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Characterization of Rheology of Fresh Cement-based Materials for Extrusion via Direct Shear Test

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Abstract— **In this paper, direct shear box test was employed for characterizing rheology of semi-solid, dough-like fresh cement-based materials tailored for extrusion. In total, five fresh cementitious mixtures, suitable for extrusion, were prepared and tested under various values of consolidation pressure and their shear yield strength was obtained. The results were interpreted by Mohr-Coulomb failure envelope. Effects of fiber volume, and water content on rheological properties of the fresh cement-based materials for extrusion were investigated. It was concluded that shear box test was an effective method for characterization of rheology of semi-solid fresh cementitious composites for extrusion. Water and fibers demonstrated different influence on overall rheology of the semi-solid, dough-like fresh cementitious composites for extrusion. The research findings will help for tailoring and optimization of material formulations of cement-based materials for extrusion.**

Keywords—extrusion, fresh cement, nanoclay, rheology, semi-solid, shear box

I. Introduction

As an economical, energy efficient, and environmental-friendly continuous material processing technology, extrusion is widely-used in plastic, food, ceramic, high-temperature metal alloy, and pharmaceutical industry. This technique has been introduced into concrete industry for manufacturing cement-based building products like flat and corrugated sheets, solid façade panels, honeycomb panels, window/door frames, door panels, roof tiles, down pipes etc. [1-8]. These represent a revolutionary change in manufacturing cement-based products from batch processing to continuous processes, which can improve mechanical properties and durability of cement composites [9-10]. A successive extrusion process largely depends on rheological properties of materials being processed. It is expected that the fresh materials for extrusion exhibit homogeneous dispersion of ingredients; soft enough to be extruded through the die continuously with reasonably low energy consumption; high enough 'green strength' to maintain its shape upon exit of the die; and appropriate deformability rate so that reasonable high production rate can be achieved by an extrusion system. The rheological behaviour of extrudable fresh cementitious pastes/mortars is largely different from that of traditional cement pastes, mortars, concrete, suspension, slurry, self-compacting concrete etc. They are semi-solid, dough-like materials exhibiting almost no fluidity, but high cohesion and pseudo-plasticity under normal condition [11].

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So far there are limited quantitative data available for describing rheological properties of the semi-solid, dough-like fresh cement-based materials for extrusion, mainly due to the complexity of their rheological properties and also lack of appropriate rheology test methods. Alfani and Guerrini [12] reviewed most promising 'non-traditional' rheology test methods for concentrated and cohesive extrudable fresh cement-based materials and found that those 'promising' test methods were initially developed for materials like plastics, rubber, clays, soils and metals, rather than traditional cement pastes or mortars. Zhou et al. [13] summarized the 'non-traditional' rheology test methods for semi-solid, dough-like fresh cement-based materials for extrusion. These test method include ram extrusion [11, 14], capillary extrusion [15], orifice extrusion [13], upsetting and squeezing flow [16-18]. An elasto-viscoplastic constitutive model was proposed by Zhou and Li [19] for simulation of coupled elasto-visco-plastic rheological properties of semi-solid, dough-like cement-based materials for extrusion. The constitutive model was employed for simulating monotonic upsetting [20] and ram extrusion process [21]. However, few researches have been reported on direct measurement of shear yield strength of semi-solid dough-like fresh cementitious materials for extrusion. Therefore, there is great need to explore a suitable test method for direct measurement of shear yield strength of semi-solid, dough-like fresh cement-based materials for extrusion. The direct shear box test is widely used for measurement of shear parameters of geo-materials like soil and clay because of its simplicity in operation and suitability for a wide range of geo-materials including discrete sand and semi-solid continuous clay [22-23]. The direct shear test has become a standard method recommended by British Standards Institute (BSI 1377 part 7: 1990) and American Society of Testing and Materials (i.e., ASTM D3080 / D3080M – 11). In this paper, this technique was employed to investigate the rheology of semi-solid, dough-like fresh cement-based materials tailored for extrusion. To tailor cement-based materials for extrusion, it is vital to figure out effects of individual ingredients on the overall rheological properties of the semi-solid, dough-like fresh cement-based composites.

II. Experimental

A. *Apparatus and test method*

As shown in the Fig. 1(a), the apparatus used for this research is a lever-loading direct shear box (SHEARTEST VJT2760). The vertical and shear displacements are measured by two dial gauges. Shear load is obtained by converting measurement from the horizontal dial gauge attached to the load cell (ring) (see Fig. 1(a)). The test method adopted here conforms to BS 1377 part 7: 1990 which is similar to ASTM

D3080 / D3080M – 11 used for testing clay and soil. Direct shear tests are carried out on square prism specimens of 60 mm square and 20 mm high. The shear box set-up consists of the specimen being sandwiched between the two perforated plates as well as the porous plates and a loading pad (see Fig.

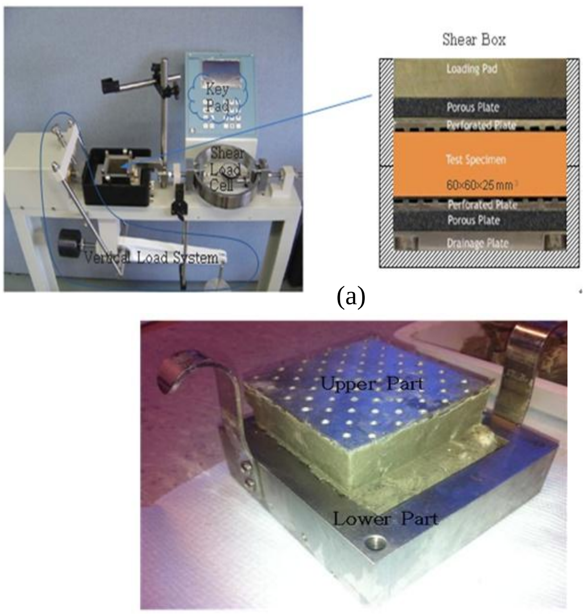


Figure 1. Shear box test set-up & deformed sample

1(a)). During test, the sample is consolidated first under a vertical normal load, i.e., primary consolidation. Upon the completion of the primary consolidation, the sample is sheared horizontally along its mid-height plane (between the two halves of the shear box) at certain displacement rate while subjected to a constant vertical normal stress, i.e. the primary consolidation pressure. The shearing stress is created by imposing a movement of the lower half of the shear box. The shearing resistance offered by the material sample in the shear box is recorded during test. Yielding occurs when the shearing resistance reaches the maximum value which is usually represented by a plateau in the measured shear stress vs. shear displacement curve obtained from experiment. During the test, horizontal displacement rate is usually kept constant. Readings of the horizontal dial gauge attached to the ring are recorded at regular intervals of horizontal displacement.

In order to study the relation between the shear yield strength and vertical normal stress, a Mohr-Coulomb model (Eq. 1) for cohesive material was adopted to interpret experimental data.

$$\tau_f = c' + \sigma_n \tan\varphi' \qquad (1)$$

where c' and φ' are the shear strength parameters referred as the cohesion and the angle of shearing resistance, respectively. The cohesion represents shear strength of the material under zero normal stress. By carrying out direct shear tests of a series of specimens, with the same formulation, but under different values of vertical nominal stress, the relationship between shear yield strength, τ_f, and normal stress, σ_n, can be obtained. If a linear relationship (i.e. Eq. 1) between them is assumed, linear regression analyses can be performed yielding the two shear yield strength parameters c' and φ'. The slope of the fitted line gives the angle of shearing resistance φ', and the intercept gives the cohesion c'. Therefore, direct shear test enables the effective shear strength parameters c' and φ' to be derived for semi-solid dough-like materials on top of shear yield strength directly obtained from it.

B. *Materials and formulations*

Raw materials for preparing the semi-solid, dough-like mixtures included Portland cement CEM II/A-L 42.5 N (PC) from LAFARGE Cement (UK), pulverised fuel ash (PFA) from Scottish and Southern Electricity (UK), 4mm-long polyvinyl alcohol (PVA) fibres from Kuraray Co. Ltd. Japan, limestone powder, rheology modifier in this case WALOCELTM VP-M-7701 hydroxyethyl methyl cellulose (HEMC) from Dow Chemicals (EU), superplasticizer in this case ADVA650 in solution format from Grace Construction Products UK and/or Dellite®43B nanoclay which is a type of nanoclay derived from a naturally occurring montmorillonite specially purified and modified with a quaternary ammonium salt. Dellite®43B is off-white in colour with particle size between 7 to 9 μm. Its bulk density is $0.4g/cm^3$ and specific density $1.6g/cm^3$. The addition of limestone filler into PC can improve hydration rate of cement compounds and consequently increases the strength at early ages. The HEMC rheology modifier is able to improve rheology of semi-solid fresh cement-based materials towards favourite of extrusion, which enables the mixtures reaching dough-like plasticity and enhances their water retention capacity to avoid low-viscosity liquid phase being squeezed out from the mixtures during extrusion. The formulations of the fresh cement-based, dough-like materials for extrusion are listed in Table 1.

Table 1. Formulations of mixtures

	Mix A	Mix F	Mix G	Mix H	Mix I
PC (g)	100	100	100	100	100
PFA (g)	100	100	100	100	100
Limestone powder (g)	150	150	150	150	150
SP (ml)	3.0	3.0	3.0	3.0	3.0
Nanoclay (g)	8.0	8.0	8.0	8.0	8.0
PVA fibre (g)	5.4	10.7	0.0	5.3	5.5
Rheology modifier (g)	3.6	3.6	3.6	3.6	3.6
Water (distilled) (g)	100	100	100	90	110
W/B	0.5	0.5	0.5	0.45	0.55

SP: superplasticizer

In all formulations, the ratio of PC, PFA, and limestone powder was kept constant at 1:1:1.5 by weight, and superplasticizer was added at the dosage of 1.5% by weight of cementitious materials, i.e., cement+PFA. It should be noted that in some researches that limestone is counted as cementiotious material or partially counted as cementitious material, but this is not the case in this study. Rheology modifier HEMC was added at the dosage of 1.0% by weight of solid materials, i.e., PC+PFA+limestone+fibre+nanoclay (Dellite®43B). The difference among Mix A, F and G is the fibre volume ratio which is 2% for Mix A, 4% for Mix F and 0% for Mix G, respectively. Finally, the difference among Mix A, H and I falls in water-to-binder ratio with the value of 0.50, 0.45 and 0.55, respectively.

C. *Samples preparation and direct shear box test*

All fresh mixtures were prepared with a Kenwood Major KM020 bench-top food mixer. First, all dry ingredients, in this case PC, PFA, limestone filler, rheology modifier, nanoclay (Dellite®43B), and PVA fibres were blended by the Kenwood mixer for 3 minutes with the speed of 61 rpm. Then, liquid ingredients in this case water with superplasticizer were added into the mixture. The wet mixture was then blended for another 3 minutes with the speed of 61 rpm. Then, a higher speed (122 rpm) was adjusted for the mixer and the composite was blended for another 5 to 10 minutes till semi-solid dough-like mixture was formed. Then 155g of such dough-like fresh material was taken from the fresh mixture and compressed into the cell of a shear box forming a sample of 60×60×20 mm^3 giving a nominal density of the sample 2150 Kg/m^3.

A set of direct shear tests on the five formulations were carried out in this research. Each formulation was tested under vertical nominal stress (σ_n) of 27.22, 54.44, and 81.67 kPa, respectively, with three samples tested under each value of normal stress for repeatability. Shear displacement rate was kept constant at 2 mm/min for all formulations. All the samples were sheared to the maximum horizontal shear displacement of 17 mm. Fig. 1(b) shows the final deformation of a sample after the completion of shear test. The duration of one single test was around 20 minutes after the sample is prepared, which was consistent with the fact that the freshly prepared cementitious materials are to be extruded out of die and form desirable products in 20 minutes after readily prepared. In total, the whole test of a sample is completed in between 30 to 40 minutes after cement starts contacting water. This time scale was strictly complied with throughout the test program and only one sample was prepared and subsequently tested each time to minimize the possible inconsistency due to change in rheological properties with time and also human being operation. During test, shear displacement, vertical displacement and the reading of the calibrated dial gauge attached to the loading ring were recorded at regular interval. Based on these data, rheological behaviour of the semi-solid fresh cement-based materials for extrusion was characterised.

III. Results and discussion

Linear regression analysis was performed on the shear yield strength with respect to normal stress of the five fresh mixtures. It should be noted that both normal stress (i.e. consolidation pressure) and shear stress are calculated based on the nominal area of the sample, in this case 60×60 mm^2. The correlation coefficients are almost equal to 1.0 in all linear regression analyses indicating the Mohr-Coulomb model is appropriate for the semi-solid, dough-like fresh cement-based materials investigated in this study. Table 2 lists the shear yield strength parameters (c' and φ') of all the five formulations.

Table 2. Shear yield strength parameters of various mixtures

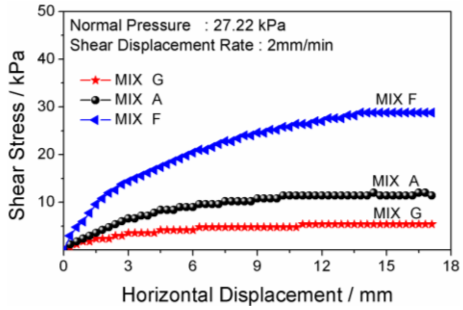
Composite / Parameters	Mix A	Mix F	Mix G	Mix H	Mix I
φ' (°)	6.29	18.87	3.15	22.20	3.15
c' (kPa)	9.21	19.62	3.81	12.82	7.61

A. *Effects of fibres*

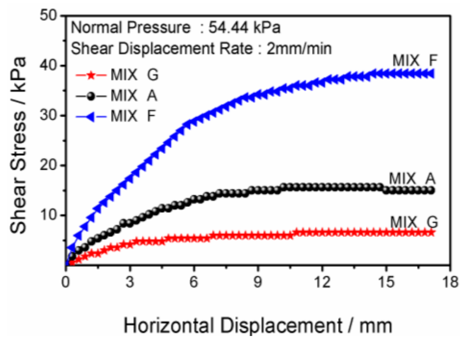
Fig. 2 presents the relationship between shear stress and horizontal shear displacement of Mix G, A and F with 0, 2 and 4% by volume of PVA fibres under different values of vertical normal stress obtained from direct shear box test. With the addition of 2% PVA fibres by volume (Mix A), shear yield strength of the fresh mixture increased 120% compared with the plain one (Mix G without fibres). With further increase of fibres to 4% by volume (Mix F), shear yield strength further increased 140% compared with Mix A (with 2% PVA fibre by volume). The behaviour of the three semi-solid fresh mixtures in consolidation is presented in Fig. 3. Results indicate that as fibre content increases, the mixture gets harder.

As semi-solid cementitious materials are particulate and porous in nature resulting in discontinuous contact in matrix, PVA fibres act as wires in wire reinforced concrete, which consequently strengthens the semi-solid cementitious materials. In addition, the soft PVA fibres entangle after blended with other ingredients making the cementitious composites exhibit higher green strength, in this case shear yield strength. This is beneficial to achieve higher strength once the composites harden which is the main reason of adding short discrete fibres into cementitious composites. But it also increases the difficulties for extrusion. Therefore, in order to balance the demand of appropriate rheological property at fresh state and high strength at hardened state, it is necessary to investigate some effective rheological modifiers to improve rheology of the cementitious composites at fresh state, but not to scarify their strength at harden state.

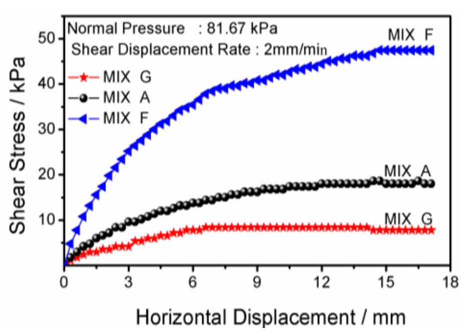
The shear strength parameters (c' and φ') of the Mix A, G and F are shown in Table 2. It can be seen that both shear strength parameters (c' and φ') increase with the increase of fibre content. With fibre volume increases from 0% (Mix G) to 2% (Mix A) and further to 4% (Mix F), the cohesion varies from 3.81 to 9.21 and further to 19.62 kPa and the angle of shearing resistance from 3.15^{o} to 6.29^{o} and further to 18.87^{o}. This further indicates that the increase of shear yield strength of fibre-reinforced semi-solid fresh cement-based materials is directly related to fibre-cement matrix interaction. Inside the semi-solid cementitious materials, the randomly distributed fibres act as spatial network, interlocking with matrix to form a net reinforced structure. This coherent structure increases cohesion (c') and the angle of shearing resistance (φ') of the materials, i.e. the bonding resistance to resist shearing.



(a) Vertical normal stress 27.22 kPa

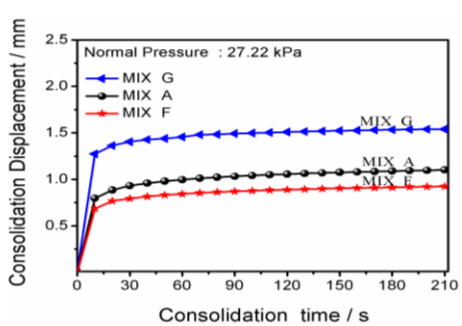


(b) Vertical normal stress 54.44 kPa

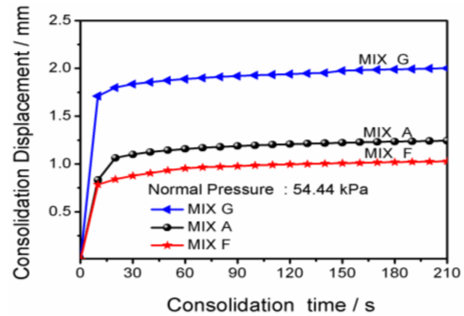


(c) Vertical normal stress 81.67 kPa

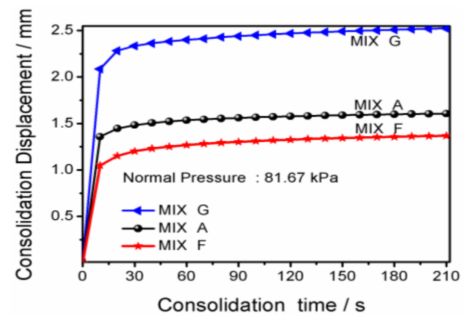
Fig. 2 Shear stress vs. shear displacement of Mix A, F and G



(a) Vertical normal stress 27.22 kPa



(b) Vertical normal stress 54.44 kPa

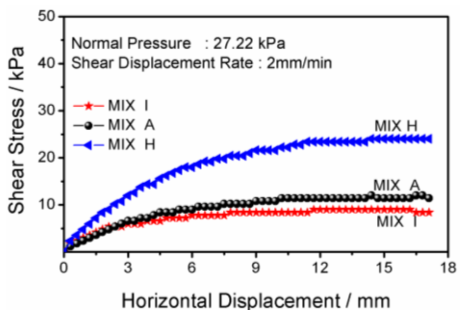


(c) Vertical normal stress 81.67 kPa

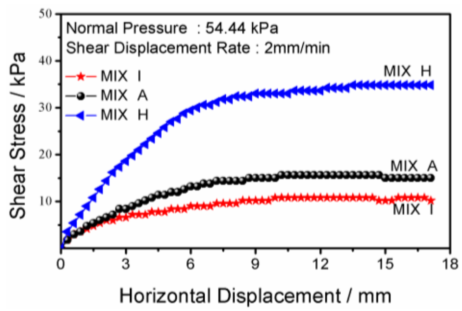
Fig. 3 Consolidation displacement vs. consolidation time of Mix A, F and G

B. ***Effects of water***

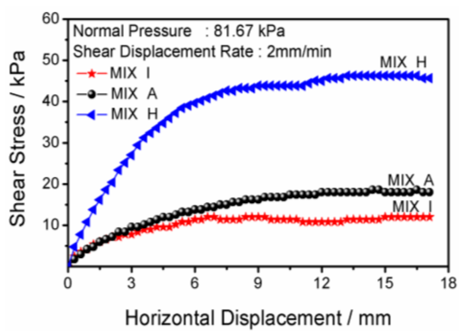
The shear stress vs. horizontal displacement curves of three formulations, i.e., Mix I, A and H with the water-to-binder ratio of 0.55, 0.50 and 0.45, respectively, under various values of consolidation pressure are plotted in Fig. 4. It can be seen that shear strength of the mixtures decreases with the increase in water-to-binder ratio. The shear yield strength of Mix H, with the water-to-binder ratio of 0.45, is around 2-3 times higher than that of Mix I with the water-to-binder ratio of 0.55. The results indicate that shear yield strength of the semi-solid fresh cementitious mixtures for extrusion increases significantly with the decrease in water-to-binder ratio. This can be ascribed to the fact that higher water content increases porosity and decreases strength of the fresh cementitious composites. Thus the adhesion and frictional resistance of the interfacial bond are reduced, leading to lower shear strength. According to Table 2, it can be seen that both shear strength parameters (c' and φ') increase with the decrease of water-to-binder ratio. With water-to-binder ratio increases from 0.45 (Mix H) to 0.5(Mix A) and further to 0.55 (Mix I), the cohesion varies from 12.82 to 9.21 and further to 7.61kPa and the angle of shearing resistance from 22.20^{o} to 6.29^{o} and further to 3.15^{o}. Fig. 5 plots consolidation displacement versus time for Mix I, A, and H. It can be seen that consolidation displacement decrease with the decrease in water-to-binder ratio, indicating the bulk stiffness of the mixture increase with the decrease in water-to-binder ratio which could make the mixture harder for extrusion.



(a) Vertical normal stress 27.22 kPa

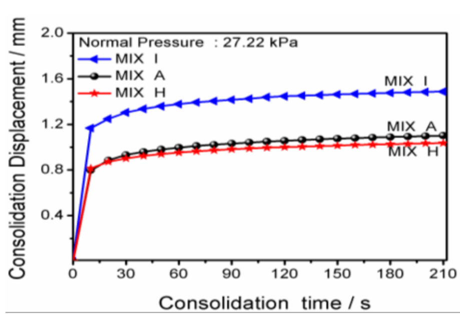


(b) Vertical normal stress 54.44 kPa

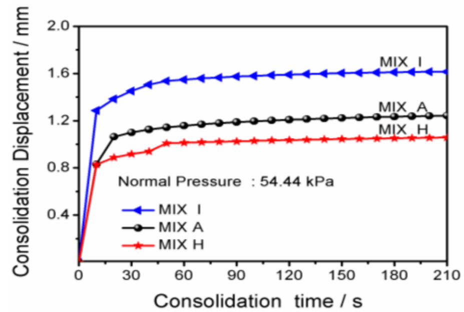


(c) Vertical normal stress 81.67 kPa

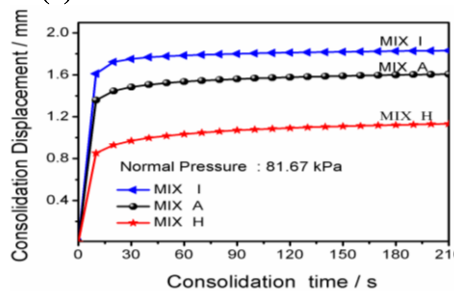
Fig. 4 Shear stress vs. shear displacement of Mix A, I and H



(a) Vertical normal stress 27.22 kP



(b) Vertical normal stress 54.44 kPa



(c) Vertical normal stress 81.67 kPa

Fig. 5 Consolidation displacement vs. consolidation time of Mix A, I and H

IV. Conclusions

Rheological behaviour of semi-solid, dough-like fresh cement-based materials for extrusion was studied through direct shear box test. Nine formulations, with various amounts of nanoclay, fibres and/or water, were tested under various values of vertical normal stress. Based on the study, the following conclusions can be drawn:

(1) Direct shear box test is an appropriate and efficient method for investigating rheological properties for semi-solid, dough-like fresh cement-based materials for extrusion. Its simplicity in instrument and operation means it is efficient for optimizing formulations of cement-based materials for extrusion.

(2) Shear yield strength of the semi-solid fresh cementitious materials is sensitive to fibre content. Shear yield strength, stiffness and shear strength parameters (c' and φ') all significantly increase with the increase of fibre content, leading to increased difficulty for extrusion but likely enhance mechanical properties once the extrudate hardens.

(3) Water is another vital factor controlling rheological behaviour of the semi-solid fresh cement-based materials. Shear yield strength and shear strength parameters (c' and φ') all increase significantly with the decrease of water-to-binder ratio. As water content decreases, the semi-solid fresh cementitious composites become harder for extrusion.

Acknowledgment

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