New Tools to Measure Emulsified Asphalt Properties

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Abstract

New test methods for viscosity and residue measurements of emulsified asphalts are evaluated. In this study, three instruments are used to measure the rotational viscosity of emulsified asphalts: a Brookfield Rotational Viscometer (RV), a Cannon Marine Fuel Viscometer and a Bohlin CVO Rheometer (CVO). Preliminary results show that the viscosity obtained from the Cannon Marine Fuel Viscometer had the highest correlation ($R^2 > 0.90$) with the viscosity measured on the Saybolt Furol viscometer, while measurements with RV and CVO gave more detailed information on the rheological behaviors of emulsified asphalts.

A moisture analyzer was used to automate and decrease the testing process to obtain results for an emulsified asphalt residue. The test was completed in less than 20 minutes. Results from the moisture analyzer tests correlated well with results obtained from evaporation ($R^2 > 0.90$) as described in ASTM D 244.

Introduction

Viscosity and residue of emulsified asphalts have been used as key specification parameters for manufacturers and state agencies. For monitoring emulsified asphalt production, test results need to be generated in a timely fashion. However, current standard methods for testing viscosity (Saybolt Furol) and residue by evaporation of emulsified asphalts continue to be time consuming [1]. Until reliable on-line instrumentation becomes available faster automated and accurate test methods needs to be evaluated.

Rotational viscometry has been used in characterizing the rheological properties of emulsified asphalts [2, 3]. Previous researches have shown that the steady-state viscosity of emulsified asphalts determined by rotational viscometry correlates well with the Saybolt Furol viscosity [4, 5]. Brookfield Rotational Viscometer (RV) was used to determine the equilibrium viscosity. While emulsified asphalts show thixotropic behaviors, equilibrium viscosity can be obtained after 20 minutes [6]. To further evaluate the use of rotational viscometry as a quality assurance tool, two more instruments for measuring rotational viscosity were selected: a Cannon Marine Fuel Viscometer (MFV) and a Bohlin Dynamic Shear Rheometer (DSR). Results obtained from these equipments were correlated with measurements from a Saybolt Furol viscometer.

Asphalt residue affects the physical properties of the emulsified asphalt such as density, viscosity, impacts on its workability in the field. Manufacturers without an automated process control system would like to determine accurately and quickly an emulsified asphalt residue during production. Emulsion mill operators need this information to adjust the process and to maintain the emulsion quality. The standard determination of emulsified asphalt residue by evaporation takes several hours [1], and is not a cost-effective quality assurance test. Moisture analyzers are widely used in the chemical, pharmaceutical, environmental, food and beverage industries, since they can be automated, reduce testing process cycle time and are precise. In this study, a moisture analyzer is evaluated as a convenient and faster method to determine the emulsified asphalt residue.

Experimental Design

Materials

The determination of emulsified asphalt residue was performed on sixty-five different emulsified asphalts. Several cationic and anionic emulsions types were used for viscosity measurements: CRS-2P (polymer modified rapid set), CRS-2R (polymer modified rapid set), CRS-2 (rapid set unmodified), CRS-2h, (rapid set unmodified hard base), CMS-2s (medium set with solvent), CSS-1 (slow set), CHR-3P (high residue polymer modified rapid set), HFRS-1P (high float polymer modified rapid set), and HFMS-1 (high float medium set), and RS-1 (anionic rapid set).

Equipment

For viscosity experiments, a Brookfield RVDV-III+ programmable rheometer, a Cannon MFV-1000 Marine Fuel Viscometer and a Bohlin CVO dynamic shear rheometer were used for the rotational viscometry test. Saybolt Furol viscosity was determined using a Koehler K214.

Residue determination was performed with a programmable Mettler Toledo HR73 Halogen moisture analyzer.

Test Protocol

<u>Viscosity</u>

The preparation of emulsified asphalts samples for viscosity determination was performed according to ASTM D-244 [1]. The tests on the Brookfield rheometer were conducted following method 50-50-21 [4, 5]. Emulsified asphalts were tested using SC4 spindle No. 21 at 50°C and at 50 RPM, which equals to a shear rate of 46.5 sec^{-1}. The rheometer was programmed for a 30-minute continuous run. The reading at 30 minutes was used as the rotational viscosity result for the emulsion. For the Marine Fuel Viscometer, the test procedure was conducted based on the manufacturer's instruction [7]. The sample was heated to 50°C and sheared with a paddle/spindle (5 cm x 4.8 cm) and after 20 seconds, the result is read from the viscometer. For the DSR, a Bohlin Peltier system equipped with a 25 mm diameter cup-and-bob (Couette) geometry was used. A shear rate of 46.5 sec^{-1} was targeted. A reading is obtained after 30 minutes of continuous shearing at 50°C. Both Brookfield and DSR were programmed to record readings at one-minute time intervals.

Residue

Emulsified asphalt samples were prepared according to the procedure described in ASTM D-244 [1]. In all cases, 0.5±0.2g of emulsion was used for the tests. The moisture analyzer was programmed to heat to 163°C. The analyzer stopped the test automatically when the change in weight was within 1mg for more than 140 seconds. The residue is continuously monitored and can be printed out at the end of the run.

Results and Discussions

Viscosity

Thixotropic behavior of emulsified asphalts

Figure 1 shows a plot of the viscosity versus time for a CRS-2P emulsion measured on the Brookfield rheometer and CVO. This time-dependent behavior was observed for all emulsified asphalts studied. Previous work has suggested that this behavior may be related to the breaking of the emulsion microstructure [6]. Within the testing time of 30 minutes, the viscosity decreases to a relative steady level. This equilibrium viscosity obtained at 30 minutes for all emulsions were compared with the Saybolt-Furol viscosity.

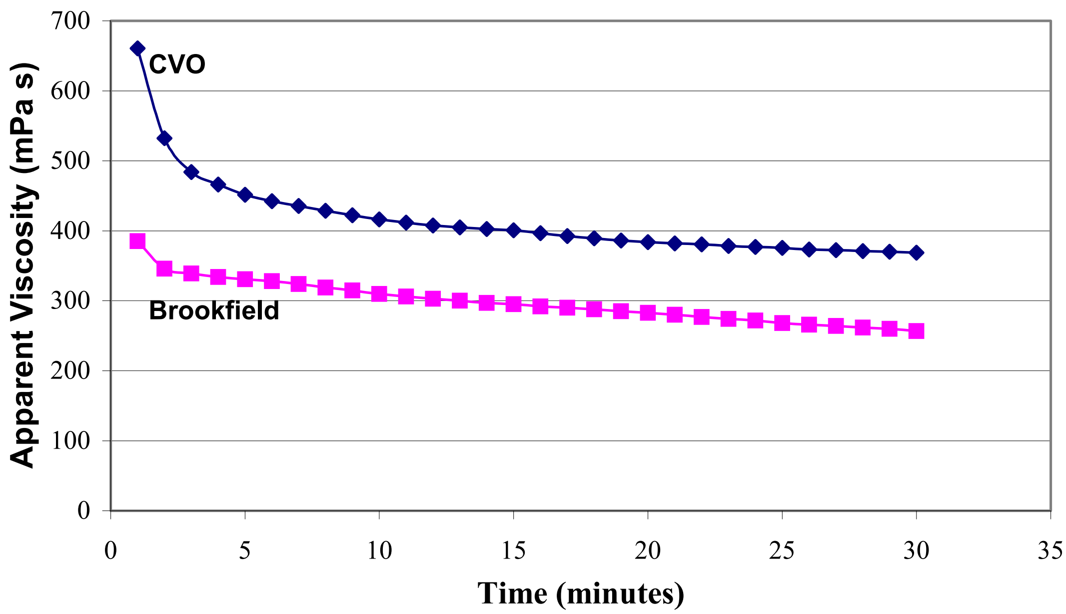

Figure 1: Thixotropic behavior of emulsified asphalt (CRS-2P)

Geometry Dependence

Figure 1 also indicates that there are differences between viscosity data obtained from different viscometers even though with the same geometry (coaxial). Table 1 shows examples of viscosity data obtained from different instruments. For the same emulsion, different viscosity values were obtained from the three instruments. This may be caused from the different geometry for the three instruments. The difference in geometry may have different effects on the breakdown of the emulsion microstructure. The final emulsion equilibrium microstructures will be different under different geometries, which result in different equilibrium viscosity values. Further investigation is needed to understand these effects.

Table 1: Viscosity Data from Different Methods

Emulsions	Viscosity (mPas)			
	Saybolt(SFS	Brookfield	MFV	CVO
CRS-2P	186.8	302.8	504.9	246.7
CRS-2R	193.4	253.3	582.9	350.7
CRS-2	214.2	336.6	701.1	344.5
CMS-2S	298.1	604.6	990.3	513.4

Correlation between Saybolt and Rotational Viscometry

Figures 2 through 4 show the correlation between different methods and Saybolt viscosity. For both Brookfield and CVO, R^2 is close to 0.8 (80%), while the correlation between MFV and Saybolt has an $R^2 > 90\%$. The high R^2 values indicate that all three methods can be used for the determination of emulsified asphalt viscosity. Since MFV shows the higher correlation in these three methods, MFV is a better tool for the determination of emulsified asphalt viscosity and a reliable quality assurance test method.

The shorter shearing time for MFV, may introduce the least disturbance to the microstructure of the emulsion. The microstructure of the emulsion in the MFV may remain more similar to the emulsion microstructure in the Saybolt tubes.

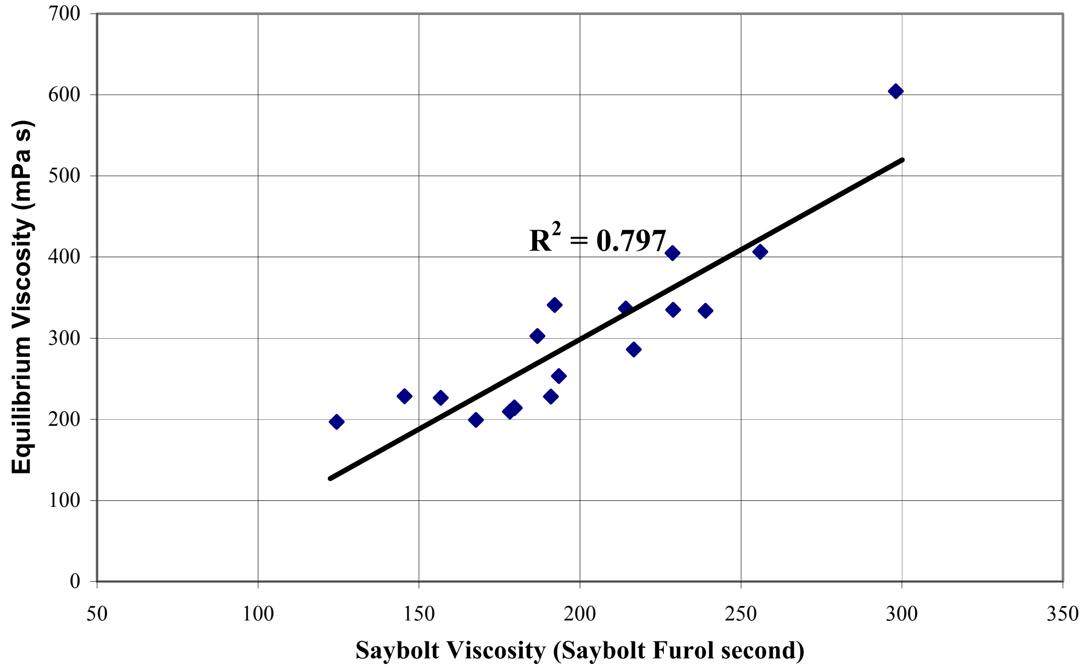

Figure 2: Brookfield vs. Saybolt

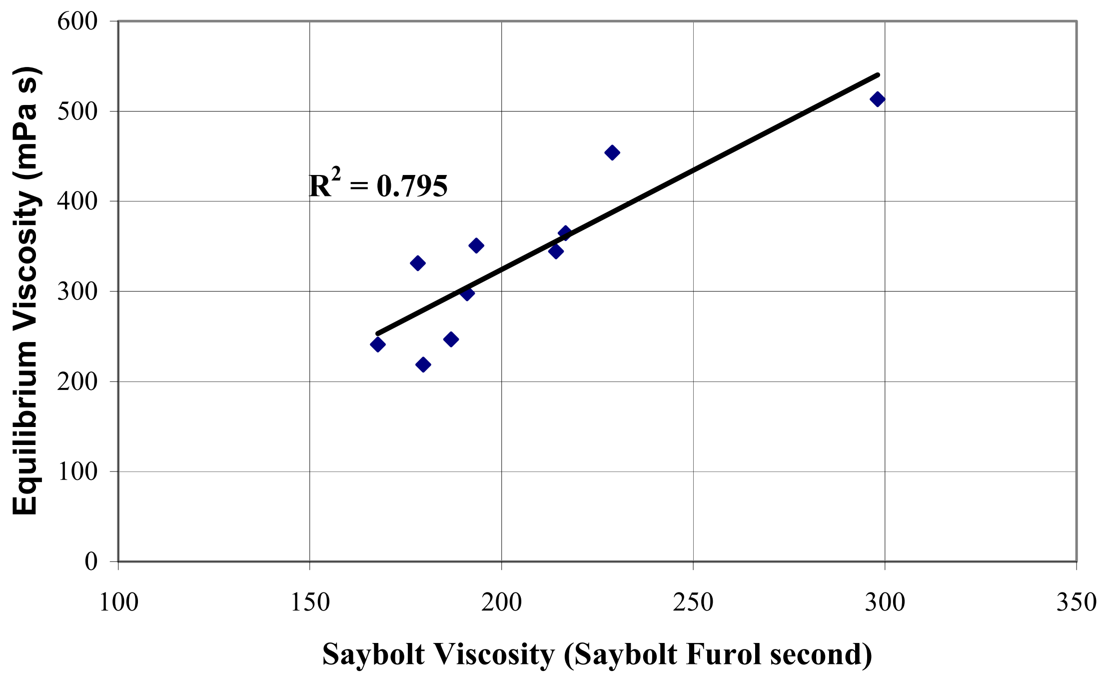

Figure 3: Bohlin CVO vs. Saybolt

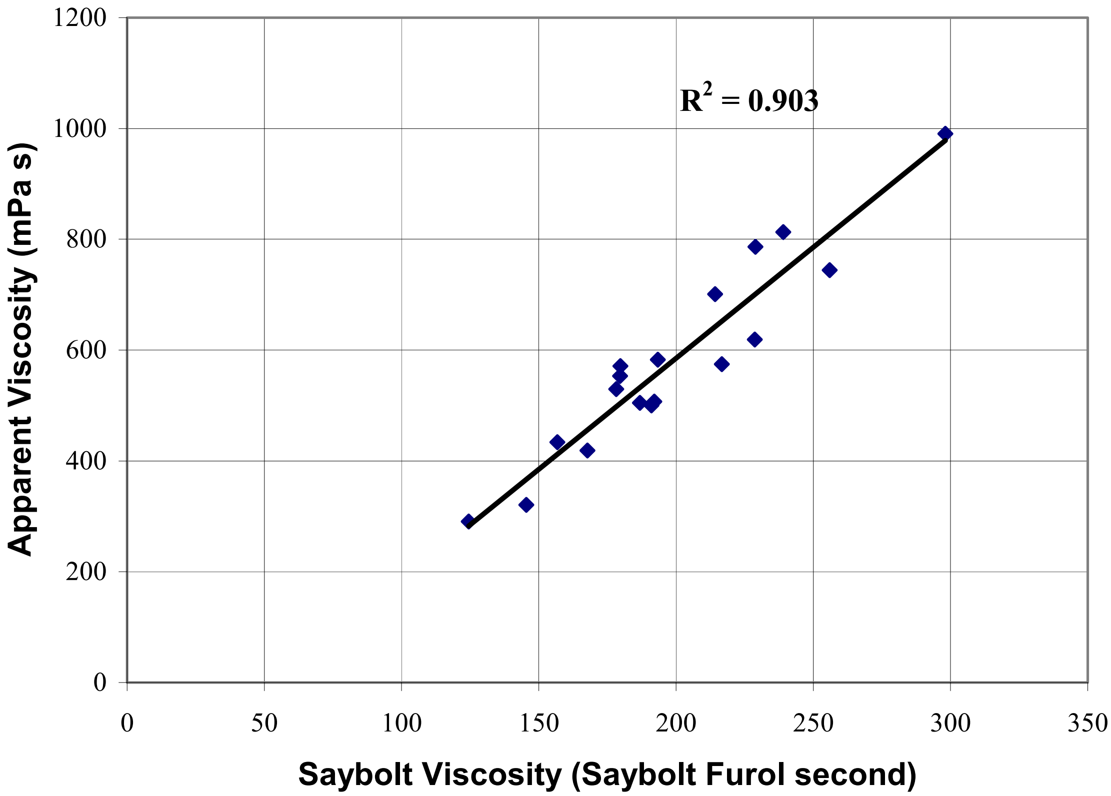

Figure 4: MFV vs. Saybolt

Residue

Figure 5 shows the correlation between emulsion residues obtained by evaporation with that determined using the moisture analyzer. The trend line intercept was set to (0, 0). From the figure, the slope of the line is 0.9958 with an R^2 close to 0.96. This shows the moisture analyzer and ASTM D-244 methods have a one to one linear relationship. The residue determined using the moisture analyzer could be considered as the same residue determined by evaporation.

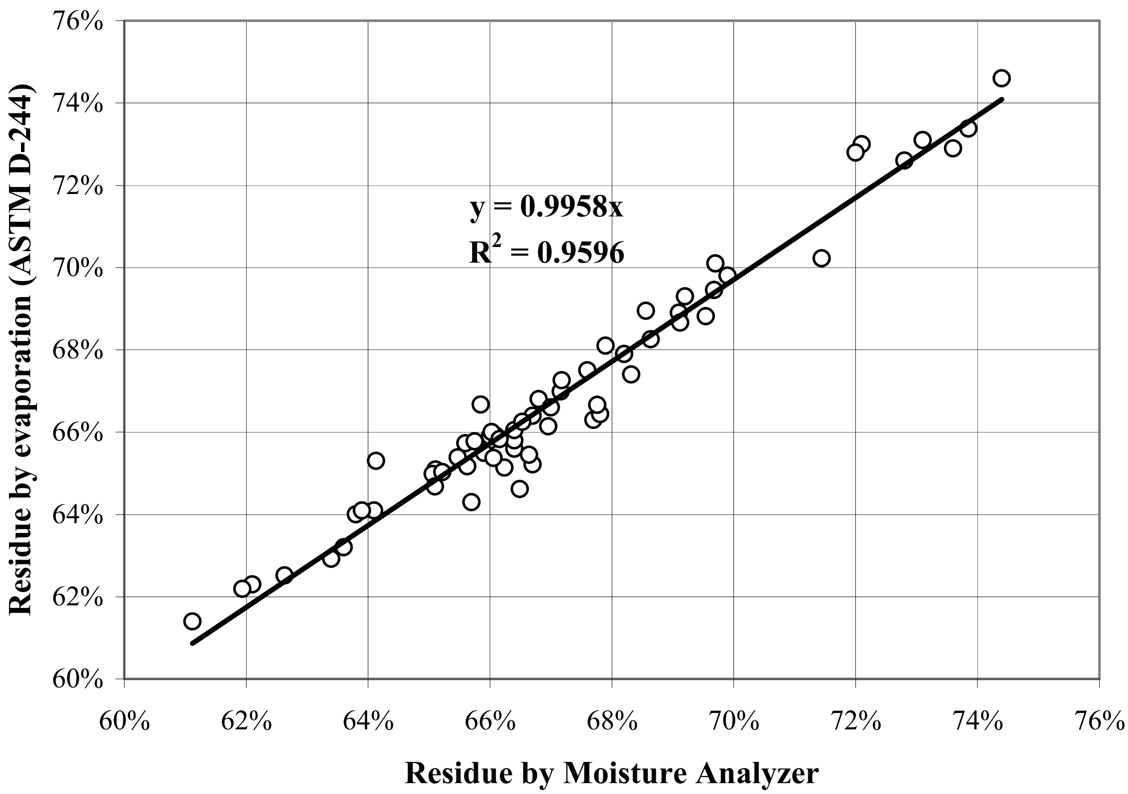

Figure 5: Residue by Evaporation vs. Moisture Analyzer

Unlike the evaporation method, the moisture analyzer residue determination had a shorter test cycle time and used less material. The average test time was 15 minutes compared to 3 hours for the evaporation method. As a tool for spot-checking residue, the moisture analyzer is a more practical tool than the evaporation procedure described in ASTM D-244. However, the recovered residue is not enough to run other tests such as a Penetration test. Due to the sensitivity of the moisture analyzer balance, it could not handle larger emulsion samples (> 30 g) efficiently. It also takes a longer time to complete the test for a larger sample size. Usually it takes more than 1.5 hrs. For now this limits the usage of the moisture analyzer method to recover residue for further testing. Emulsion sample size can be increased, sufficient to obtain a recovered residue to measure on a dynamic shear Rheometer (DSR). Preliminary results for a CRS-2P indicate a reasonable comparison ($G^*/\sin \delta$ @ 64°C) between residue recovery by the moisture analyzer (1.228 kPa) and the standard distillation recovery method (1.018 kPa). The moisture analyzer could serve as a well controlled and rapid recovery method for emulsified asphalts for further testing on a DSR.

Conclusions

1. Rotational viscometry can be used as a quality assurance tool for emulsified asphalts. Brookfield viscometer, Cannon Marine Fuel Viscometer and Bohlin CVO DSR can all be used for this task. MFV shows the best correlation with current method.
2. Viscosity determined using rotational viscometry shows time dependence. The value approaches that of a steady-state viscosity of the emulsion as time increases. It is likely that this steady-state viscosity is closer in value to that exiting the nozzle of the spray bar or to that of emulsions that have been pumped.
3. Rotational viscosity determined by a CVO and a Brookfield shows geometry dependence. This may be related to the breakdown of the emulsion microstructure.
4. Residue determined by a moisture analyzer has a one to one linear relationship with the residue determined using the procedure described in ASTM D-244. The residue rheological properties can further be determined on a DSR.

Acknowledgements

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