouche at LMT, ENS Cachan, France [15]. The detailed composition of the mortar is presented in Table I. In order to minimize the effect of sand grading on the rheological properties of concrete, strict grading component control commercial normalized CEN 196-1 sand, was used. This is European standard sand (ISO 679), which is very clean, with round shaped particles of the same size. It is dried, screened and prepared in a modern factory, ensuring quality and consistency, packed in bags containing 1350±5g (Figure 3).

TABLE I. MORTAR COMPOSITION

Constituents	% wt. of dry mixture
White cement	30
Normalized sand	70
MKX 70000 PP01	0.5
Water	25

Walocel MKX 70000 PP01 hydroxyethyl methyl cellulose has been added in the composition of mortar with fixed percentage of dry mixture (0.5%). It imparts well-balanced properties, including open time, adhesion and shear strength, adds good workability and enhances water retention. The selected particle size distribution provides quick, lump-free dissolution. It is compatible with all conventional mineral and organic binders.

Fig. 3. Normalized sand used in the experiments.

For evaluating the influence of the squeezing rates to the yield stress of the mortar, testing samples were squeezed at 20 and 200mm/s squeezing speeds. After relaxation, the testing samples were pulled out at different speeds, including 2, 20 and 200mm/s. The variation of the yield stress of the testing material was studied by analyzing the recorded flow curves during the experiments. Additionally, the height of the sample should be at least 10 times greater than the maximum particle size to avoid wall effects [5]. Hence, the predefined thickness of the testing material was taken as 3.5mm.

IV. RESULTS AND DISCUSSION

A typical flow curve obtained in the squeeze tack experiment is presented in Figure 4. The maximum calculated stress is considered as the yield value of the material. From the flow curves, the value F_{max} is recorded and given in Table II. The yield stress of the material is calculated by (1) and is presented in Table III.

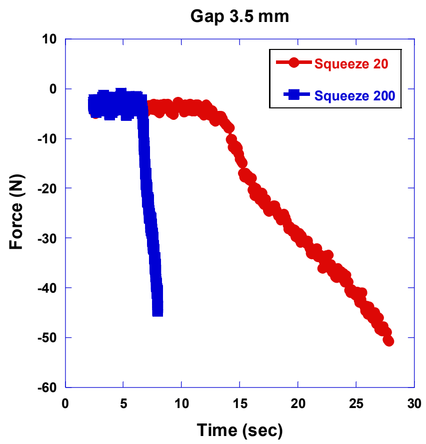

Fig. 4. A typical curve obtained in the squeeze period of squeeze flow test.

TABLE II. RECORDED MAXIMUM LOAD IN A SQUEEZE PERIOD

	v_t = 2 mm/s	v_t = 20 mm/s	v_t = 200 mm/s
v_s = 20 mm/s	40.788	38.102	48.173
v_s = 200 mm/s	59.084	60.427	47.838

TABLE III. YIELD STRESS UNDER DIFFERENT SQUEEZING RATES

Squeeze – tack velocity	Yield stress (N/mm^2)
S20 T2	0.017168
S20 T20	0.016037
S20 T200	0.020276
S200 T2	0.024869
S200 T20	0.025434
S200 T200	0.020135

The variation of material's yield stress versus the squeeze rate is plotted in Figure 5. As it can be easily seen from the Figure, at a constant pulling velocity the materials' yield stress increases with increasing squeeze velocity. This increase is evident at low tensile speeds (2mm/s) and fast pulling speeds (200mm/s). At an average speed of 20mm/s, this increase is not obvious. The above results show that the pressing speed in phase I (construction phase) has a significant impact on the ability to remove the mortar and the upper surface (masonry bricks, tiles adhesive, etc.). High squeezing speed will increase the ability to resist peeling of materials when sticking to mortar. The extensional viscosity (η_B) is calculated by (4) and the results are shown in Table IV.

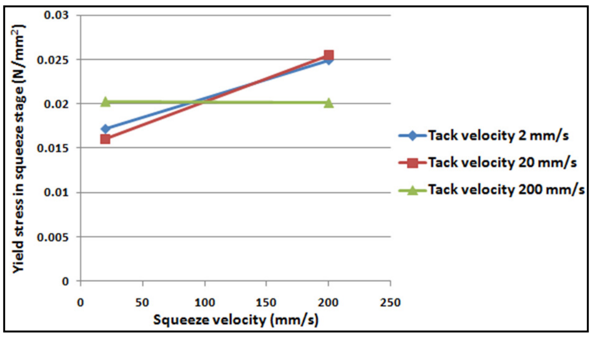

Fig. 5. Material yield stress versus squeeze rate at different tack velocities.

TABLE IV. EXTENSIONAL VISCOSITY OF TESTING MATERIALS UNDER DIFFERENT SQUEEZING RATES

Squeeze – tack velocity	Extensional viscosity (Pa.s)
S20 T2	6,034.17
S20 T20	6,733.67
S20 T200	7,187.94
S200 T2	879.11
S200 T20	890.55
S200 T200	705.68

The evolution of material's extensional viscosity versus the squeeze rate is shown in Figures 6 and 7. The interpretation of the viscosity results obtained by squeeze flow must consider that, although the downward velocity remains constant (20 and 200mm/s), the elongational strain rate is constantly increasing as the gap height decreases (within a single test). Similar observations were recorded at different experiments with tack speeds of 2mm/s, 20mm/s and 200mm/s. Elongational viscosity values increased as a result of gap reduction for all the tested samples. Furthermore, unlike rotational rheometry, where the gap is constant and the increase of viscosity with the strain rate in fact represents dilatancy, the reduction of the gap during squeezing stimulates different units (grains getting closer to each other) causing the observed increase of the mortar's elongational viscosity. This finding is similar to another case of cementitious materials, which has been reported in [5].

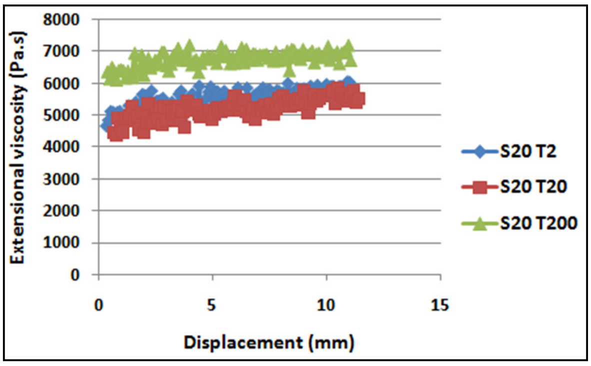

Fig. 6. Calculated material extensional viscosity at squeeze velocity of v_s=20mm/s.

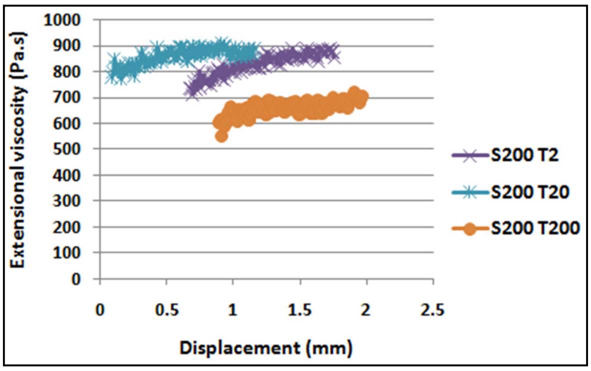

Fig. 7. Calculated material extensional viscosity at squeeze velocity of v_s=200mm/s.

However, when the squeeze speed was high (200mm/s) and the strain rate increased due to the high displacement rates, a significantly reduction in the mortar's elongational viscosity was observed in comparison with those obtained when the squeeze speed was low (20mm/s). It can be easily seen that the average elongational viscosity is about 5000-7000Pa.s in the case of high squeeze speed, compared with about 600-900Pa.s in the case of low squeeze speed. Despite the fact that this behavior is associated to fluid–solid phase separation which occurs for low displacement rates, these viscosity values actually represent the behavior of the material in practical situations when submitted to different velocities. The increase in the displacement rate of one order of magnitude from 20 to 200mm/s caused a reduction in the viscosity of one order of magnitude.

V. CONCLUSIONS

The squeeze flow is a simple and versatile method for the rheological characterization of mortars in a wide range of consistencies, providing much important information of the rheological behavior of materials in practical applications. From the flow curves obtained by the squeeze tack experiments, the rheological parameters, including yield stress and elongational viscosity, have been investigated. The results show that the material's yield stress increases with the increase of squeeze velocity. This increase is evident at low tensile speeds and is not obvious at high tack velocities. Elongational viscosity values increased as a result of gap reduction for all the tested samples. However, when the squeeze speed was high and the strain rate was increased because of the high displacement rates, a significantly reduction in the mortar's elongational viscosity was observed compared with those obtained when the squeeze speed was low. Despite that this behavior is associated to fluid–solid phase separation, which occurs for low displacement rates, these viscosity values actually represent the behavior of the material in practical situations when submitted to different velocities. The increase in the displacement rate of one order of magnitude caused a reduction in the viscosity of one order of magnitude.

REFERENCES

[1] P. F. G. Banfill, "Use of the ViscoCorder to study the rheology of fresh mortar," *Magazine of Concrete Research*, vol. 42, no. 153, pp. 213–221, Dec. 1990, doi: 10.1680/macr.1990.42.153.213.

[2] R. G. Pileggi, "Novel tools for the study and development of refractory castables," (in Portugese), Ph.D. dissertation, Federal University of Sao Carlos, Sao Carlos, Brazil, 2001.

[3] B. Belahcene, A. Mansri, and A. Benmoussat, "Investigation on the Rheological Behavior of Multigrade Oil under the Effect of Organic and Inorganic Impurities," *Engineering, Technology & Applied Science Research*, vol. 4, no. 6, pp. 711–713, Dec. 2014.

[4] V. T. Phan, "Influence of re-dispersible powder on the properties of mortars," *Journal of Materials and Engineering Structures*, vol. 1, no. 1, pp. 2–10, Jan. 2014.

[5] F. A. Cardoso, V. M. John, and R. G. Pileggi, "Rheological behavior of mortars under different squeezing rates," *Cement and Concrete Research*, vol. 39, no. 9, pp. 748–753, Sep. 2009, doi: 10.1016/j.cemconres.2009.05.014.

[6] K. Kardjilova, V. Vozarova, and M. Valah, "Influence of Temperature on Energetic and Rheological Characteristics of PLANTOHYD Bio Lubricants – a Study in the Laboratory," *Engineering, Technology & Applied Science Research*, vol. 3, no. 3, pp. 424–428, Jun. 2013.

[7] G. H. Meeten, "Yield stress of structured fluids measured by squeeze flow," *Rheologica Acta*, vol. 39, no. 4, pp. 399–408, Aug. 2000, doi: 10.1007/s003970000071.

[8] N. Ozkan, C. Oysu, B. J. Briscoe, and I. Aydin, "Rheological analysis of ceramic pastes," *Journal of the European Ceramic Society*, vol. 19, no. 16, pp. 2883–2891, Dec. 1999, doi: 10.1016/S0955-2219(99)00054-0.

[9] J. Engmann, C. Servais, and A. S. Burbidge, "Squeeze flow theory and applications to rheometry: A review," *Journal of Non-Newtonian Fluid Mechanics*, vol. 132, no. 1, pp. 1–27, Dec. 2005, doi: 10.1016/j.jnnfm.2005.08.007.

[10] O. H. Campanella and M. Peleg, "Squeezing Flow Viscosimetry of Peanut Butter," *Journal of Food Science*, vol. 52, no. 1, pp. 180–184, Jan. 1987, doi: 10.1111/j.1365-2621.1987.tb14000.x.

[11] B. H. Min, L. Erwin, and H. M. Jennings, "Rheological behaviour of fresh cement paste as measured by squeeze flow," *Journal of Materials Science*, vol. 29, no. 5, pp. 1374–1381, Mar. 1994, doi: 10.1007/BF00975091.

[12] J. D. Sherwood and D. Durban, "Squeeze-flow of a Herschel–Bulkley fluid," *Journal of Non-Newtonian Fluid Mechanics*, vol. 77, no. 1, pp. 115–121, May 1998, doi: 10.1016/S0377-0257(97)00099-2.

[13] D. N. Smyrnaios and J. A. Tsamopoulos, "Squeeze flow of Bingham plastics," *Journal of Non-Newtonian Fluid Mechanics*, vol. 100, no. 1, pp. 165–189, Sep. 2001, doi: 10.1016/S0377-0257(01)00141-0.

[14] F. A. Cardoso, A. K. Agopyan, C. Carbone, R. G. Pileggi, and V. M. John, "Squeeze flow as a tool for developing optimized gypsum plasters," *Construction and Building Materials*, vol. 23, no. 3, pp. 1349–1353, Mar. 2009, doi: 10.1016/j.conbuildmat.2008.07.017.

[15] N. Delhaye, A. Poitou, and M. Chaouche, "Squeeze flow of highly concentrated suspensions of spheres," *Journal of Non-Newtonian Fluid Mechanics*, vol. 94, no. 1, pp. 67–74, Nov. 2000, doi: 10.1016/S0377-0257(00)00130-0.

AUTHORS PROFILE

Van Tien Phan was born in Nghe An, Vietnam, in 1984. He received the degree of Civil Engineering in the University of Communication and Transport, Hanoi in 2007, Master degree in Advanced Modelling of Materials and Structures from the ENS Cachan, France in 2008, and a PhD degree in Mechanical Engineering from LMT, ENS Cachan, France, in 2012. He is the author of one book and more than 50 articles, and the principle investigator of more than 10 research projects. His research interests include material sciences and mechanical engineering.

Duy Duan Nguyen received a BSc degree of Civil Engineering at Vinh University, Vietnam in 2008, a MSc degree of Structural Engineering at the National University of Civil Engineering, Vietnam in 2012, and the PhD degree of Civil Engineering at Konkuk University, Korea in 2019. His research interests are focused on structural and earthquake engineering and high-performance materials for civil engineering structures. He has published more than 30 papers in high-qualified journals and conferences proceedings.