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Viscosity of coating clay slurry

1. Scope

1.1 This method describes a procedure for the determination of the low- and high shear viscosity of coating clays. This is accomplished by the preparation of a completely dispersed 70% solids aqueous clay suspension with incremental introduction of dispersant to obtain the optimum dosage (minimum viscosity) for the low and high shearing rates.

1.2 At 70% solids' content, not all clay slurries are sufficiently fluid to permit viscosity determinations with the usual instruments. The test as written is thus substantially limited in its applicability to the type of coating clays suitable for high solids coating. A similar test procedure using lower solids content is informative in the case of clays such as calcined and delaminated coating clay that are not suited to testing at 70% solids. In each case the solids content should be maintained at as high a level as possible to accentuate differences between the clays in question.

1.3 Coating clays are available in predispersed slurry form, generally at 69.5-70.5% solids. The procedure for determining the viscosity characteristics of these clay slurry shipments is also incorporated in this method.

2. Significance

2.1 Aqueous clay slurries usually display non-Newtonian types of flow. It is therefore necessary to control carefully the rate of shear in order to measure flow properties reproducibly. Because the viscosity varies with the rate of shear, it is highly desirable to measure flow properties at low and high shearing rates. Unfortunately, no single instrument is readily available that will measure viscosity under both conditions. A rotating spindle apparatus operating at a maximum torque of 0.7187 mN•m (7187 dyne-cm) is used for low-shear measurements. A rotating bob apparatus calibrated to measure torque in 10 mN•m (7187 dyne-cm x 10³) at increasing shear rates up to a maximum of 185,000s⁻¹ is used for high shear measurement. Both instruments give reproducible results and have been selected arbitrarily as standard instruments for this test method.

Approved by the Standard Specific Interest Group for this Test Method



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3. Apparatus

3.1 *Low-shear viscometer*¹, a rotating spindle apparatus with a full-scale spring torque, 0.7187 mN•m (7187 dyne-cm) and operating at 20 rpm with an appropriate spindle for mid-scale readings, as shown in Figure 1 and fully described in the appendix. This instrument can be operated at 10, 20, 50, and 100 rpm spindle speed. Viscosity values obtained at these four spindle speeds may be helpful in determining differences in rheological properties of various samples since it is not unusual for samples to have similar values at a given spindle speed but differ significantly at other speeds. In general, the 20-rpm value is the one normally referred to in pigment product specifications. A digital version of this instrument may be substituted for the version described here and in the appendix.

3.2 *High-shear viscometer*, a high-shear rotational bob instrument equipped with necessary accessories, as shown in Figures 2a and 2b and fully described in the Appendix.

3.3 *Heavy-duty high-speed mixer*, a variable speed mixer capable of developing a peripheral speed under load of about 1068 m/min. (3500 ft/min.) In a container having a 1:2.4 impeller-container diameter ratio and positioned so that the mixer blades are immersed to within 1-1.5 cm of the inside bottom of the mixing cup.

3.3.1 *Malted milk-type mixer*, high speed, with a 107-W (1/7 hp), 20,000-rpm motor, and equipped with a four-blade folding impeller about 32.5 mm (about 1.25 in.) diameter with accompanying round mixing cup, is satisfactory for most applications.

3.4 *Mixer*, low-speed, laboratory variable speed to 2000 rpm, with 50 mm (2 in.) slotted type propeller, to be used to incorporate any necessary dilution water and/or dispersant additions to the prepared pigment suspension during solids adjustment or during incremental dispersant additions as described under Section 7.

3.5 *Oven*, maintained at $105 \pm 3^\circ\text{C}$ ($221 \pm 5^\circ\text{F}$).

3.6 *Beaker*, 600 mL tall form (Berzelius).

3.7 *Miscellaneous*: balance, with a capacity of 2000 g; suitable to accurately weigh small increments of dispersant i.e., TSPP 74- μm (200-mesh) sieve; thermometer graduated from 0 to 100°C ; beakers, 600-mL.

3.8 *Alternative*: Microwave oven with an analytical balance can be used to determine the slurry solids. Infrared oven/analytical balance combinations can also be used.

4. Calibration

4.1 *Low-shear viscometer*.

4.1.1 Calibrate the instrument with the standard viscosity oils, available from instrument manufacturer, using the same spindle, volume, and temperature, and similar container employed for the sample. Draw a calibration curve for the instrument based on at least two standard oils having higher and lower viscosities than the sample being measured. If the measured viscosity of the standard oil with an established factor differs by less than 2% from that given from the standard oil, the viscosity of the specimen may be found by applying a proportional correction factor. If the difference is more than 2%, have the instrument reconditioned by the manufacturer before use.

4.2 *High-shear viscometer (Spring Set Model)*.

4.2.1 Calibrate the instrument with a manufacturer's certified test fluid. A rheogram of the fluid is determined with the A bob, 1.0 Nmm (100,000 dyne-cm/cm) spring set and a 0-1100 rpm bob speed range according to the manufacturer's operating instructions. The apparent viscosity of the test fluid should agree within $\pm 1\%$ of the certified value. If the difference is more than $\pm 1\%$, a new factory-calibrated 1.0-Nmm spring set should be installed.

4.3 *High-shear viscometer (torque sensor model)*.

4.3.1 A calibration check should be performed with a manufacturer's certified test fluid using the A bob, 1.0 Nmm (100,000 dyne-cm) spring setting at 1100 rpm speed setting according to the manufacturer's operating instructions. The apparent viscosity of the test fluid should agree within $\pm 1\%$ of the certified value. If the difference is more than $\pm 1\%$, the instrument should be recalibrated.

¹ Names of suppliers of testing equipment and materials for this method may be found on the Test Equipment Suppliers list, available as part of the CD or printed set of Standards, or on the TAPPI website general Standards page.

5. Reagents and materials

5.1 *Dispersing agent*, tetrasodium pyrophosphate, $\text{Na}_4\text{P}_2\text{O}_7$, or a freshly prepared 50% solution, by weight, of the sodium hexametaphosphate (NaPO_3)₆ in deionized or distilled water, or a standard sodium polyacrylate dispersant (molecular weight 3400-3600 amu, usually supplied as a 43% aqueous solution that is suitable for kaolin slurry dispersion).

5.2 *Standard viscosity oils and fluids* having a certified viscosity range.

NOTE 1: Do not keep these oils beyond the period specified by the supplier, since their viscosities tend to change with time.

6. Sampling and test specimen

6.1 Obtain approximately 600 g of clay in accordance with TAPPI T 657 "Sampling of Fillers and Pigments." After determining the moisture content in accordance with TAPPI T 671 "Free Moisture in Fillers and Pigments," weigh out the equivalent of 550 ± 0.1 g of moisture-free clay as the test specimen.

7. Procedure

7.1 Pour 235 g of water, minus the free water contained in the 550 g clay specimen itself, into a tared mixer cup. Add the equivalent of 0.2% of dispersant on the dry weight of the clay to the water in the mixing cup (for slurry samples, see 7.3). If the clay is in "predispersed" form, this dispersant addition step should be omitted. Place the cup on either of the high-speed mixers and start the motor at low speed, adding the clay in small increments and allowing it to mix thoroughly between additions. The clay specimen addition is normally accomplished within a 2-3 minute period. When all the clay has been added, agitate the mixture at high speed for 15 minutes, then remove the cup from the mixer, cover with a watch glass and cool in a water bath to $26 \pm 0.5^\circ\text{C}$.

7.2 Determine the percent solids of the cooled slurry by evaporating to dryness approximately 10 g of slurry in the drying oven and weighing the residue to constant weight. A drying oven/analytical balance combination instrument can be used as an alternative.

NOTE 2: If the percent solids of the clay-water suspension is less than the desired 70% solids, it will be necessary to repeat the test - taking care that the free moisture content of the clay used in preparing the slurry has been determined accurately. This initial slurry percent solids determination is of prime importance since the solids value obtained will be used to determine the dilution water required to adjust the slurry sample to the desired solids as discussed in 7.4.

NOTE 3: It is also important to measure accurately the dosage of dispersing agent added, because small differences from the optimum may cause large differences in the resulting viscosity.

7.3 In the case of clay slurry shipment, mix slurry samplings for 2 min. with the low-speed mixer. Following this, determine the percent solids of the slurry as prescribed, and conduct testing in accordance with the prescribed procedure below for both slurry and dry clay situations.

NOTE 4: In the case of these preslurried clays, omit the various steps concerned with making the clay into full dispersions, as the supplier has already performed this task; however, the procedure outlined for determining the optimum amount of dispersant required to achieve minimum viscosities should be followed.

7.4 Screen the remaining slurry through the 74- μm (200-mesh) sieve. Transfer a quantity of slurry equivalent to 500 g moisture-free dry clay into the tared 600-mL beaker and adjust the solids to exactly 70.0% by the addition of water. Adjust the temperature of the mixture to $25 \pm 0.5^\circ\text{C}$, then mix 2 min. with the low-speed mixer at a speed setting just sufficient to result in a gentle surface movement of the slurry towards and down into the vortex resulting from the stirring action.

7.5 Measure the low-shear viscosity on the viscometer at 20 rpm using an appropriate spindle by the following technique so as to produce a scale reading on the viscometer between 30 and 70% of full-scale deflection. Immerse the spindle in a slightly tilted state until the spindle disc is completely submerged, thus preventing entrapment of air beneath the disc which could affect the accuracy of values obtained. After the disc is submerged, straighten the spindle shaft to a vertical position. Connect the spindle to the viscometer and lower the spindle to the correct shaft immersion mark. Set the speed control knob, depress the viscometer's clutch, and start the motor. Release the clutch and allow the unit to rotate for 30 seconds. Depress the clutch, stop the motor, and take the