YIELD STRESS OF CEMENTITIOUS MATERIALS UNDER DIFFERENT SQUEEZING RATES

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Abstract: In the present work, the squeeze flow techniques were used to investigate the influence of squeezing rates to the yield stress of different cementitious materials, including mortars and cement pastes. Tested samples were prepared under similar conditions of room temperature and atmospheric pressure. Compositions of motars and cement pastes were tested at three squeezing rates (20 and 200 mm/s) 15 min after mixing. The results indicate an increasing in the material's yield stress with the rising of the squeeze velocity. This increase is evident at low tensile speeds (2 mm/s) and fast pulling speeds (200 mm/s). At an average speed of 20 mm/s, this increase is not obvious. It shows that the pressing speed in construction period 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.
Keyword: Squeeze-tack experiment; cementitious materials; yield stress.

1. Introduction

The fresh state of a cement-based material 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 these kinds 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 indetermined, 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 rheometer has 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 materials formulations, including mortar, especially for the simulation of mixing and pumping situations [17].

However, during applications, the mortar is spread over a substrate and then squeeze 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 rheometry is not suitable in these cases.

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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 of cementitious materials, including cement paste and mortar.

2. 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 rheometry such as slip, disruption of plastic materials and the difficulty to load very thick and fiber-cointaining fluids in rotational devices [5].

Squeezing technique can characterized cementitious materials by compressing of a cylindrical specimen between to parallel surfaces by controlled force or displacement rate. This method has been previously used by various researchers for characterizing the rheological behavior of a cement pastes [11], Herschel Bulkley fluid [12], [16], Bingham plastic [13], etc. The typical load vs. displacement profile of a constant velocity squeeze flow experiment was determined [11] and is used to obtain rheological parameters of testing materials, including yield stress and viscosity.

Direct measurements of yield stress are uniquely performed by stress-controlled rheometry [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 materials's yield stress, unless the force required to overcome this value exceeds the load limit of the testing device.

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

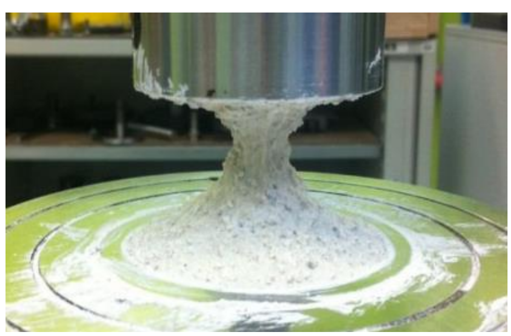

Figure 1: *A squeezing experiment of mortar in process*

It is possible to conduct indirect yield stress determination by the extrapolation of the flow curves in the third period of the experiment. The yield stress is calculated dividing the maximum force required to separate the two surafces by the area at that time. The formula is as follows: $\sigma_t = \frac{F_t^{\max}}{A_t}$, in which A_t is the average area of the testing sample at the moment that the tack forces is max, $A_t = \frac{V}{h'} = \frac{\pi R^2 h}{h'}$, in which h is the predefine thickness of the test material, R is the radius of the sample and h' is the material's thickness at the moment of the maximum tack force.

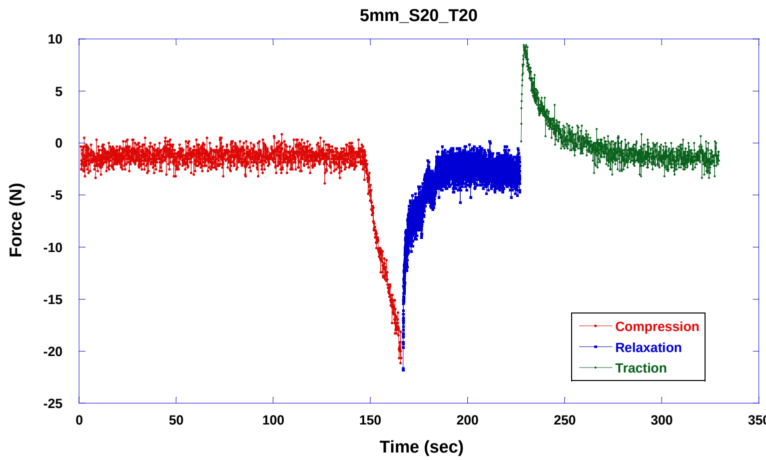

Figure 2: *A typical curve obtained from a squeeze tack test*

3. Experimental study

The material composition used in this investigation is presented in Table 1.

Table 1: *Material composition*

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

In order to minimize the effect of sand grading on the rheological properties of concrete, strict grading component control commercial sand, normalise sand CEN 196-1, was used. This is European standard sand (ISO 679), which is very clean, with particles of the same size and round shape. It is dried, screened and prepared in a modern factory, ensuring quality and consistency, packed in bags containing 1350 ± 5 g (Figure 3).

Figure 3: *Normalise sand, used in the experiment*

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.

For evaluating the influence of the squezzing rates to the yield stress of the mortar, testing samples were squeezed at various squeezing speed, including 20 and 200 mm/s. After relaxation, testing sample was pulled out at different speed, including 2, 20 and 200 mm/s. By analyzing the recorded flow curves during the experiments, the variation of the yield stress of testing material will be investigated.

Additionally, the height of the sample should be at least ten times greater than the maximum particle size to avoid wall effects [5]. Hence, the predefined thickness of the testing material is taken 3.5 mm.

4. Results and discussion

A typical flow curve obtain in the squeeze tack experiment is presented in Figure 4. The maximum calculated stress is considered the yield value of the material. From the flow curves, the value F_{max} is recorded and given in Table 2.

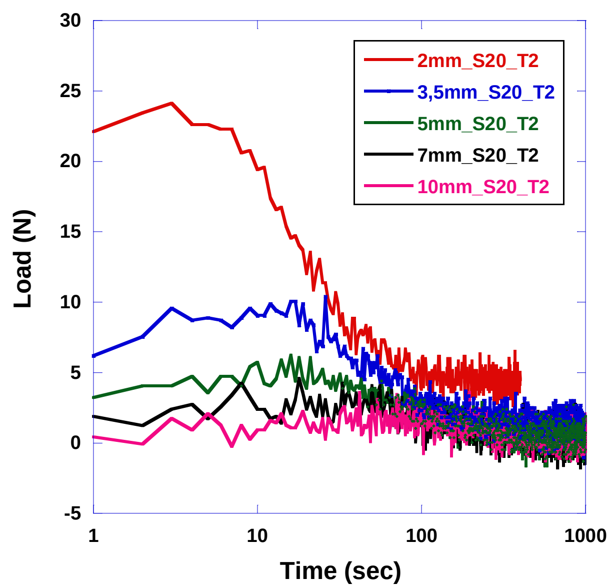

Figure 4: *A typical curve obtained in the tack period of squeeze flow test*

Table 2: *Recorded maximum load in tacking period of squeeze-tack test (N)*

	Traction velocity V_t=2 mm/s	Traction velocity V_t=20 mm/s	Traction velocity V_t=200 mm/s
Squeeze velocity Vs=20 mm/s	8.5604	14.603	25.513
Squeeze velocity Vs=200 mm/s	8.0569	14.771	18.128

The yield stress of the materials is calculated by the aboving formulation and presented in Table 3.

Table 3: *Yield stress of testing materials under different squeezing rates (N/mm^2)*

Squeeze - Tack velocity	S20 T2	S20 T20	S20 T200	S200 T2	S200 T20	S200 T200
Yield value	0.00452727	0.006838755	0.011566969	0.00367877	0.006732514	0.007933204

The variation of materials' 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 material's yield stress increases with the increasing of the squeeze velocity. This increase is evident at low tensile speeds (2 mm/s) and fast pulling speeds (200 mm/s). At an average speed of 20 mm/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.

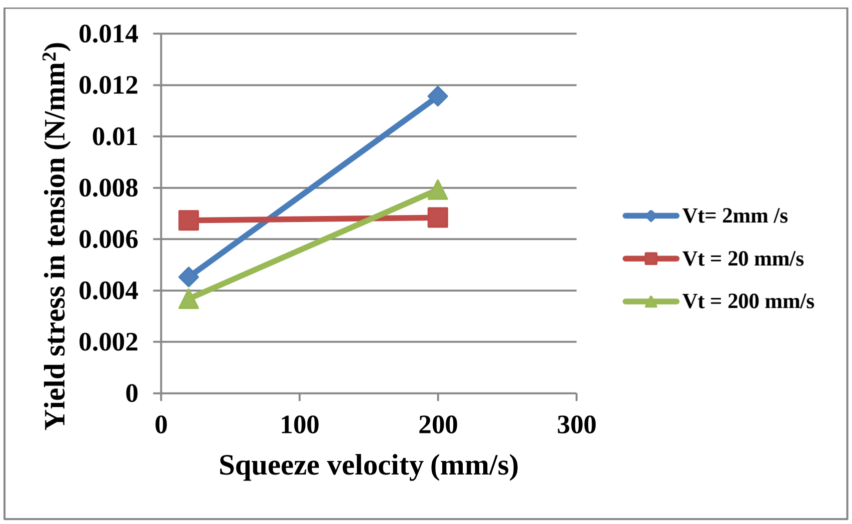

Figure 5: *Calculated materials' yield stress versus squeeze rates at different tack velocities*

Observing the graph in Figure 5, we see that when the average tack speed is 20 mm/s, the yield stress in tension of the mortar is almost unchanged for different pressing speed. This result is clearly different from the other tack speeds, 2 and 200 mm/s. To assess the cause of this phenomenon, it is necessary to study other properties of mortar such as viscosity, ... This may also be a critical result.

5. 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 application. From the flow curves obtained in the squeeze tack experiments, the rheological parameters, including yield stress in tension, has been investigated. 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 fast pulling speeds. At an average speed of 20 mm/s, this increase is not obvious. It proves 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.

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TÓM TẮT

ỨNG SUẤT GIỚI HẠN CHẢY CỦA VẬT LIỆU XI MĂNG CHỊU CÁC TỐC ĐỘ ÉP KHÁC NHAU

Trong nghiên cứu này, kỹ thuật ép - kéo được sử dụng để tìm hiểu ảnh hưởng của tốc độ ép trong quá trình thi công tới ứng suất giới hạn chảy của vật liệu xi măng, bao gồm vữa và hồ xi măng. Các mẫu thí nghiệm được chuẩn bị trong cùng điều kiện nhiệt độ phòng và áp suất khí quyển. Trong thí nghiệm, các mẫu vật liệu được ép với các tốc độ khác nhau, bao gồm 20 và 200 mm/s, 15 phút sau khi trộn. Kết quả cho thấy ứng suất giới hạn chảy khi kéo của vật liệu tăng khi tăng tốc độ ép ở giai đoạn thi công. Sự tăng này khá rõ ở tốc độ kéo thấp nhất và nhanh nhất. Ở tốc độ kéo trung bình (20 mm/s), sự tăng này không đáng kể. Điều đó cho thấy tốc độ ép ở giai đoạn thi công có ảnh hưởng đáng kể đến khả năng bóc tách liên kết giữa vữa và các lớp liên kết (vữa xây, vữa trát tường, …). Tốc độ ép cao sẽ tăng khả năng chống lại sự bong tróc của vật liệu khi dính bám với vữa.

Từ khóa: Thí nghiệm ép kéo; vật liệu xi măng; ứng suất giới hạn chảy.