FIELD TESTING EQUIPMENT AND TESTING METHODS
PROJECT 0-5820, DELIVERABLE 0-5820-P1

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FIELD TESTING EQUIPMENT AND TESTING METHODS

Testing Equipment

The objective of this research project was to develop a dynamic shear rheometer (DSR) based field laboratory test and equipment to predict potential cement-admixture incompatibilities through the direct measurement of cement paste rheology. The primary use of DSR is to measure viscous and elastic behavior of asphalt binders. The DSR developed in this project was modified from its original condition in the following ways to measure the rheological properties of cement paste effectively:

- To prevent slippage, 240 grit size paper with adhesive back was installed on both upper and lower plates (25 mm diameter for both the plates). See Figure 1.
- A fluid jacket heating/cooling system was installed (Figure 1) instead of a water-based temperature controller because the cement paste should not come in contact with water. Most DSRs used at state departments of transportation (DOTs) have water circulation systems for temperature control where a specimen comes in contact with water directly. Since fresh cement paste is a water-sensitive material unlike asphalt binder, the existing temperature control system needed to be changed. With the fluid jacket system, it was possible to keep the temperature of the cement paste sample constant during the entire time span of the rheological test.
- A sealing cap was applied to the DSR to prevent the cement paste specimen from water loss due to evaporation during the test procedure. The research team came up with three different methods to prevent evaporation: (1) applying a thin layer of mineral oil (immiscible with the sample) especially at the periphery of the parallel plates, (2) placing a humidifier in close proximity to maintain high relative humidity (RH) in the surrounding areas, and (3) encapsulating the sample chamber by a plastic sealing cap. Preliminary tests results showed that the third option was the most effective method.
- Gap size was adjusted to a 1 mm gap between lower and upper plates as an optimum gap determined by performing experiments with five different plate gaps (i.e., 0.2, 0.5, 1.0, 1.2, and 1.5 mm). Test results indicated that the DSR with 1 mm plate gap best distinguished three studied mixtures based on their rheological parameters with the best reproducibility and sensitivity.

- Test duration was changed to a longer test duration (up to 2 hours with 10, 30, 60, 90 and 120 minutes testing intervals), necessary for a total evaporation control situation in order to derive an effective rate of change of rheological parameters.
- The range of applied shear rate was fixed from 0 to 200/s, which yielded the most reproducible rheological parameters. The cement paste sample placed between two parallel plates tended to suffer a segregation problem (accumulation of more liquid part at the periphery of the plate) as a shear rate larger than 200/s was applied.

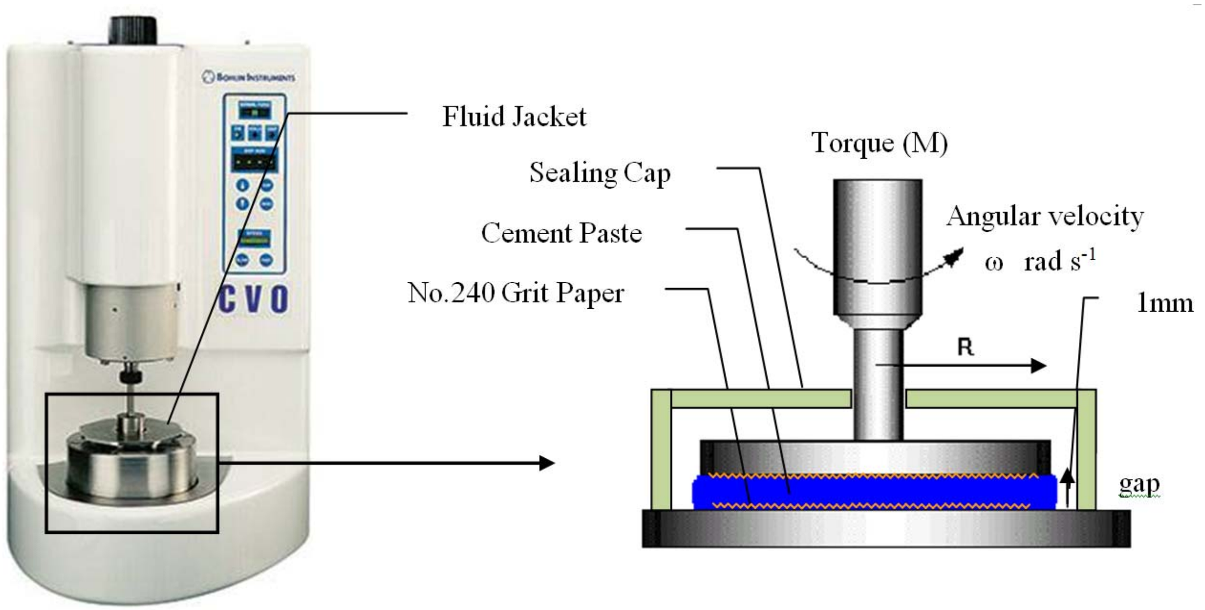

Figure 1. The Modified Dynamic Shear Rheometer for Cement Paste Rheology Measurement.

Test Methods

A test procedure was developed using the modified DSR as the rheology measuring device along with a high-shear mixing procedure under differing temperature conditions as described below.

Step 1. Storage of Materials

All the materials that were used in the research [i.e., cement, deionized water, supplementary cementitious materials (SCM), and chemical admixtures] were kept under controlled ambient temperature at least for one day before mixing started. A refrigerator stored materials and mix at a low temperature (i.e., 10°C/50°F) to represent winter temperature conditions, whereas an oven kept materials and mix at a high temperature (i.e., 35°C/95°F) to represent summer temperature conditions (as shown in Figure 2). Storing materials and mixing inside a lab room with 24°C/75°F temperature represented mixing at intermediate ambient temperature conditions. The cement and SCMs (fly ashes, slag, etc.) with predetermined proportions according to the experimental design were dry blended well before being stored.

(a) (b)

Figure 2. (a) The Use of a Refrigerator to Mix at Low Temperature (10°C) and (b) The Use of an Oven to Mix at High Temperature (35°C).

Step 2. Mixing Procedure

The mixing procedure to prepare the cement paste sample was developed by Texas Transportation Institute with steps based on the procedure developed by Portland Cement Association (PCA) and later on National Institute of Standards and Technology (NIST). A high-shear mixer, i.e., a kitchen blender (Figure 3), was used to develop the mixing procedure. The maximum mixing speed used during mixing procedure was 6000 rpm instead of 10000 rpm in order to reduce high heat generation due to friction. The steps involved in the mixing procedure (Figure 4) are described below:

- Keep the mixing bowl along with all the ingredients inside the refrigerator/oven/room for pre-conditioning under the selected studied target temperatures.
- Keep the predetermined quantity of cement and SCM blend in the mixing bowl of the mixer.
- Measure water into the mixing bowl containing cement and SCM blend followed by switching on the mixer with 3000 rpm speed for 30 seconds.
- Add a chemical admixture to the cement and water mixture in the container slowly within 50 seconds and mix again with 3000 rpm setting for another 10 seconds.
- Increase mixing speed to 6000 rpm and continue mixing for another 30 seconds.
- Stop mixing for a 2-minute pause and scrape the sides of the mixing bowl with a rubber paddle.
- Mix again in the same high-shear blender at 6000 rpm for another 30 seconds.

The ingredients kept in the refrigerator or oven were mixed immediately after bringing them outside according to the above mixing procedure and then the mixing bowl with cement paste was put back inside the refrigerator or oven immediately after mixing in order to make the heat gain or loss minimal.

Mixing Speed	1.	2.	3.	4.	5.
Level	Stir	Chop	Mix	Puree	Liquify
RPM	3000	6000	8000	10000	13000

(a) (b)

Figure 3. (a) The High-Shear Mixer, KSB560OB Kitchen Aid Company and (b) RPM Corresponding to Different Mixing Speed Levels.

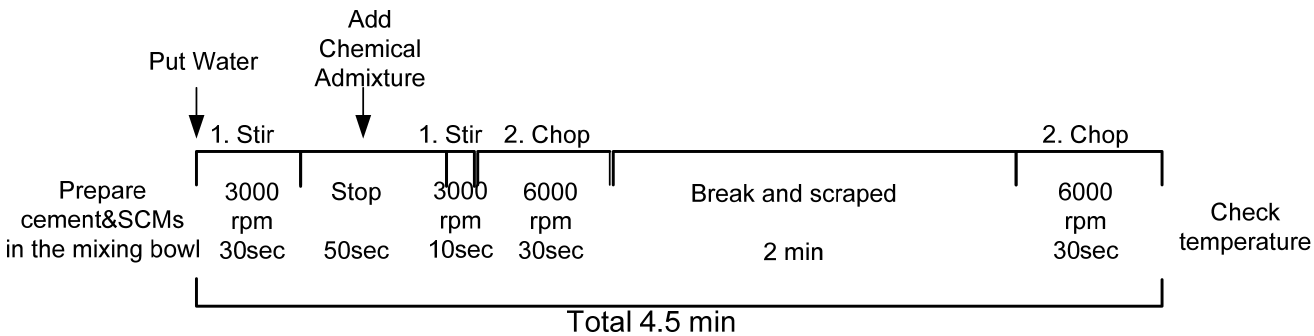

Figure 4. A Schematic Representation of the Different Steps Involved in the Mixing Procedure.

Step 3. Rheometer Test Procedure

The rheometer test procedure is given below:

- Take cement paste specimen from the mixing bowl using a 3 ml syringe immediately after mixing procedure. Return mixing bowl with remaining cement paste to the refrigerator or oven immediately after taking the specimen. Place 1.5 ml cement paste between the two parallel plates (25 mm plate diameter). A 1 mm plate gap was found to be optimum through different trials.
- Load the predetermined quantity of cement paste onto the lower plate of the rheometer from the syringe.
- Sandwich the specimen between the two parallel plates with 1 mm plate gap and shear with shear rate from 0 to 200/s representing the up curve followed by 200 to 0/s representing the down curve. Record the shear stress as a function of the shear rate. Each complete test run with one cycle consisting of one up curve and one down curve takes approximately 3 minutes.
- Start the first run approximately 10 minutes after adding water to the cement. Conduct another four runs with different time intervals of 30, 60, 90 and 120 minutes following the same procedure described above.

Typical data showing shear rate versus shear stress are presented in Figure 5(a). The plastic viscosity and yield stress determined using Bingham model are shown in Figure 5(b). The plastic viscosity is calculated from the slope of the linear region of the down curve, whereas yield stress is calculated from the interception as shown in Figure 5(b).

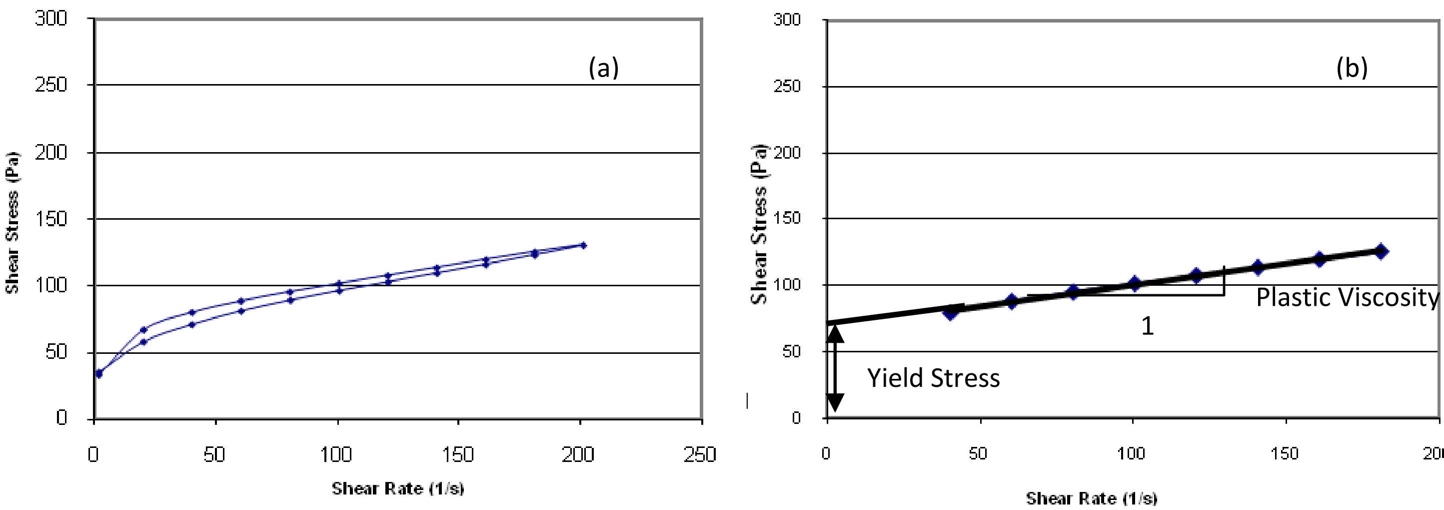

Figure 5. (a) A Typical Plot Shear Stress vs. Shear Rate and (b) Calculation of Rheological Parameters.

The rheological parameters, i.e., plastic viscosity and yield stress, corresponding to five different time intervals were calculated as described above. Figure 6(a) demonstrates the calculation of the plastic viscosity corresponding to five time intervals. Figure 6(b) shows change of plastic viscosity as a function of time. The slope of the linear region in Figure 6(b) represents the rate of change of plastic viscosity (RPV) within 2 hours time period. The rate of change of yield stress (RYS) within 2 hours time period is calculated by applying the same procedure.

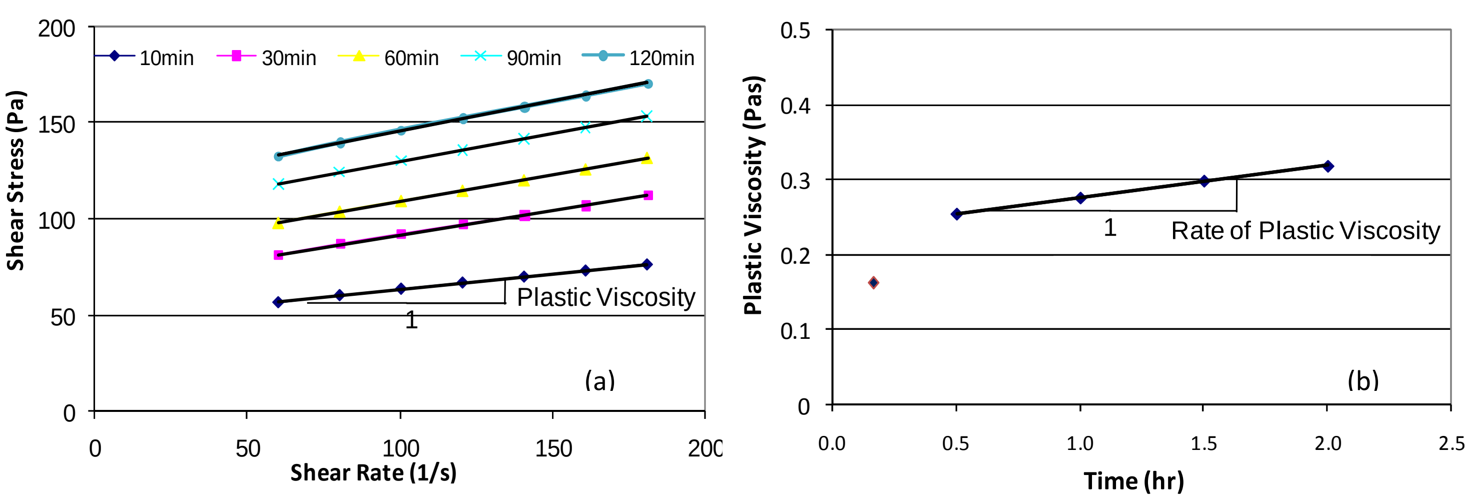

Figure 6. (a) Plastic Viscosities with 5 Time Intervals and (b) Calculation of Rate of Plastic Viscosity.

Reproducibility of the Rheological Parameters

Reproducibility of the rheological parameters (both absolute values and rates) based on the two mixes under three different temperatures are presented in Table 1(a) and 1(b) as a part of Task 8 (conducting field validation).

Table 1. (a) Reproducibility of Plastic Viscosity (PV) and Yield Stress (YS) of the Two Mixes at Three Different Temperature Conditions.

			Plastic Viscosity	**PV Average**	**COV %**	**Yield Stress (YS)**	**YS Average**	**COV %**
37 C4+F35+X15DD	10°C	1	0.1598	0.1553	2.54	42.1	41.06	3.21
		2	0.1524			41.51		
		3	0.1537			39.58		
	24°C	1	0.1638	0.1616	1.36	46.92	39.70	16.93
		2	0.1594			33.62		
		3	0.1617			38.56		
	35°C	1	0.2181	0.2099	3.86	69.89	65.53	5.80
		2	0.2019			63.84		
		3	0.2096			62.87		
38 C4+F35+D17DD	10°C	1	0.124	0.1221	1.35	14.57	15.54	6.51
		2	0.1215			16.59		
		3	0.1209			15.47		
	24°C	1	0.1437	0.1381	3.55	23	20.03	15.75
		2	0.1348			16.72		
		3	0.1357			20.37		
	35°C	1	0.1536	0.1538	4.10	47.587	51.25	8.84
		2	0.1602			56.32		
		3	0.1476			49.85		

Table 1. (b) Reproducibility of Rate of Plastic Viscosity (RPV) and Rate of Yield Stress (RYS) of the Two Mixes at 3 Different Temperature Conditions.

Experimental Design #			RPV	RPV Average	COV%	RYS	RYS Average	COV%
37 C4+F35+X15DD	10°C	1	0.0356	0.0370	3.48	21.59	24.17	9.93
		2	0.0381			26.34		
		3	0.0374			24.57		
	24°C	1	0.0436	0.0437	4.81	24.75	26.80	6.72
		2	0.0459			28.12		
		3	0.0417			27.54		
	35°C	1	0.0517	0.0548	7.38	29.56	32.78	12.09
		2	0.0534			31.58		
		3	0.0594			37.21		
38 C4+F35+D17DD	10°C	1	0.0016	0.0015	6.67	6.969	6.49	17.02
		2	0.0014			5.23		
		3	0.0015			7.28		
	24°C	1	0.0115	0.0120	4.21	8.14	9.07	9.58
		2	0.0119			9.21		
		3	0.0125			9.86		
	35°C	1	0.0204	0.0227	9.92	14.72	16.20	11.26
		2	0.0249			15.65		
		3	0.0228			18.24		

Table 1(a) and 1(b) indicate that the coefficient of variation (COV) % of both absolute values of PV and RPV is under 10 percent. The COV% of the YS and RYS is also under 10 percent for the 60 percent of the cases. The COV% of the YS and RYS for the remaining 40 percent cases is under 17 percent.

Based on the available test results, it is observed that criteria based on rate of change of rheological parameters, especially plastic viscosity as in Figure 6(b), calculated using data of five intervals (e.g., 10, 30, 60, 90, and 120 minutes) with 2 hours testing period are the most appropriate to be considered as acceptance criteria (Task 7- Establishing acceptance criteria). The range of value of the rate change (e.g., as an acceptance criterion) is a function of temperature and mix design parameters (e.g., w/cm, type and content of SCMs, etc.) and cannot be considered as a common criterion for all situations. Possible future work will be discussed in the full report for this project (Report 0-5820-1).