Computers and Concrete, *Vol. 15, No. 6 (2015) 903-919*
DOI: http://dx.doi.org/10.12989/cac.2015.15.6.903

Sensitivity and accuracy for rheological simulation of cement-based materials

Jae Hong Kim[1a], Hye Rim Jang[1b] and Hong Jae Yim[*2]

[1]School of Urban and Environmental Engineering, Ulsan National Institute of Science and Technology, Ulsan 698-798, Republic of Korea
[2]Department of Construction and Disaster Prevention Engineering, Kyungpook National University, 2559 Gyeongsang-daero, Sangju, Gyeongsangbuk-do, 742-711, Republic of Korea

(Received September 2, 2014, Revised March 4, 2015, Accepted June 1, 2015)

Abstract. The flow of freshly mixed cement-based material shows thixotropy, which implies some difficulties on robust measurement of its rheological properties: The flow curve of thixotropic materials depends on the used protocol. For examples, higher viscosity is obtained when the rate of shear strain is more quickly increased. Even though precise measurement and modelling of the concrete rheology needs to consider the thixotropic effect, engineers in the concrete field prefer considering as a non-thixotropic Herschel-Bulkley fluid, even more simply Bingham fluid. That is due to robustness of the measurement and application in casting process. In the aspect of simplification, this papers attempts to mimic the thixoropic flow by the non-thixotropic Herschel-Bulkley model. Disregarding the thixotropy of cement based materials allows us to adopt the rheological concept in the field. An optimized protocol to measure the Bingham parameters was finally found based on the accuracy and reproducibility test of cement paste samples, which minimizes the error of simulation stemming from the assumption of non-thixotropy.

Keywords: cement paste; suspension; thixotropy; rheology; flow simulation; VOF techniques

1. Introduction

The workability of concrete affects the quality of its freshly mixed and hardened state. The performance of concrete structures is consequently dependent on how to control the workability of concrete and obtain quality assurance. In a construction field, the slump of concrete (ASTM C143), a measure of its workability, is importantly controlled and monitored. The slump is, however, the result of an empirical test for evaluating the properties of freshly mixed concrete. Its scientific background is weak. Nevertheless, its robustness and wide adoption in almost all construction sites allow it to be considered a standard test. For flowable concrete, such as self-consolidating concrete (SCC), the slump flow test (ASTM C1611) is applied even though it has the same weak point.

The use of SCC triggers studying the rheology of concrete, which makes it possible to simulate

[*]Corresponding author, Assistant Professor, E-mail: yimhongjae@gmail.com
[a] Assistant Professor, E-mail: jaekim@unist.ac.kr
[b] Graduate Student, E-mail: kaerin21@gmail.com

http://www.techno-press.org/?journal=cac&subpage=8 ISSN: 1598-8198 (Print), 1598-818X (Online)

casting and placing processes of concrete structures. Flow of SCC in a form could be predicted by means of computational fluid dynamics. Flow blockage and insufficient filling are expected to be predicted before execution. The rheological simulation also predicts slump flow of concrete (Chidiac and Habibbeig 2005) and evaluates its casting performance pertinent for producing complex-shape girders or plates. Roussel *et al.* (2007) reviewed three fresh concrete flow simulation methods to predict a concrete flow: volume-of-fluid (VOF), discrete particle flow, and suspension flow techniques. Among them, the VOF technique is widely used especially for evaluating the filling and passing ability of fresh concrete. The technique assumes that the freshly mixed cement-based material is a single fluid and uses the flow curve as a material property.

One of the most important material properties for the rheological simulation is the flow curve. It is defined as the relationship between the rate of shear strain (in the unit of s-1) and the shear stress (in the unit of Pa). It is usually obtained by using a rotational rheometer. Consistent and accurate measurement of the flow curve of fresh concrete has been studied for long time (NISTIR 6819, NISTIR 7154), still it is not easy to be concluded. Homogeneity of a construction material needs high volume of measurement. Theoretically, the sufficient volume is reportedly more than 1 m3. Inert aggregates locally disturb the consistent stress field on the fluid flow to be measured. Therefore, so-called concrete rheometers, specialized for highly condensed suspension having wide-scale inclusions, are big and need high volume of concrete sample (a few tens of liters). They also adopt a vane rotor to apply a wide range of nonsingular shear field. Nevertheless, the current technology guarantees consistent measurement only by the use of a single device.

On the other hand, measuring the flow curve of binder paste can plausibly adopt a general rheometer used for chemical and mechanical engineering materials. Note that the rheological properties of cement paste in a freshly mixed concrete determine the flow of concrete. The particle size of Portland cement is generally less than 200 μm, which is acceptable to be tested with general gap size of measuring geometries. The current issue on the cement paste rheology is developing a standard protocol for the rheological measurement. That is obviously the first step in a science-based approach to casting and placing process of concrete construction. For the purpose, a test standard guide was proposed by ASTM C1749 recently. The guide provides an open form of protocol that a user should detail with respect to the interested application. This paper proposes the optimized protocol for the case of gravity-induced flow. The targeted flow behavior for simulation is both mini-slump flow and channel flow tests.

The engineers in the field prefer considering concrete as a non-thixotropic Bingham fluid because of the robustness of the measurement and application. Accordingly, the optimized protocol for measurement of concrete flow is essentially needed to obtain the rheological properties of concrete. The optimized protocol can also be applied in the field to predict concrete flow, and it can be used for precisely simulating of gravity-induced flow based on the measured yield stress and plastic viscosity.

2. Volume-of-fluid simulation

A rheological model idealizes the flow curve generally measured by using a rotational rheometer. The Herschel-Bulkley model reportedly well describes the flow curves of cement-based materials under the assumption of non-thixotropy (de Larrard *et al.* 1998). The Herschel-Bulkley model is a nonlinear function that relates shear stress (τ) to the rate of strain rate ($\dot{\gamma}$)

$$\tau = \tau_0 + k\dot{\gamma}^n \tag{1}$$

where τ_0 is the yield stress, k is a flow consistency parameter and n is an exponent. For a simple response of a material, the zero-shear stress can be ignored (τ_0=0) and the model complexity is reduced and represented by k and n only, which is called a Power-law fluid model. The most widely used model for cement-based materials is Bingham fluid model, which is particular case of Herschel-Bulkley model (n=1), and it is described by two representative rheological properties such as the yield stress (τ_y=τ_0) and the plastic viscosity (η_p=k). A linear response of fluid (τ_0=0 and n=1) is the most simply idealized by Newtonian fluid model with the viscosity (η=k). More scientific models to consider the thixotropy of non-Newtonian fluid was developed in the literature (Roussel 2005, Roussel 2006, Wallevik 2006), but here only non-thixotropic models are explained for application in engineering practice.

Prior to the investigation of cement-based materials, a reference material was investigated for verifying the accuracy of VOF simulation. The used reference material was carbomer gel (polyacrylic acid) dilution, which is a non-Newtonian non-thixotropic fluid. The flow curve of the reference material was measured using a parallel-plate geometry. The gap size and the diameter of plates were 1 mm and 25 mm, respectively. The measured data and their model fitting can be found in Fig. 1. The measured data points were well fitted into either Power-law or Herschel-Bulkley model. Table 1 reports the fitting parameters based on each model. For simplicity, the Power-law model will be used for the following simulation.

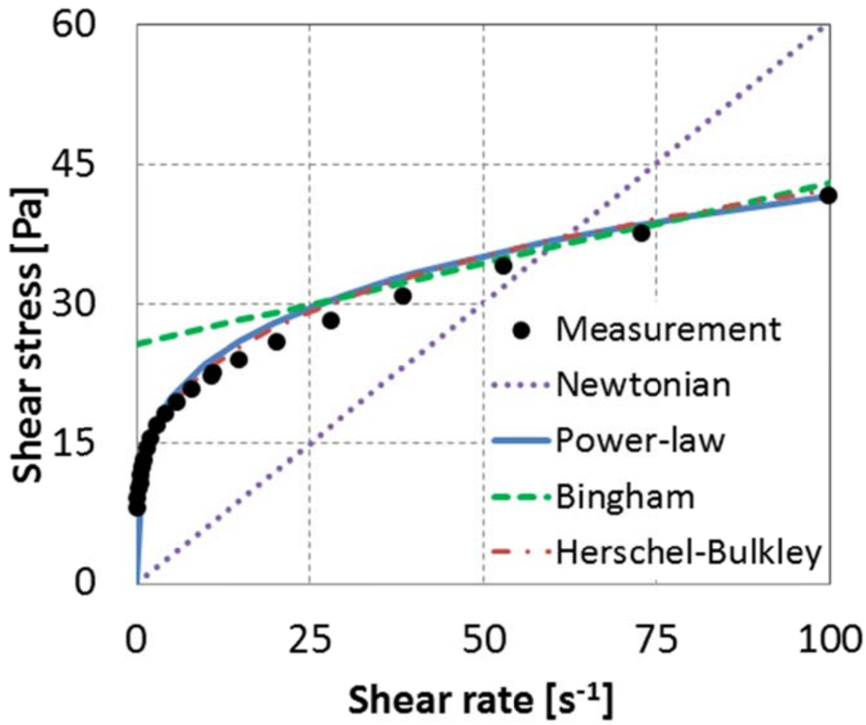
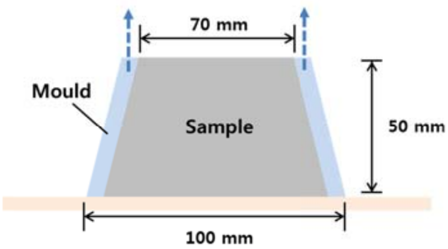


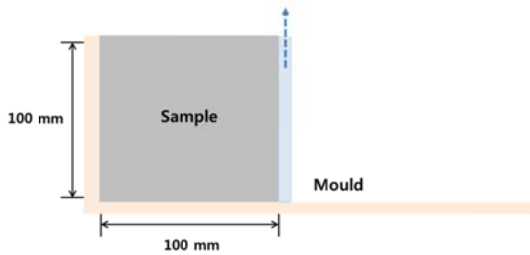
Fig. 1 Flow curve of the reference material

Table 1 Rheological models for the reference material

Model	Fitting parameters
Newtonian fluid	$\tau = 0.603\dot{\gamma}$
Power-law fluid	$\tau = 13.4\dot{\gamma}^{0.245}$
Bingham fluid	$\tau = 25.7 + 0.173\dot{\gamma}$
Herschel-Bulkley fluid	$\tau = 2.57 + 10.5\dot{\gamma}^{0.289}$



(a) mini-slump cone



(b) channel box

Fig. 2 Configuring the test equipment

The flow problems to be simulated are both the mini-slump and the channel flow tests. Fig. 2 shows a mini-slump cone and a channel box containing a sample. A sample of 0.27 L is filled in the mini-slump cone and then lifting the cone makes it flow due to gravity. The mini-slump flow test is widely performed with a flow table cone (ASTM C230, 0.1 m diameter at the bottom), and the diameter of its final spread represents consistency of a mix. On the other hand, the channel flow test is a modified type designed to show higher sensitivity on the fluidity, where its final spread length shows large variation in a small change in rheology of a mix. A sample of 1.0 L is filled in the channel box (0.1 m × 0.1 m × 0.1 m) and lifting a vertical plate allows the sample to flow. Both tests let a sample flow by its self-weight: gravity-induced flow.

The VOF technique allows us to track free surface of fluid flow. The field equations of fluid flow velocity, $\boldsymbol{v}$, is given by Navier-Stokes equation,

$$-\frac{1}{\rho} + \frac{\mu}{\rho}\nabla^2\vec{v} + \vec{g} = \frac{D\vec{v}}{Dt} \tag{2}$$

where ρ is the density of the fluid, μ is its tangential viscosity defined by Lamé constants, g is the gravity acceleration, and D/Dt indicates material derivative. A so-called Eulerian volume fraction, C, is additionally defined in each Eulerian element. The volume fraction is a scalar variable indicating the fluid volume fraction of each element. If C=1.0 the element is fully filled with a specified fluid. If $0<C<1$ the element is partially filled and C=0 indicates empty element. The volume fraction is governed by an advection equation,

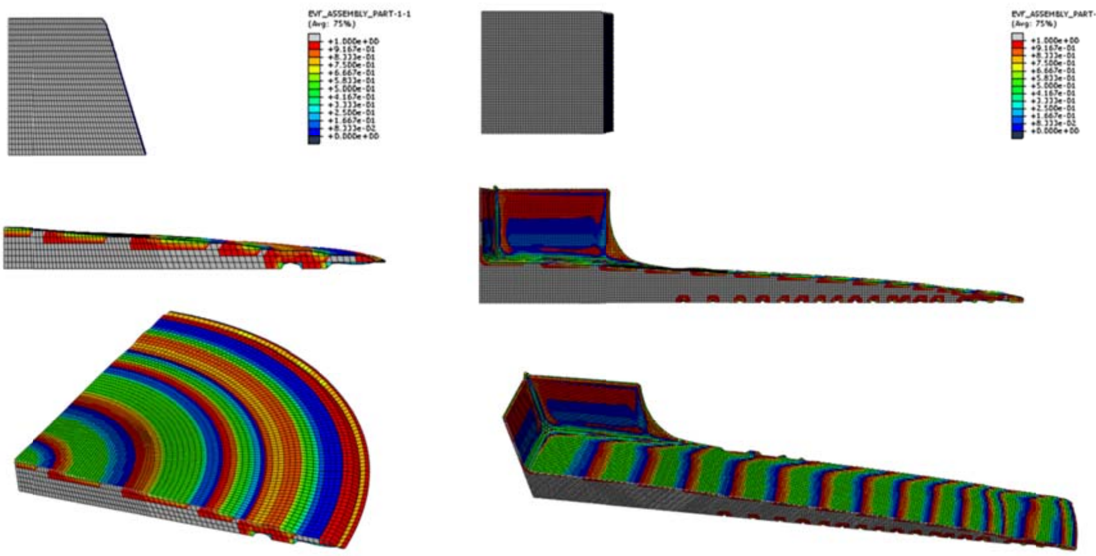
$$\frac{\partial C}{\partial t} + \vec{v}\cdot\vec{\nabla}C = 0 \tag{3}$$

The above two equations can be solved by finite element method. The strong form of the equations is converted to their weak forms, and the method of weighted residuals evaluates the velocity vector and volume fraction for each element at each incremental step. The result of volume fraction composes a discontinuous function in the field. The discontinuity is released by geometrical reconstruction of the surface of the fluid. Linear slope for the two-dimension model or planer facet for 3-dimensional model is widely used. Updating fraction functions in each control volume reformulates the initial condition and then solving the field equations is repeated for specified time of flow.

In this study, the VOF simulation was performed using a commercialized software, ABAQUS 6.10. The elements were 8-node linear Eulerian brick (EC3D8R). Preliminary simulation was conducted to verify an acceptable mesh size. For the mesh test, a Newtonian fluid model in Table 1 was used: η=0.603. The side walls and bottom plate of the model were restrained; the displacement on the surface is fixed to all direction. The gravity acceleration was input for gravity-induced flow. On the post-processing step, filling of each element was determined by a criterion of 50% volume ratio of fluid. Fig. 3 shows the snap shot of the flow simulation.

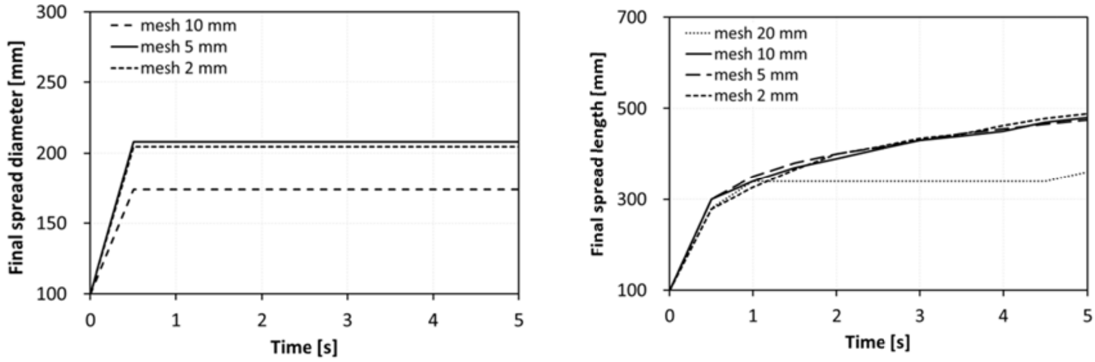
The spread curves of both tests are reported in Fig. 4. As it can be observed in Fig. 4 the spread curve of mini-slump flow is similar if the element size is larger than 5 mm. That of channel flow is consistent for larger than 10 mm elements. The current modeling of the circular fluid front, as well as its thin layer in the mini-slump flow, needs finer mesh. Thus, the models discretized by the 5 mm and 10 mm elements were respectively used. The number of elements for each test was 3,808 and 2,700 elements, respectively.

The simulation using the accepted mesh is compared with experiment results in Fig. 5. The experimental results were extracted from snap shots of the tests. The final mini-slump flow was 220 mm, and the final spread of channel flow was 470 mm. To determine the material properties of the reference material, the Power-law fluid model was applied, and the obtained properties(k=13.4, n=0.245) are described in Table 1. Finally, error on the mini-slump flow test can be found in the final flow: 10 mm difference. The reliability of mini-slump flow is found in the error



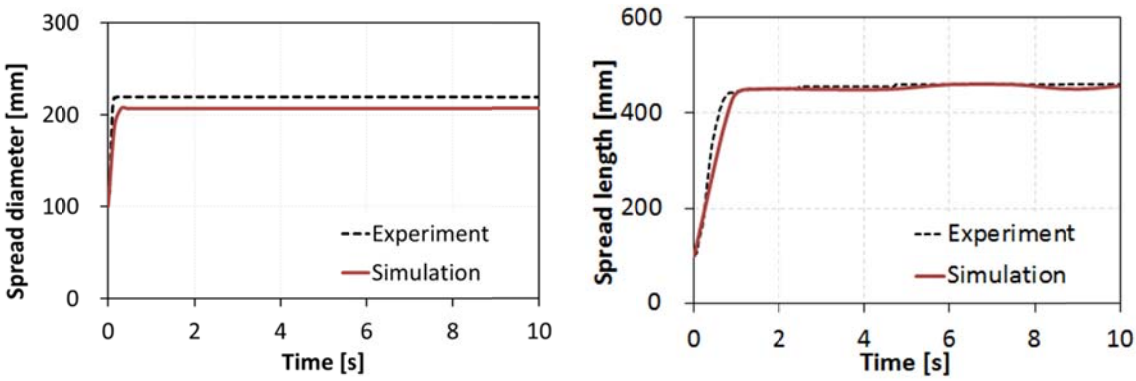
(a) mini-slump flow test (b) channel flow test

Fig. 3 Simulation of VOF technique which model was designed by 2 mm of mesh



(a) mini-slump flow test (b) channel flow test

Fig. 4 Spread curve analyzed simulation as size of mesh



(a) mini-slump flow (b) channel flow

Fig. 5 Spread curves of the reference material

Table 2 Oxide composition of the cement.

Oxide	CaO	SiO_2	Al_2O_3	SO_3	MgO	Fe_2O	K_2O
Percent (%)	63.5	19.3	4.6	3.9	3.3	33.1	1.1

bound of 10 mm; the difference can be considered within the variation of the experimental results. On the other hand, error of the channel flow test occurs on the damping of flow. The time to get the final channel flow was approximately 0.5 s on the experiment, but it took approximately 1.0 s in the simulation: 0.5 s difference. The difference of both results seems to be due to the effect of inertia and it was hard to reproduce the initial time of channel flow related to the inertia effect. For example, the speed of lifting the gate of the channel flow changes the inital spread. Disregarting the marginal difference on the intial time of spread allows us to get the following reverification: The VOF simulation is acceptable to trace the gravity-induced flow of non-thixotropic non-Newtonian fluid, carbomer gel in this study.

Table 3 Mix proportions

Label		Hand mixing of 36 mL	Mechanical stirrer of 90 mL	High shearing of 90 mL	Planetary mixer of 1,500 mL
C40	Water (g)	20	50	50	837
	Cement (g)	50	125	125	2,090
C50	Water (g)	22	55	55	-
	Cement (g)	44	110	110	-
C60	Water (g)	24	59	59	-
	Cement (g)	39	98	98	-
C40A20	Water (g)	20	50	50	837
	Cement (g)	50	125	125	2,090
	HWRA (g)	0.100	0.251	0.251	4.182
C40A35	Water (g)	20	50	50	837
	Cement (g)	50	125	125	2,090
	HRWRA (g)	0.176	0.439	0.439	7.315

3. Experiment

3.1 Sample preparation

A total of 5 cement paste samples were prepared to measure their rheological properties. The variation of the samples was on (1) water-to-cement ratio, *w*/*cm*=40%, 50%, and 60%; and (2) high-range water-reducing admixture (HRWRA) dosage, 0.20% and 0.35% of the cement content by mass. Ordinary Portland cement was used for all the samples. Its specific gravity and Blaine number were 3.14 and 3,320 cm^2/g, respectively. The oxide composition was described in Table 2. The HRWRA was incorporated to enhance the fluidity of the last two samples. That was polycarboxylates based, and its solid content was 22%.

The rheological properties of cement-based materials also depend on the mixing method and protocol. A total of 4 mixing methods were considered for each sample. (1) The first method was hand mixing with a small glass beaker and stick. The rotational speed was kept constant manually. Not to confirm it accurate but it was about 280 rpm. (2) A mechanical stirrer was used with the same rotational speed of 280 rpm. The vane was a conventional 4 rectangular blades. (3) A planetary mixer corresponding to ASTM C 305 practice was used, and its rotational speed was also 280 rpm approximately. Due to the volume of the mixing bowl a large volume of the samples, 1.5 L, was produced in the mixing method. (4) Finally, the standard practice of mixing for the rheological test, ASTM C 1738, was considered where the use of a high-shear mixer (up to 12,000 rpm) was needed. The high shearing supposedly eliminates the thixotropy of the materials and is expected to yield a reliable measurement.

ASTM C 1738 specifies the mixing protocol. The most important guide on the mixing is the limitation of continuous mixing within 0.5 min due to heat release by high shearing of cement suspension. Following the limitation but keeping the total mixing time constant with the other mixing method, the protocol of high-shear mixing was slightly adjusted: (1) mixing for 0.5 min; (2) scraping on the upper part of the mixing bowl for 4 min; (3) additional mixing for 0.5 min, and (4) placing for 3 min. The total mixing time was the same for all mixing types: 5 min and then 3 min placing. However, when the hand mixing, the stirrer and the planetary mixer were used, the direct mixing time was extended to produce a homogeneous sample: (1) mixing for 2 min; (2) scraping for 1 min; (3) additional mixing for 2 min; (4) placing for 3 min.

3.2 Conventional protocol for flow curve measurement

A standard guide recently proposed by ASTM C1749 specifies a procedure to measure the flow curve of cement paste samples. The rheological test for the hydraulic cementitious paste starts at the minimum shear strain rate and would be progressed by increasing the shear rate up to maximum speed and then descending to the lowest speed serially. Each rate is kept for 20 s or until the measured shear stress is converged to a certain value. The duration of each step can be adjusted according to thixotropic characteristics of a sample. Finally, a converged value of shear stress is plotted at each shear rate and then the best fit for a rheological model is found by regression analysis.

The standard is an open protocol far from robust application, and a specific protocol has to be agreed upon. The following important criterion is given to user's choice. Firstly, it does not provide the range of the rate of shear strain. How fast a concrete flow in the field determines the rate of shear strain of a sample. For example, concrete pumping applies more than 400 s^{-1} even though it depends on the pumping rate. The gravity-induced flow is in the range of a few tens reciprocal seconds. Secondly, consideration of the thixotropic effect is not fully understood. A maximum 20 s duration for the flow curve measurement step is expected to minimize the thixotropic effect, but it was not fully verified. Based on the following test results, 20 s is not enough to get rid of the thixotropic effect of a cement paste sample.

The weak points of the standard protocol would be reported prior to developing an optimized protocol. A conventional protocol is shown in the Fig. 6. The step time was set to 20 s as on ASTM C1749. The range of the shear rate includes the interest of gravity-induced flow as from the previous simulation. The minimum and maximum values of the shear strain range are not provided by the conventional protocol, but using a stepwise protocol is the only rule to be considered.

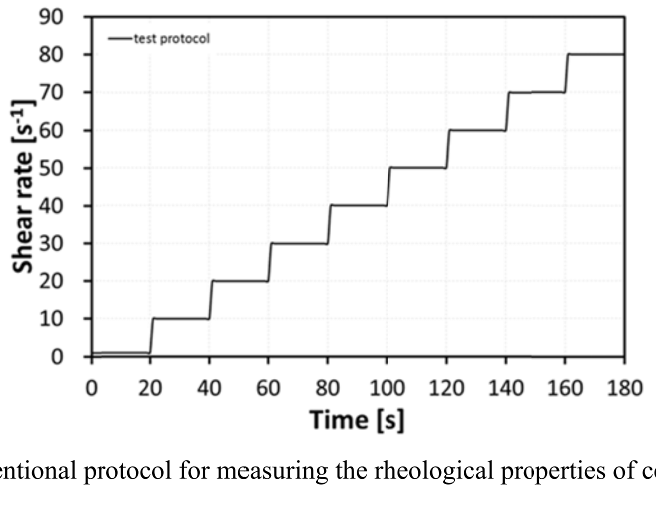
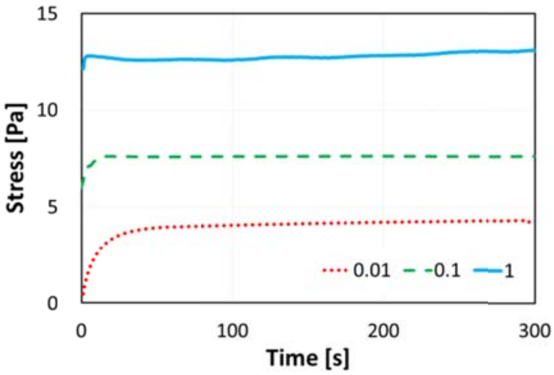
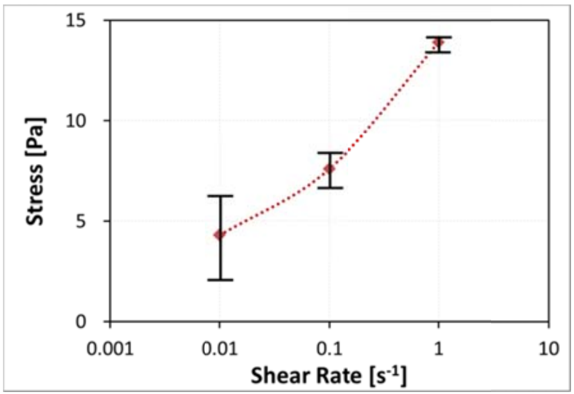


Fig. 6 Conventional protocol for measuring the rheological properties of cement paste



(a) at a constant shear rate of 0.01 s^{-1}, 0.1 s^{-1}, and 1 s^{-1}



(b) error bar at each shear rate

Fig. 7 Shear stress measurement of the reference material

3.3 Results by the conventional protocol

3.3.1 Systematic error with non-Newtonian fluid

The study measured the rheological properties using a rotational rheometer (HAAKE MARS III, Thermo Fisher Scientific Inc.). A gap size between the parallel-plates is 1 mm and used plate geometry is changed. When a non-Newtonian fluid is tested with a rheometer, a certain time is needed to get a stable shear stress for an applied rate of shear strain. The step time of a protocol should be about 20 s in order to release systematic error and, therefore, get the stable measurement. The reference non-Newtonian fluid, carbomer gel, as well as cement paste, shows the unstable measurement when an applied share rate is transiently imposed.

Fig. 7 shows the transient fluctuation at a step of 0.01 s^{-1}, 0.1 s^{-1}, and 1 s^{-1}, where the reference non-Newtonian fluid was tested. Parallel plates having the diameter of 20 mm were used. The gap size between the two plates was 1 mm. The low shear rate measurement took more time to get a stable shear stress. When the shear rate of 0.01 s^{-1} is applied, 58 s needed for a convergence of the shear stress. However, in the case of 1 s^{-1}, 4 s are enough for convergence. A slope of 0.01 Pa/s of the shear stress was set as a convergence criterion. Note that a Newtonian fluid such as standard mineral oil instantaneously shows a stable measurement.

If the step time of 20 s as the conventional protocol in Fig. 6 is adopted, the shear stress fluctuation could be represented by an error bar at each shear rate as shown in Fig. 7(b). The error bar at a low shear rate is very wide. We confirmed that the transient fluctuation is not negligible for the other sensors: the parallel plates having 35 mm diameter and the concentric cylinder having 25 mm diameter were tested, and the same phenomena were found. Conclusively, at such a low rate of shear strain (0.01 s^{-1}) the systematic error was found, but it almost vanished at rate of 1 s^{-1}.

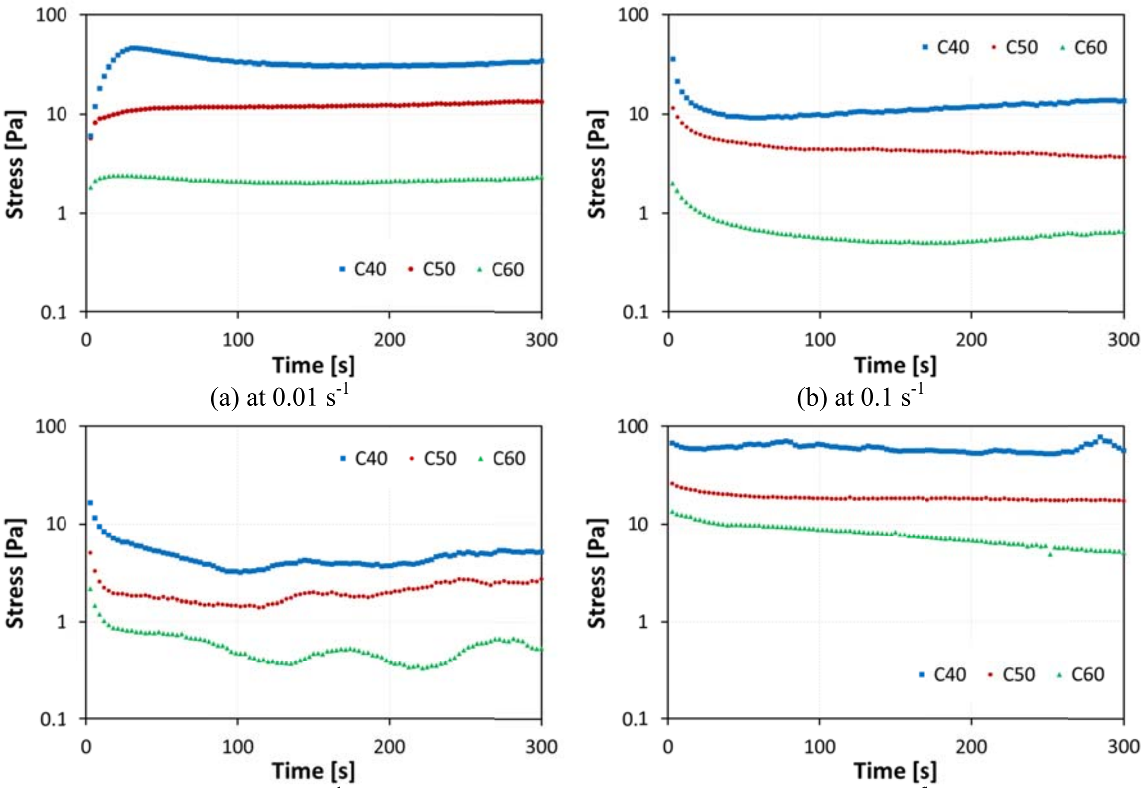
Even though the reference fluid is not thixotropic, it shows the transient fluctuation and, therefore, takes time to get the convergence of shear stress. Shear thinning fluid has a higher viscosity at a lower shear rate; the degree of transient fluctuation was enlarged at the low shear rates.

3.3.2 Systematic error and thixotropy with cement paste

Cement paste is expected to show thixotropy in addition to the transient fluctuation. The single step of specific shear rate was also applied to the pastes for 5 min with the parallel plates sensor used in the previous test. The gap size between two plates was 1 mm which is 5 times larger than the maximum particle size of cement to be tested. Fig. 8 shows the shear stress measurement of mixes C40, C50 and C60. At all ranges of the rate of shear strain, higher resistance (higher shear stress) was measured with a lower *w*/*cm*, as expected.

At the highest shear rate of 100 s^{-1}, as shown in Fig. 8(d), almost a constant shear stress was measured for mixes C40 and C50, but that of C60 monotonically decreased. The monotonic decrease for C60 was due to shear-induced migration, which was observed at the side of the parallel plates during the test. Such a water-dominated suspension is likely to migrate at a high shear rate. On the other hand, the constant shear stress measured on mixes C40 and C50 indicates that the transient fluctuation and thixotropic effect are negligible at the high rate of shear strain.

When the rate of shear strain decreases up to 1.0 s^{-1} and furthermore 0.1 s^{-1}, as shown in Figs. 8 (b) and (c), the thixotropy is dominantly observed in all samples. The shear stress decreases over time and converges to a certain value, which is due to the breakage of cement flocs (Yim *et al.* 2013). The time to get the convergence depends on the mix proportion and the applied shear rate. For example, at the shear rate of 1.0 s^{-1} the thixotropy vanished after 78 s, 23 s, or 16 s has passed for mix C40, C50, or C60, respectively. The thixotropy-dominant time is definitely larger than the step time (20 s) of the conventional protocol, which results in causing variability of the flow curve measurement.



(a) at 0.01 s^{-1}

(b) at 0.1 s^{-1}

(c) at 1.0 s^{-1}

(d) at 100 s^{-1}

Fig. 8 Shear stress measurement of the cement pastes

At the lowest shear rate of 0.01 s^{-1}, as shown in Fig. 8(a), the transient fluctuation was observed. The shear stress increased up to 30 s as the reference non-Newtonian fluid showed the same phenomenon. A thick mix C40 then shows slight thixotropy with decrease-and-convergence pattern of shear stress measurement. The transient fluctuation and thixotropy were measured at the low shear rate. If one needs to measure a stable shear stress, the step time will be at least 130 s, 40 s, or 12 s for mix C40, C50, or C60, respectively.

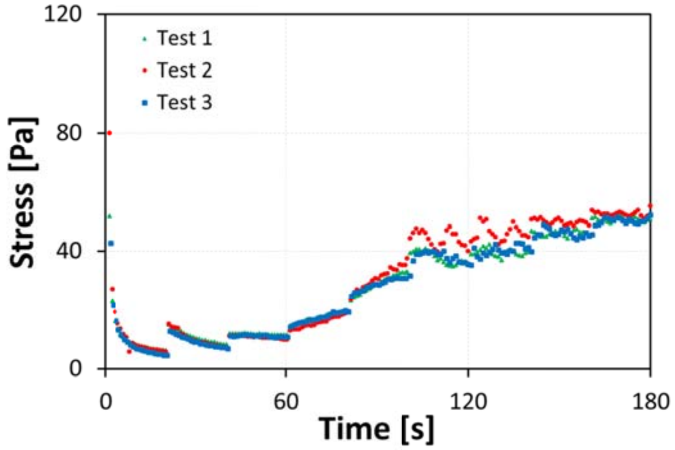
3.3.3 Reproducibility of the stepwise protocol

Further examination of the systematic error and thixotropy was accomplished by applying the conventional protocol in Fig. 6. Fig. 9 shows the measurement results of a superplasticized cement paste (C40A35), where the parallel plate sensor of 35 mm diameter was used. A total of 3 samples mixed in the same mix proportion, 40% *w/cm* and 0.35% HRWRA dosage, were distinguished by their mixing method: hand mixing at approximately 280 rpm, mechanical stirrer at 280 rpm, and high shear mixer at 12,000 rpm. The rheological properties of cement-based materials depend on the mixing process. High shearing on the mix used increases the resistance to flow and is expected to provide high yield stress and plastic viscosity on fitting of Bingham fluid model. The results of hand mixing and mechanical stirrer are comparable with a similar mixing speed of 280 rpm, but they are not identical. The thixotropy of each sample was still observed at low rates of shear strain (1 s^{-1} and 10 s^{-1} corresponding to 0 to 40 s measurement). It is worthwhile to remark that the thixotropy still prevails with 20 s steps, and it does with the sample produced even by high shear mixing. As already expected, the thixotropy of cement paste cannot be disregarded by any testing protocol to measure the rheological properties.

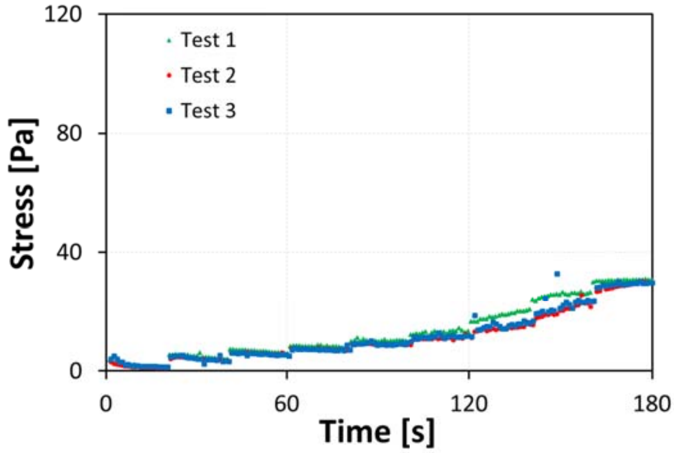
Each sample was tested 3 times for evaluating its reproducibility. All replicated samples showed an identical response to the applied protocol, but only small scattering could be found at the high rate of shear strain, 60 s^{-1} to 80 s^{-1} corresponding to 120 s to 180 s (see Fig. 6). Low-speed mixing is supposed to leave very small agglomerates in a mix, which results in inhomogeneity at high-velocity field. The small scatters are thought to be negligible by eliminating bias points. Therefore, it is concluded that the stepwise protocol together with any mixing method discussed in the study provides sufficient reproducibility so as to apply the fitting of a Bingham fluid model.

Two more mix proportions, C40 and C40A20, were considered to evaluate the reproducibility of the stepwise protocol. Each mix was produced by the 3 different mixing methods again. Similar response to those in Fig. 9 was obtained, and then the shear stress reading at the end of 20 s step was taken at the applied shear rate. A shear stress-rate relationship was developed by Bingham fluid model as shown in Fig. 1. The reproducibility of the stepwise protocol and mixing process was evaluated by testing a total of 10 replicated samples. Figs. 10 and 11 show the results of reproducibility tests.

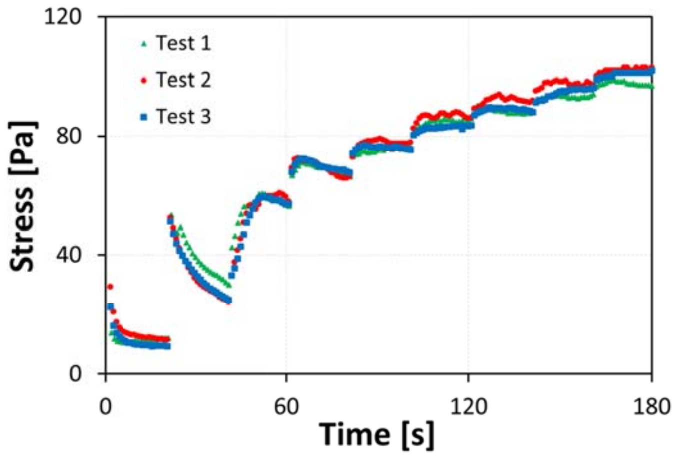
Compared to hand mixing and mechanical stirrer, high shear mixing provides higher yield stress and plastic viscosity. Especially for the neat cement paste, scatters on the yield stress measurement were enlarged with high shear mixing. High shearing thickened the sample and caused the clumping of cement powders in the suspension, which results in high values of the result and unstable measurement of rheology. When an HRWRA was used, the thickening and clumping is weakened even by high shear mixing. The measurement of yield stress and plastic viscosity were generally reproducible as shown in Fig. 11. Overall, low speed mixing such as hand mixing and mechanical stirrer, combined with the stepwise protocol, provides a good reproducibility to determine the rheological properties of cement paste.



(a) Hand mixing

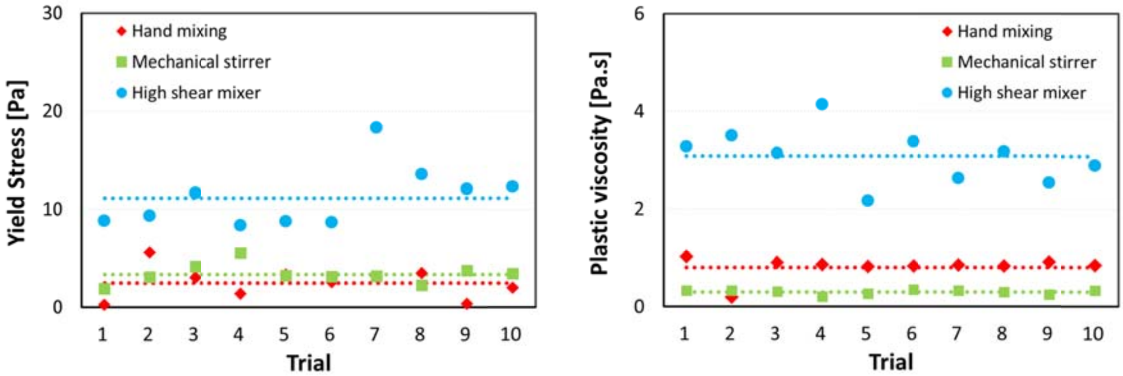


(b) Mechanical stirrer



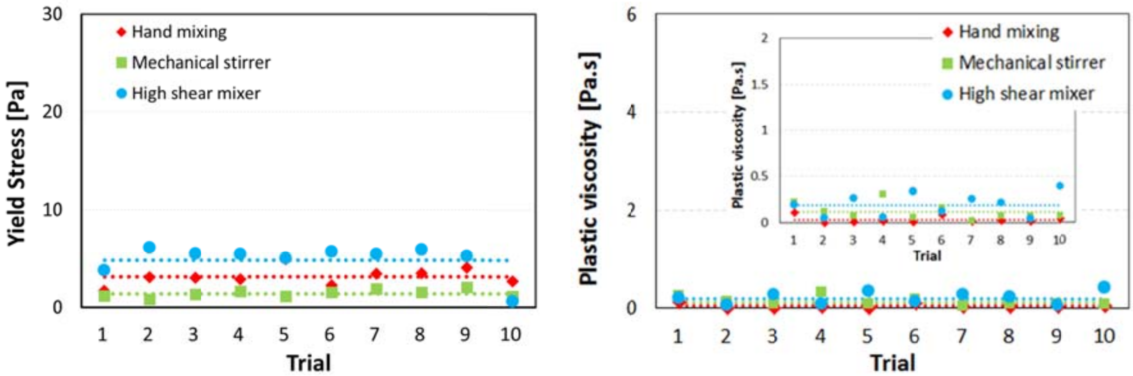
(c) High shear mixer

Fig. 9 Thixotropy of a superplasticized cement paste (C40A35)



(a) yield stress (b) plastic viscosity

Fig. 10 Reproducibility of a neat cement paste (C40)



(a) yield stress (b) plastic viscosity

Fig. 11 Reproducibility of a superplasticized cement paste (C40A20)

4. Optimized protocol for gravity-induced flow

In the previous sections, it was confirmed that the experimental result measured by use of the conventional protocol includes the effect of transient fluctuation and thixotropy of sample. Two phenomena affect the accuracy of the measurement. It still remains to be checked whether the obtained rheological properties predict the flow of cement-based materials even though the reproducibility of the measurement was verified. In this study, the optimized protocol is proposed to guarantee the accuracy of gravity-induced flow such as the slump flow test. For the purpose two important variables, which were not specified by the conventional test standard, should be given: (1) the range of shear strain to be tested and (2) the step duration of the stepwise protocol.

This paper focuses on the gravity-induced flow. The channel flow of the reference fluid, 470 mm in Fig. 5(b), was revisited and reanalyzed with the limits of shear rate. Limiting the range of shear rate finds its sensitive range to the flow behavior. The lower limit was onset of applying the power-law fluid model, as can be shown in Fig. 1, while the flow curve in the range less than the lower limit was set to a constant (1.0 Pa). When the lower limit of shear rate was set less than 0.1 s^{-1}, the final spread of channel flow was accurately simulated at 470 mm as shown in Fig. 12(a).

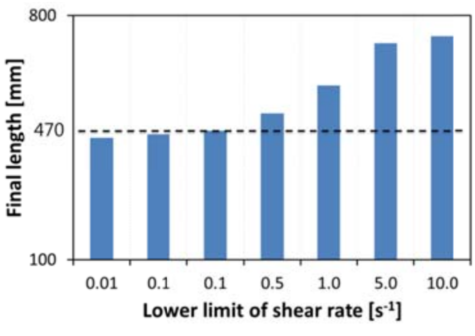
However, the accuracy was lost with more than 0.5 s^{-1}. On the other hand, on Fig. 12(b), the upper limit of shear rate was evaluated. The power-law model was applied up to the upper limit, but the flow curve higher than the upper lit was set to a constant (100 Pa). As can be seen in Fig. 12(b) the accuracy of the simulation never be disturbed by the assigned range of the upper limit. Therefore, the range of 0.1 s^{-1} (lower limit) to 1 s^{-1} (upper limit).

In addition, gravitational drop of a sample at the beginning of slump flow test is found on the simulation result. On the simulation contour there exist elements sheared by more than 10 s^{-1}. Covering various cases of the gravitational drop needs the response at a higher rate of shear strain. Up to 50 s^{-1}, the rate of shear strain is reportedly high enough to represent a concrete flow induced by gravity (Tregger 2010). Therefore, the range of shear strain is concluded 0.1 s^{-1} to 50 s^{-1} to simulate a gravity-induced flow.

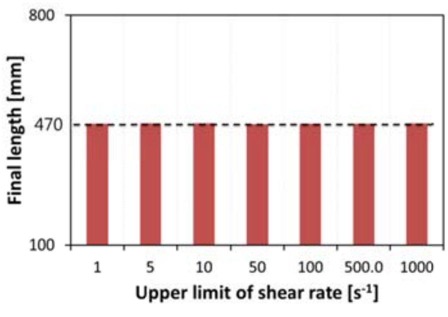
On the other hand, the thixotropy of cement paste is not removed with the conventional step duration, max 20 s, as investigated in the previous section. Again limiting a problem within gravity-induced flow allows us to specify a protocol to measure the non-thixotropic rheological properties that can simulate the thixotropic flow of cement-based materials. The step level and duration of a protocol needs to be adjusted so as to mimic a given flow test. A variety of step level and durations were tested accurately to simulate the channel flow of two samples. One is C40 showing 155 mm mini-slump flow and 355 mm channel flow, and the other is C40A20 showing 210 mm mini-slump flow and 495 mm channel flow. The planetary mixer was used to produce both samples because more than 1 L is needed to accomplish both the channel flow test and the rheological measurement together. Among trial step level and durations the best fit for the simulation can be found as follows

- The duration of each step is 5 s, which complies with ASTM C 1749. The max step time is 20 s. In addition, 5 s is enough to disregard a transient fluctuation occurred at higher than 0.1 s^{-1}.
- The rates of shear strain are 0.01 s^{-1}, 0.1 s^{-1}, 1 s^{-1}, 3 s^{-1}, 5 s^{-1}, 10 s^{-1}, 20 s^{-1}, 30 s^{-1}, 40 s^{-1}, and 50 s^{-1}, to be sequentially applied. A pseudo step of 0.01 s^{-1} is added to the range of interest, 0.1 s^{-1} to 50 s^{-1}. The pseudo step releases the transient fluctuation, and it is excluded for fitting of Bingham fluid model.
- The descending rates of shear strain are continuously applied if necessary. That is also noted in ASTM C 1749.

Fig. 13 shows the optimized protocol, where the x-axis is the time for testing and y-axis is the rate of shear strain assigned. A total time of rheological test is then 100 s.



(a) lower limits of shear rate



(b) upper limits of shear rate

Fig. 12 Range of shear rate at the field experiment

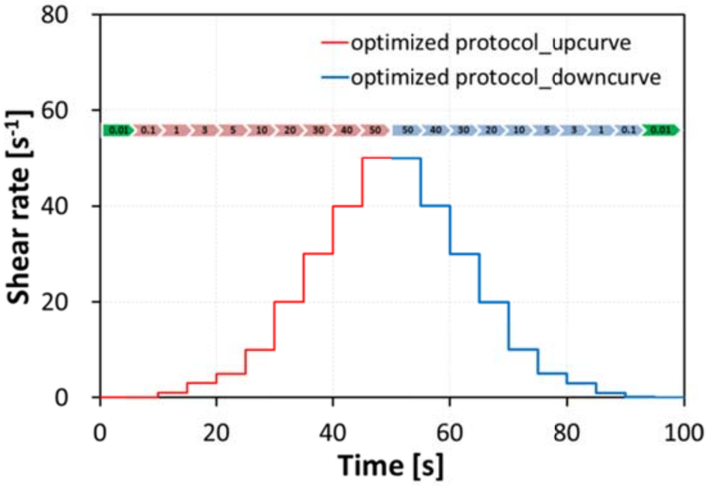
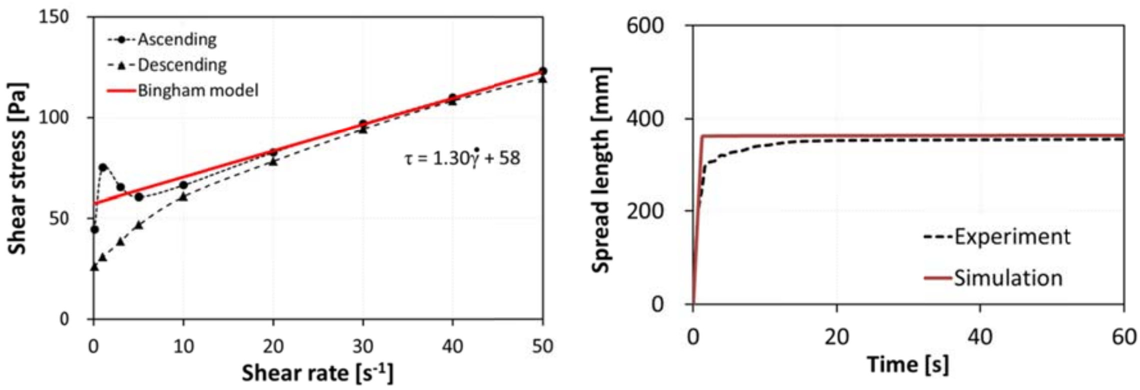
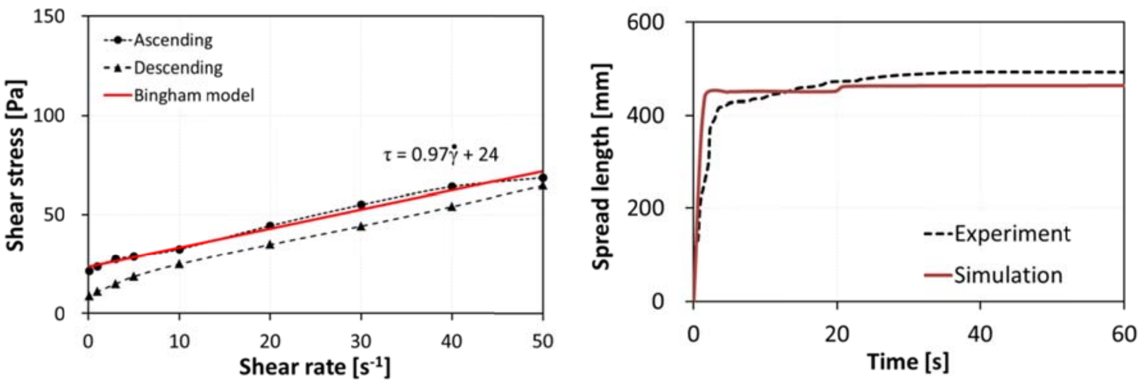


Fig. 13 Optimized protocol to match gravity-induced flow



(a) flow curve (b) simulation

Fig. 14 Flow curve and simulation result of a neat cement paste (C40)



(a) flow curve (b) simulation

Fig. 15 Flow curve and simulation result of a superplasticized cement paste (C40A20)

The optimized protocol was applied using the parallel-plates geometry. The gap size and the diameter of plates were 1 mm and 25 mm, respectively. In order to disregard slippage on the plates, sand paper was attached on the plate surfaces. The measured rheological properties and the simulation result for the channel flow test are given by Figs. 14 and 15. Due to the high thixotropy of neat cement paste a local peak is observed at the shear strain rate of 1 s^{-1} on the ascending measurement. Curve fitting to the Bingham model gave the yield stress of 58 Pa and the plastic viscosity of 1.30 Pa·s. In the case of superplasticized cement paste, the flow curve is relatively better suited for Bingham model. The yield stress of 24 Pa and the plastic viscosity of 0.97 P·s. Finally, the evaluated rheological properties predict the spread of channel flow within the standards of accuracy.

5. Conclusions

The rheology of cement-based materials predicts flow of fresh concrete and the performance of placing process. Yield stress and plastic viscosity are two important material properties, but a test standard to measure the properties does not agree yet. In order to develop a protocol to measure the rheological properties in all mix-proportions of cement paste, this paper evaluated the accuracy and reproducibility of the measurement using various cement paste samples and simulated gravity-induced flow with the measured parameters.

The first consideration of the measurement protocol was the transient fluctuation and thixotropy. The transient fluctuation occurs when a non-Newtonian fluid is tested. Releasing the transient fluctuation needs more than 4 s step duration at shear rates higher than 0.1 s^{-1}. The shear rate of 0.1 s^{-1} is also critical to simulate gravity-induced flow accurately. A material experiences 0.1 s^{-1} to 1.0 s^{-1} when such a flow gets stopped. Therefore, an optimized protocol for accurate simulation of gravity-induced flow includes the range of shear rate, and the duration of stepwise protocol is set higher than 4 s. The optimized protocol reflects the foregoing consideration, but the thixotropy cannot be neglected in the measurement. Therefore, the protocol is found to provide the least error between the simulation and experimental results. The optimized protocol to measure the yield stress and plastic viscosity is recommended for the simulation of gravity-induced flow such as slump flow test.

Acknowledgments

This research was supported by Basic Science Research Program through the National Research Foundation of Korea (NRF) funded by the Ministry of Education, Science and Technology (NRF-2015R1A1A1A05001382).

References

ABAQUS 6.10 Finite Element Analysis, SIMULIA.
ASTM C 143-12 (2012), *Standard Test Method for Slump of Hydraulic-Cement Concrete*, American Society for Testing and Materials (ASTM) International, West Conshohocken, PA.
ASTM C 1611-05 (2005), *Standard Test Method for Slump Flow of Self-Consolidating Concrete*, American

Society for Testing and Materials (ASTM) International, West Conshohocken, PA.
ASTM C1749-12 (2012), *Standard Guid for Measurement of the Rheological Properties of Hydraulic Cementious Paste Using a Rotational Rheometer*, American Society for Testing and Materials (ASTM) International, West Conshohocken, PA.
ASTM C230/C230M-13 (2013), *Standard Specification for Flow Table for Use in Tests of Hydraulic Cement*, American Society for Testing and Materials (ASTM) International, West Conshohocken, PA.
ASTM C305-13 (2013), *Standard Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency*, American Society for Testing and Materials (ASTM) International, West Conshohocken, PA.
ASTM C1738/C1738M-14 (2014), *Standard Practice for High-Shear Mixing of Hydraulic Cement Pastes*, American Society for Testing and Materials (ASTM) International, West Conshohocken, PA.
Chidiac, S.E. and Habibbeigi, F. (2005), "Modelling the rheological behaviour of fresh concrete: An elasto-viscoplastic finite element approach", *Comput. Concr.*, **2**(2), 97-110.
Comparison of Concrete Rheometers: International Tests at LCPC (Nantes, France) in October, 2000, (Eds. Ferraris, C.F. and Brower, L.E.), National Institute of Standards and Technology, NISTIR 6819, 2001.
Comparison of Concrete Rheometers: International Tests at MB (Cleveland OH, USA) in May, 2003, (Eds. Ferraris, C.F. and Brower, L.E.), National Institute of Standards and Technology, NISTIR 7154, 2004.
de Larrard, F., Ferraris, C.F. and Sedran, T. (1998) "Fresh concrete: a Herschel-Bulkley material", *Mater. Struct.*, **31**, 494-498
Roussel, N., (2005), "Steady and transient flow behaviour of fresh cement pastes", *Cement. Concrete. Res.,* **35**(9), 1656-1664.
Roussel, N. (2006), "A thixotropy model for fresh fluid concretes: Theory, validation and applications", *Cement. Concrete. Res.,* **36**(10), 1797-1806.
Roussel, N., Geiker, M.R., Dufour, F., Thrane, L.N. and Szabo, P. (2007) "Computational modeling of concrete flow: general overview", *Cement. Concrete. Res.*, **37**(9), 1298-1307.
Tregger, N.A. (2010), "Tailoring the Fresh State Properties of Concrete", Ph.D. Dissertation, Northwestern University, Evanston IL.
Wallevik, J.E. (2006), "Relationship between the Bingham parameters and slump", *Cement. Concrete. Res.,* **36**(7), 1214-1221.
Yim, H.J., Kim, J.H., Shah, S.P. (2013), "Cement particle flocculation and breakage monitoring under Couette flow". *Cement. Concrete. Res.,* **53**, 36-43.

CC