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Standard Guide for Measurement of the Rheological Properties of Hydraulic Cementious Paste Using a Rotational Rheometer[1]

This standard is issued under the fixed designation C1749; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 This guide covers description of several methods to measure the rheological properties of fresh hydraulic cement paste. All methods are designed to determine the yield stress and plastic viscosity of the material using commercially available instruments and the Bingham model. Knowledge of these properties gives useful information on performance of cement pastes in concrete.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *This guide offers an organized collection of information or a series of options and does not recommend a specific course of action. This document cannot replace education or experience and should be used in conjunction with professional judgment. Not all aspects of this guide may be applicable in all circumstances. This ASTM standard is not intended to represent or replace the standard of care by which the adequacy of a given professional service must be judged, nor should this document be applied without consideration of a project's many unique aspects. The word "Standard" in the title of this document means only that the document has been approved through the ASTM consensus process.*

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C305 Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency

C511 Specification for Mixing Rooms, Moist Cabinets, Moist Rooms, and Water Storage Tanks Used in the Testing of Hydraulic Cements and Concretes

C1738 Practice for High-Shear Mixing of Hydraulic Cement Pastes

E2975 Test Method for Calibration or Calibration Verification of Concentric Cylinder Rotational Viscometers

2.2 *Other Standards:*

API Recommended Practice 10B Testing Well Cements, American Petroleum Institute, Washington, DC (1997)

ISO 10426-2 (2003) Petroleum and Natural Gas Industries—Cements and Materials for Well Cementing—Part 2: Testing of Well Cements—Section 5.2

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology C125 and C219.

3.2 *Definitions of Terms Specific to This Standard:*[3,4]

3.2.1 *apparent viscosity, n*—the shear stress divided by rate of shear, in units of Pa.s.

3.2.2 *plastic viscosity, n*—in the plastic (Bingham) model, the slope of the shear stress – shear rate curve, in units of Pa.s.

3.2.3 *thixotropy, n*—a decrease of the apparent viscosity under constant shear stress or shear rate followed by a gradual recovery when the stress or shear rate is removed.

3.2.4 *yield stress, n*—the stress corresponding to the transition from elastic to plastic deformation, in units of Pa; it is also referred to as the stress needed to initiate flow. It would be calculated using the Bingham model in this guide.

[1] This guide is under the jurisdiction of ASTM Committee C01 on Cement and is the direct responsibility of Subcommittee C01.22 on Workability.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] H.A. Barnes, J.F. Hutton and K. Walters, An Introduction to Rheology, Elsevier (1989).

[4] Hackley V.A., Ferraris C.F., "The Use of Nomenclature in Dispersion Science and Technology" NIST Recommended Practice Guide, SP 960-3, 2001.

***A Summary of Changes section appears at the end of this standard**

3.2.5 *Bingham model, n*—a rheological model for materials with non-zero yield stress and a linear relationship between shear rate and shear stress, following the equation: $\tau = \tau_B + \dot{\gamma}\eta_{pl}$; where τ_B Yield stress in Pa, $\dot{\gamma}$ Shear rate in 1/s, τ Shear stress in Pa, and η_{pl} Plastic viscosity in Pa.s.

4. Significance and Use

4.1 Rheological properties determined using this guide include plastic viscosity and yield stress as defined by the Bingham model and apparent viscosity.

4.2 Rheological properties provide information about the workability of hydraulic cementitious paste. As an example, the yield stress and plastic viscosity indicate the behavior of a specific cement paste composition. As another example, the apparent viscosity indicates what energy is required to move the suspension at a given strain rate. This test may be used to measure flowability of a cement paste or the influence of a specific material or combination of materials on flowability.

4.3 Rheological properties may be sensitive to the procedure being used. This guide describes procedures that are expected to provide reproducible results.

5. Summary of Guide

5.1 This guide provides procedures for the determination of rheological properties of fresh cement paste using a rotational rheometer with geometries, such as parallel plate, narrow-gap and wide gap concentric cylinders.

6. Interferences

6.1 Rheological properties may be sensitive to the procedure, so a comparison of properties obtained using different procedures is not recommended, unless relative viscosity (ratio between the plastic viscosity of a materials and the plastic viscosity of a reference material, both measured using the same rheometer) is considered.

6.2 Rheological properties may be sensitive to the shear history of the sample, so comparison of properties using different mixing procedures is not recommended.

6.3 Paste mixtures (water and cement particles) that are very fluid may yield erroneous data using this procedure due to settling of particles. Such settling is especially likely in shear thinning and thixotropic mixtures.

6.4 Larger cement particles or aggregations of cement particles may block flow in a narrow-gap rheometer and thereby increase the shear stress. The gap between the shearing surfaces needs to be selected with consideration of the particle size of the material to be tested. Depending on the gap size, it may be necessary to remove larger particles by sieving or otherwise prevent segregation.

6.5 Incorporation of air in the paste during mixing reduces viscosity and increases flow.

6.6 The time of testing after initial contact of cement with water influences the results.

7. Apparatus

7.1 *General Description:*

7.1.1 The apparatus shall be a rotational rheometer in which the sample is confined between two surfaces (called the shearing surfaces), one of which is rotating at a constant rotational speed, Ω and the other being stationary. The apparatus shall measure both the rotational speed and the torque required to maintain that speed.

7.1.2 The rheometer geometry shall provide a simple shearing flow (laminar, without turbulence). Allowable geometries and their equations for computing stress and strain rate from the measured values of rotational speed and torque are described in 7.4.

7.2 The rotational rheometer shall be capable of measuring shear stress at strain rates in the range from 0.1 s^{-1} to 600 s^{-1}. The range of shear rates will be selected by the operator depending on the geometry used. At least five measurements need to be recorded.

NOTE 1—Most experiments found in the literature do not use the full range of shear rates prescribed here. For example, most parallel plate measurements are done between 0.1 s^{-1} to 50 s^{-1}. The selection of the shear rate range might take into account the exact geometry of the rheometer.

7.3 Regularly check the calibration and zeroing of the apparatus, as discussed in 7.9.

7.4 *Rheometer Geometry:*

7.4.1 The rheometer geometries described in this section provide simple shearing flow, essential for reliable computation of stress and strain rates. The equation for computation of stress and strain rates is given for each geometry.

NOTE 2—The following assumptions were made to develop the equations that appear in this section: *(1)* the fluid is homogeneous, *(2)* slip at the wall is negligible, and *(3)* the flow regime is laminar.

7.4.2 Selection of the geometry of the rheometer. Three geometries are described here: narrow-gap concentric cylinders, wide-gap concentric cylinders, and parallel plates. The selection of the geometry should be based on the type of rotational rheometer available. One criterion to select between the narrow-gap and the wide-gap should be based on the maximum size of the particles in the cement tested.

7.4.2.1 *Narrow-Gap Concentric Cylinder*—With this type of rheometer, the sample is confined between two concentric cylinders of radii R_1 and R_2 (R_2>R_1), one of which, the rotor, is rotating at a constant rotational speed Ω and the other is stationary. The rotation of the rotor in the presence of the sample produces a torque that is measured at the wall of the inner cylinder. The cylinder radii should be selected such that the shear stress is uniform across the gap. This condition is assumed to be satisfied if:

$$\left(\frac{R_1}{R_2}\right) >= 0.92 \quad (1)$$

where R_1 is the radius of the inner rotating cylinder (m) and R_2 is the radius of the outer stationary cylinder (m).[5]

To prevent slip (development of a liquid layer at the wall of the rotating cylinder that produces an anomalously low stress), the surface of cylinders may be serrated or at least rendered

[5] DIN 53019-1:2008, Viscometry—Measurement of viscosities and flow curves by means of rotational viscometers—Part 1: Principles and measuring geometry.

rough by attaching a sand paper, sand blasting, or other methods that roughen the surface such as serration.

The nominal shear rate and stress are calculated at the inner cylinder wall by the following expression:

$$\dot{\gamma} = \frac{R_2 \times \Omega_1}{R_2 - R_1} \quad (2)$$

where $\dot{\gamma}$ is strain rate (s^{-1}) and Ω_1 is rotational speed at the inner cylinder (r/s). The nominal shear stress is calculated at the inner cylinder wall by the following expression:

$$\tau = \frac{\Gamma}{2\pi R_1{}^2 L} \quad (3)$$

where τ is shear stress (Pa), Γ is torque (Nm), L is cylinder length (m), and R_1 is the inner radius (m). These equations assume that the slurry is homogeneous, the shear stress is uniform in the gap, the flow regime in the gap is laminar, and slip at the wall is negligible.

7.4.2.2 *Wide-Gap Concentric Cylinder*—This type of rheometer is similar to the narrow-gap concentric cylinder described in 7.4.2 except that there is no limit on the gap value and the gap is larger. Computation of strain rate and stress is simplified if it is assumed that the material follows a power-law model. In that case, the nominal shear rate, $\dot{\gamma}$, is calculated at the inner cylinder wall by the following expression:

$$\dot{\gamma} = \frac{2 \times \Omega_1}{n(1 - b^{2/n})} \quad (4)$$

where $\dot{\gamma}$ is strain rate (s^{-1}), Ω_1 is the rotational speed at the inner cylinder (rad/s), b is the ratio of the inner to the outer radius, and n is the power-law exponent. A procedure for determining the value of n is presented elsewhere.[3] Nominal shear stress, τ, is calculated at the inner cylinder wall by the following expression:

$$\tau = \frac{\Gamma}{2\pi R_1{}^2 L} \quad (5)$$

where τ is shear stress (Pa), Γ is torque per unit length (Nm), L is cylinder length (m), and R_1 and R_2 are inner and outer cylinder radii (m).

Some concentric cylinder rheometers use an extreme wide gap such that the radius of the outer cylinder approaches infinity and ($1\text{-}b^{2/n}$) approaches unity. This type of rheometer normally operates only at moderately low shear rates, typically 0.1 s^{-1} to 10 s^{-1}. For a material following a power-law model, the nominal shear rate is calculated at the inner cylinder wall by the following expression:

$$\dot{\gamma} = \frac{2 \times \Omega_1}{n} \quad (6)$$

where $\dot{\gamma}$ is strain rate (s^{-1}), Ω_1 is the rotational speed of the inner cylinder (rad/s), and n is the power-law exponent. Nominal shear stress, τ, is calculated at the inner cylinder wall by the following expression:

$$\tau = \frac{\Gamma}{2\pi R_1{}^2 L} \quad (7)$$

where τ is shear stress (Pa), Γ is torque (N.m), L is cylinder length (m), and R_1 is inner cylinder radius (m).

7.4.2.3 *Parallel Plate*—In this type of rheometer the sample is held between two parallel horizontal plates, each equal and circular cross section. The plates may be serrated to avoid slippage. When one of the plates is rotating and the other is stationary, the shear rate varies from zero at the center to a maximum at the rim, and the value at the rim is:

$$\dot{\gamma} = \frac{R \times \Omega_1}{h} \quad (8)$$

where $\dot{\gamma}$ is strain rate (s^{-1}), R is the plate radius (m), Ω_1 is the rotational speed (rad/s), and h is the gap between the two plates (m). Viscosity is given by:

$$\eta = \frac{3h\Gamma}{2\pi R^4 \Omega_1 \left(1 + \frac{1 d \ln\Gamma}{3 d \ln\Omega_1}\right)} \quad (9)$$

where η is viscosity (Pa.s) and Γ is the torque (N.m).

7.5 *Gap*—The gap between the shearing surfaces of the rheometer should be wide enough that the sample is homogeneous throughout or be of the same magnitude of the distance between aggregates in concrete (typically 0.4 mm). If the gap is too narrow relative to the size of particles in the cement paste (less than 10 times the maximum particles size), the torque will be very high or even the plate will lock and not rotate.

7.6 *Slippage*—Slippage can occur if the shearing surfaces are smooth, due to the formation of layer of water near the surface. If slippage occurs, the torque measured is smaller than it should be. It could be even zero. Therefore, some precaution should be taken to avoid slippage by serration of the shearing surfaces. It can be done either by gluing a sand paper, or by sand blasting the surfaces or by serration of the surface with grooves or a pattern.

7.7 *Evaporation*—Prevent evaporation of water from the paste by covering the paste with a vapor barrier or a water-saturated material.

7.8 *Temperature Control*—Control the temperature to the nearest 2°C. The temperature may be selected to reflect the temperature at which the cement paste would be used in the field.

7.9 *Verification*—Periodically follow the procedures suggested by the manufacturer, or use Test Method E2975 to assure the repeatability of the measurements. Using any standard oil, as recommended by the rheometer manufacturer, would allow detection of malfunctioning of the instrument.[6]

NOTE 3—Another option would be to use a Standard Reference material such as SRM 2492[7].

8. Procedure

8.1 Details of the test procedure may be varied as necessary to suit the specific apparatus.

[6] Ferraris, C.F., Geiker, M., Martys, N.S., and Muzzatti, N., "Parallel-plate Rheometer Calibration Using Oil and Lattice Boltzmann Simulation," J. of Advanced Concrete Technology, Vol. 5 #3, October 2007, pp. 363-371.

[7] Olivas A., Ferraris C.F., Guthrie W.F., Toman B., "Re-Certification of SRM 2492: Bingham Paste Mixture for Rheological Measurements", NIST SP-260- 182, August 2015, https://www-s.nist.gov/srmors/view_detail.cfm?srm=2492.

8.2 Prepare sufficient cementitious mixture (Note 4) to fill the rheometer tool selected. Mix the paste following a method that ensures full dispersion of the cement in water, such as Practice C1738. Describe the mixing method in the report (Note 5). Record the time of first contact between water and the cementitious materials.

NOTE 4—There is a practical limit to the water-to-cement ratio (w/c) that can be tested in a rheometer. If the w/c is too low, the paste will exceed the upper torque limit of the rheometer and may not maintain contact with the rotating shearing surface. The lower w/c value depends on the cement particle size distribution, on the rheometer, and on the degree to which particles have been dispersed using a water reducing admixture. If the w/c is too high, the paste will segregate during the measurement, the cement particles will fall to the bottom of the rheometer and the remaining suspension will be increased in w/c, causing an incorrect measurement of the properties.

NOTE 5—Mixing by hand or using procedures described in Practice C305 is not appropriate to ensure good dispersion of the material.

8.3 Pour the cement paste immediately into the rheometer and set the cup and bob or parallel plates to the correct operating position. Record the time when the measurement is started.

NOTE 6—While setting the operating position, gently rotating the tool either at the lowest speed or manually. This operation minimizes gelation and ensures uniform distribution of the paste.

8.4 Maintain the measuring tool at the test temperature within ± 2°C for the duration of the test. If the rheometer does not provide active temperature control, measure the temperature of the paste in the rheometer before taking the first reading.

8.5 Sweep of shear rate in a pre-selected range to calculate plastic viscosity and yield stress using a suitable model such as Bingham.

8.5.1 Take the initial torque reading 20 s after continuous rotation at the lowest speed. Take all remaining torque readings first in order of ascending strain rate and then in order of descending strain rate. Rotate continuously for 20 s at each speed before taking each reading or when the torque is stable. Shift to the next speed immediately after each reading.

8.5.2 Take readings up to a selected maximum strain rate or rotational speed. Five to ten levels of shear rates or rotational speeds shall be selected for each ramp.

8.5.3 Report the rheological measurements as a plot of shear stress or torque versus shear rate or rotational speed, and report the elapsed time from initial contact of water and cement until rheological testing commenced.

8.5.4 For improved reliability of the measurements, repeat the entire procedure several times using a freshly prepared paste. If the procedure is repeated several times, report each curve and the average of all the acceptable measurements.

9. Calculation and Interpretation of Results

9.1 Convert the raw data (rotational speeds and torque readings) to strain rate and shear stress using equations for the specific rheometer geometry (Section 7). Select a rheological model that best fits the data, either by regression analysis or by inspection of a plot of stress versus strain rate. The most commonly used equation to describe the rheological properties of hydraulic cementitious mixtures is Bingham, as described in 3.2.5.

9.2 Calculate the rheological properties, yield stress, plastic viscosity using the Bingham law using the equation described in 3.2.5.

10. Report

10.1 The report should include all relevant information about the paste tested and the rheometer used. In particular for the rheometer:

10.1.1 the type of rheometer geometry and dimension of the tools,

10.1.2 test temperature (either the controlled temperature or the temperatures measured at the beginning and the end of the test),

10.1.3 time of mixing and of testing,

10.1.4 stress and strain rate data, in the form of a table or graph,

10.1.5 the fitted model parameters, for example, plastic viscosity and yield stress, and

10.1.6 other relevant information on the test performed, such as material used , mixing method, or other model used if not Bingham.

11. Keywords

11.1 hydraulic cement; rheology; viscosity; yield stress

SUMMARY OF CHANGES

Committee C01 has identified the location of selected changes to this standard since the last issue (C1749 – 17) that may impact the use of this standard. (Approved May 1, 2017.)

(1) Added Test Method E2975 to Section 2 ("Referenced Documents").

(2) Added reference to Test Method E2975 to 7.9.

(3) Revised language in 4.2, 8.5.2, and 9.1.

Committee C01 has identified the location of selected changes to this standard since the last issue (C1749 – 12) that may impact the use of this standard. (Approved March 15, 2017.)

(1) Added new Note 3.