

MAB-DSR Procedure: Assessment of the Performance of a Moisture Analyzer Balance (MAB) to Obtain the Residue of an Emulsified Asphalt using a Dynamic Shear Rheometer (DSR) Silicone Mold and Determining its Rheological Properties.

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Abstract

Previous work on residue recovery of emulsified asphalts has shown that the ASTM D7404 standard can be used to determine the moisture content with good accuracy and precision using a moisture analyzer balance (MAB). Later work used the residue obtained from a MAB in a two-step process and measured the rheological property, G^*_{MAB} , on a dynamic shear rheometer (DSR). The G^*_{MAB} was compared to the G^* of the residue obtained from the distillation standard, ASTM D 6997, and the evaporation method ASTM D6934. The novelty of the present MAB-DSR procedure is that emulsified asphalt is placed directly in a redesigned DSR silicone mold which is then placed in the MAB to recover the emulsified asphalt residue. The cured sample is then removed from the mold and placed directly on the plate of the DSR to determine the shear Complex Modulus, $GMAB^*$. The $GMAB^*$ result is compared with that obtained from the ASTM 6997 distillation method denoted as $GDIS^*$.

Recovery is performed in the silicone molds at different temperatures and their respective $GMAB^*$ is compared to evaluate levels of thermal oxidation. In the previous study, G^* was used to rank the level of thermal oxidation for the different recovery procedures. This work is extended to include G^*_{MAB} obtained for different temperatures for the MAB test method. The MAB-DSR procedure is capable of performing recovery for emulsion samples at shorter times and with sufficient automation to reduce analysis costs while improving precision.

Previous work ranked G^* for the three methods as follows: moisture analyzer (D 7404) > Evaporation (D-6934) > Distillation (D 6997). However, the samples recovered from the MAB were reheated and placed on the DSR and therefore were subjected to additional oxidation. This new one-step process is developed to minimize oxidation and to evaluate the MAB-DSR procedure. Comparison of the G^* with the other consensus recovery procedures will be evaluated. Several different families of emulsified asphalt are compared and discussed.

1. Introduction

Emulsified asphalt residue is an important control variable in the production of emulsified asphalt and a key specification requirement for all emulsified asphalt applications. It is an indicator of asphalt content and intimately related to the viscosity of the emulsified asphalt. The residue needs to be measured quickly during the process of making the emulsified asphalt where expensive mass flow meters are not used. Measuring the residue at the production line continues to be time consuming and dependent on operator. Quality control procedures are based on evaporation of the water from the emulsified asphalt. These are well documented and described elsewhere [1, 2]. In the United States three of these procedures are widely used and are outlined in ASTM and AASHTO standards [3,4,6]. A recent Transportation Research Board publication reviewed recovery procedures for both USA and the European Union [2] and a new residue recovery procedure using a moisture analyzer balance (MAB) has been published by ASTM [5]. Recently, a new recovery procedure based on a low temperature evaporative protocol has been proposed at ASTM [7]. This procedure takes 48 hours to obtain a residue. A variation of this procedure developed by Texas Department of Transportation reduces the recovery time by about 50%. A European evaporative method similar to ASTM is also used [8].

Further work on the MAB procedure was compared with two other ASTM procedures including rheological properties of the recovered emulsified residue [9]. One of the issues with the consensus procedures is that polymer modified emulsions have not been addressed adequately. It is interesting to note that in the original Superpave standard performance specifications the use of polymers in hot asphalt was not addressed in a very consistent rheological manner and it is only now being addressed [10]. The MAB procedure is a very flexible test method since the recovery temperatures can be programmed to use lower temperatures for polymer emulsified asphalt in order to maintain polymer integrity. These recovery temperatures should not in general exceed 80°C, a typical temperature where chip seal emulsified asphalt is sprayed.

The important issue here is that laboratory recovery procedure for an emulsified asphalt, unmodified or modified, should not exceed the application temperature in the field. This is not the case for the distillation ($T > 200^\circ\text{C}$) and the evaporation procedures ($T = 163^\circ\text{C}$) that are widely used where recovery procedures exceed application and end-use temperatures. The goals of this research are two:

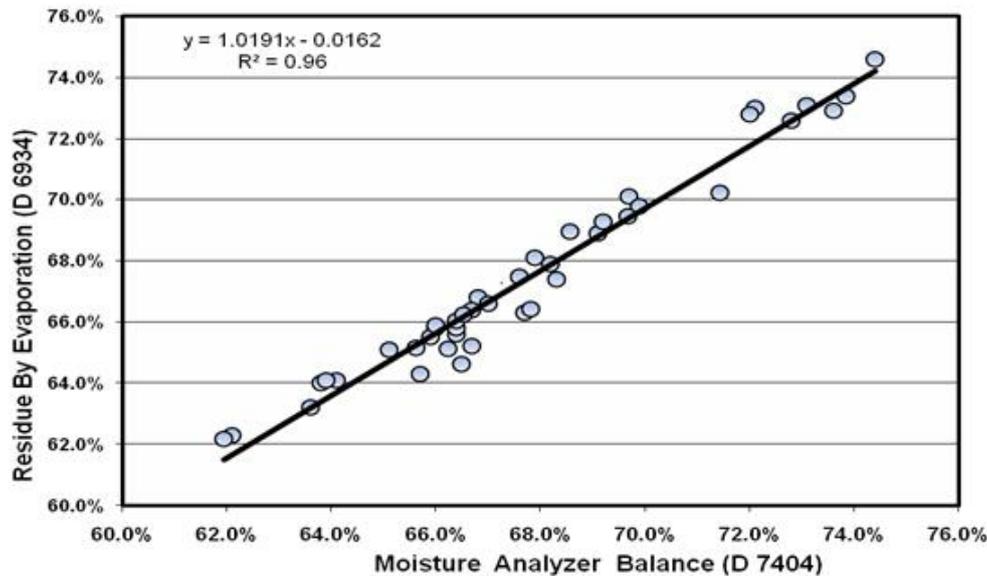
- 1) develop a one-step MAB-DSR procedure and compare the residue with that obtained from the distillation procedure and ,
- 2) perform preliminary rheological characterization (G^*) of the recovered residues from both procedures.

The rheological characterization is in place of the traditional tests for penetration, ring and ball, ductility, and absolute viscosity in some instances. The emulsion industry as a whole is searching for ways to apply rheological tests that have been successful in Superpave binder characterization, to the recovered binder from emulsified asphalts.

It is assumed that the recovered binder continues to be a viscoelastic material and therefore a similar rheological characterization of the binders may be applied. The difference is that the final rheological testing for these 'surface' treatment binders will be determined by application temperatures in the field. These temperatures are invariably below 100°C. The key to the rheological properties is that the binder is subjected to temperatures that are similar to application temperatures of asphalt emulsions in the field. This study is not to define end-use performance, but to show characterization of the residue obtained from the two procedures that were studied.

Figure 1 shows a good correlation between emulsified asphalt residues recovered by evaporation and the MAB procedure. This study focuses on a method development procedure where residue recovery is done directly in the DSR silicone mold on the MAB, followed by placement of the cured sample on the DSR and obtaining G^* values. This G^* is compared that obtained from residue recovery by distillation.

Figure 1. Comparison Residue by Evaporation (D 6934) vs. Moisture Analyzer Balance (D 7404)



2. Experimental Design

2. 1 Materials

The determination of emulsified asphalt residue was performed on six different emulsified asphalts using two different recovery methods as described. The six emulsions represent a cross section of emulsions used for various pavement treatments: SS-1, a slow set anionic use for tack coat or fog seal; MS-2, a medium set anionic; CRS-2, a rapid set cationic emulsion; and three different CRS-2P (denoted as CRS-2P-A, CRS-2P-B, CRS-2P-C) polymer modified cationic rapid set use for chip seal. The base asphalt used for all emulsions was a PG 58-28.

2. 2 Equipment

A dynamic shear rheometer equipped with a parallel plate was used to perform all rheological tests. The distillation equipment was a standard still as described in ASTM D 6997. The moisture analyzer balance was a OHAUS MB2000 equipped with a halogen lamp heater element. The typical weight of emulsified asphalt placed directly into the silicone molds with 18 to 25 mm diameter was between 1.4 to 1.9 g. The recovered residue was typical between 0.9 to 1.2 g of residue, which was placed directly on the DSR plates for measurement. The procedure is similar to that described in ASTM D 7404 except other temperatures were programmed on the MAB for the emulsions in this study.

Test Protocol and Residue Recovery

Emulsified asphalt samples were prepared according to the procedures described above. Variation of ASTM D-7404 [5] was as follows: the MAB was programmed to heat at 1260C for five minutes, then ramped to 1420C for ten minutes, then to the final temperature of 1850C for sixteen minutes. The emulsified asphalt sample was placed in a silicone mold with diameter of 22 to 25 mm. In some cases a silicone mold with a diameter of 18 mm was used. Typical sample weights were between 1.4 to 1.9 g with final residue between 0.90 to 1.2 g which was placed directly from the silicone mold on the DSR plates to determine G*. Total analysis time was close to 35 minutes. Typical temperature profiles used for recovery by MAB are shown in Figures 2 and 3. Typical heating rates for the first ramp temperature were close to 3.20C/min and the second ramp was close to 7.20C/min (Figure 2). The programmed temperature shown in Figure 3 were 4.20C/min for the first ramp and 3.20C for the second ramp. In Appendix I is shown the sequence of steps for recovering the residue of an emulsified asphalt using a silicone mold. If a MAB is not available an oven may be used, but this increases sample handling and requires additional equipment.

Figure 2. Temperature profile used for MAB residue recovery

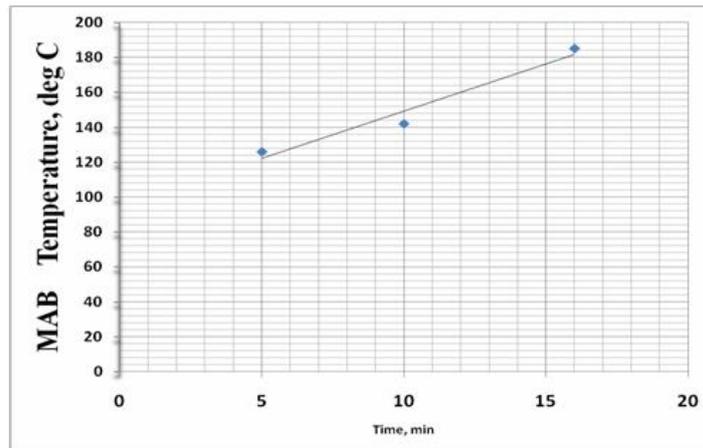
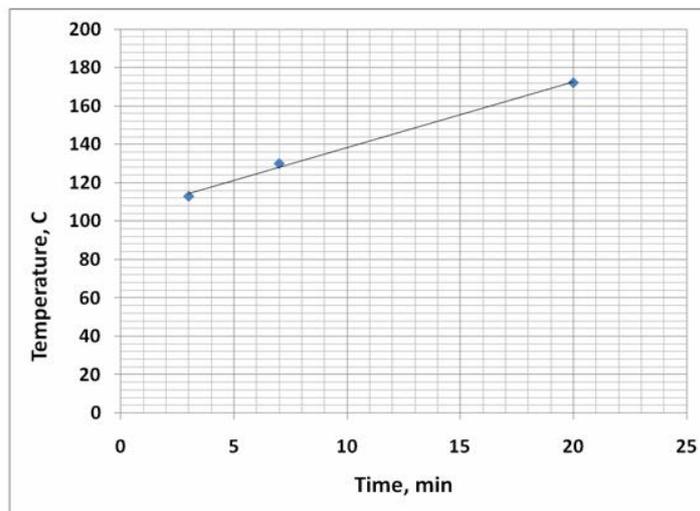


Figure 3. Temperature profile used for MAB residue recovery



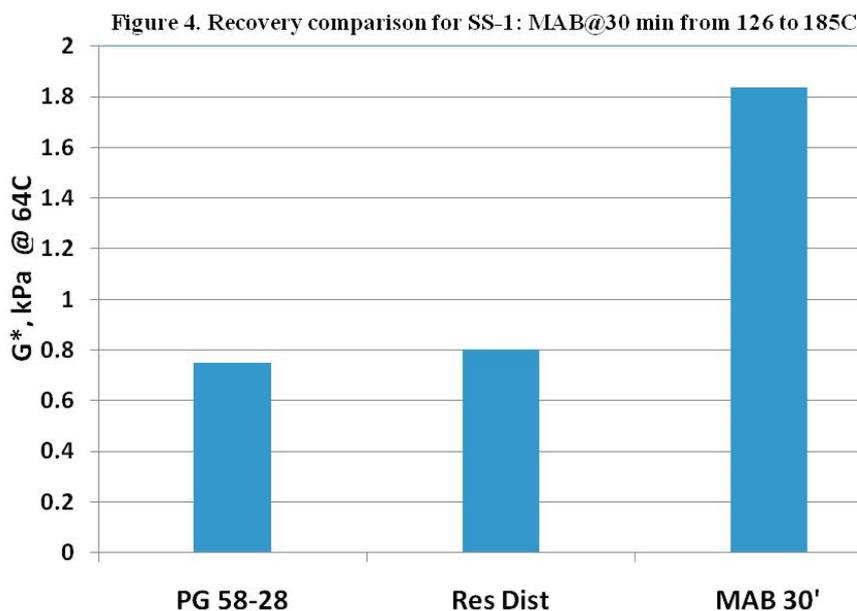
3.0 Results and Discussion

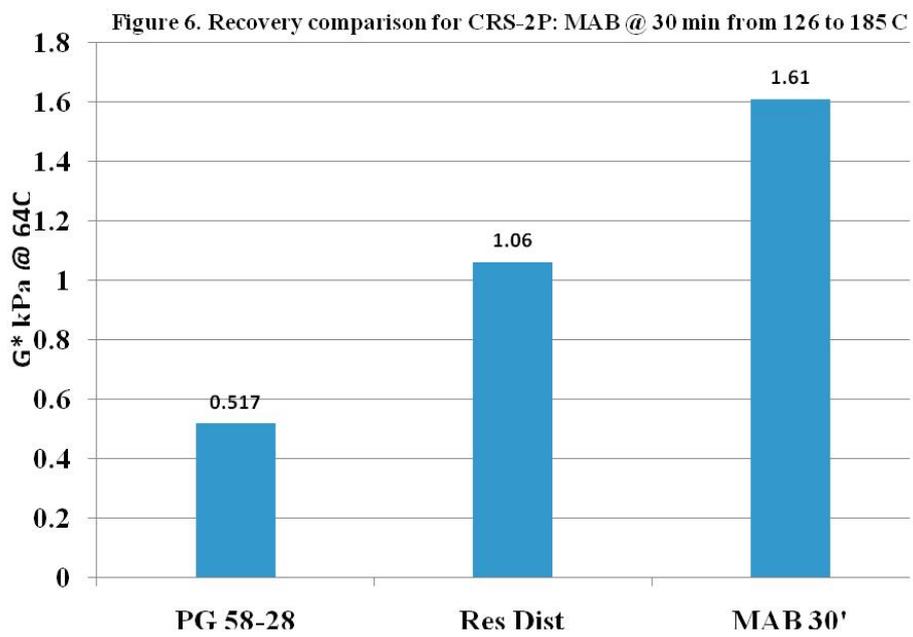
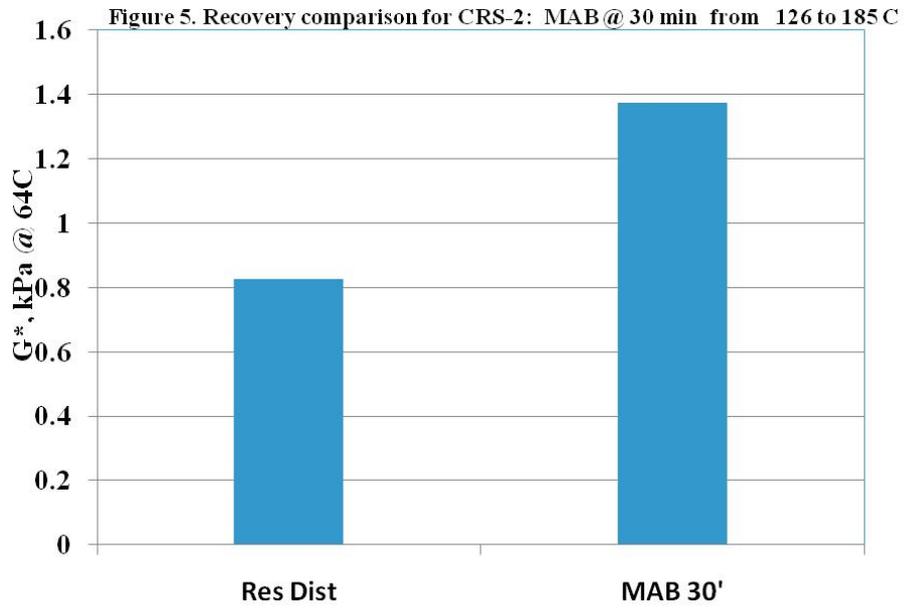
Table I shows values of the complex shear modulus, G^* , obtained from measurements with the dynamic shear rheometer for residues obtained from several different emulsions. The residues were obtained from several emulsified asphalts and recovered by MAB-DSR and by distillation procedures. The last three rows are values obtained from a lower operation temperature of the MAB. As discussed before G^* increases with MAB time of recovery at a given temperature.

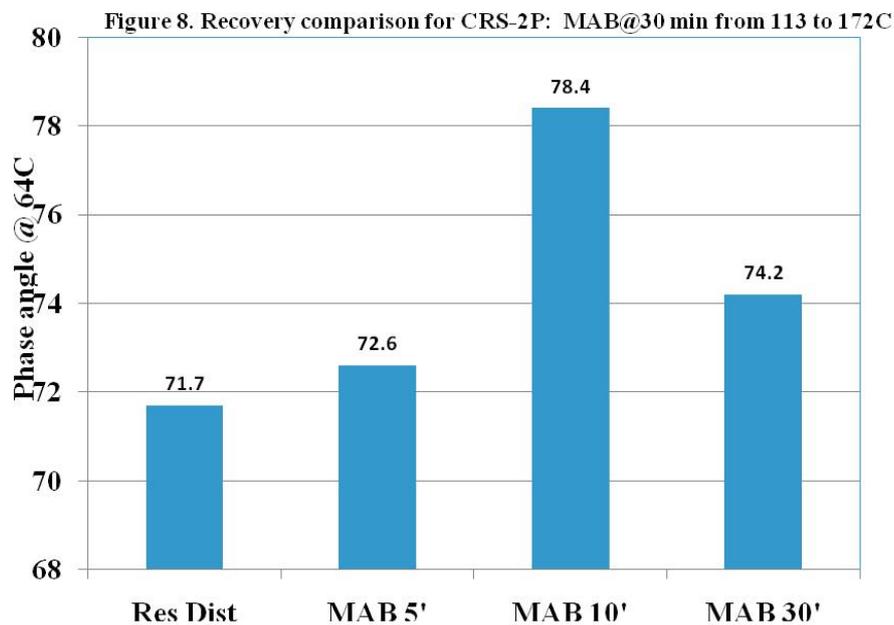
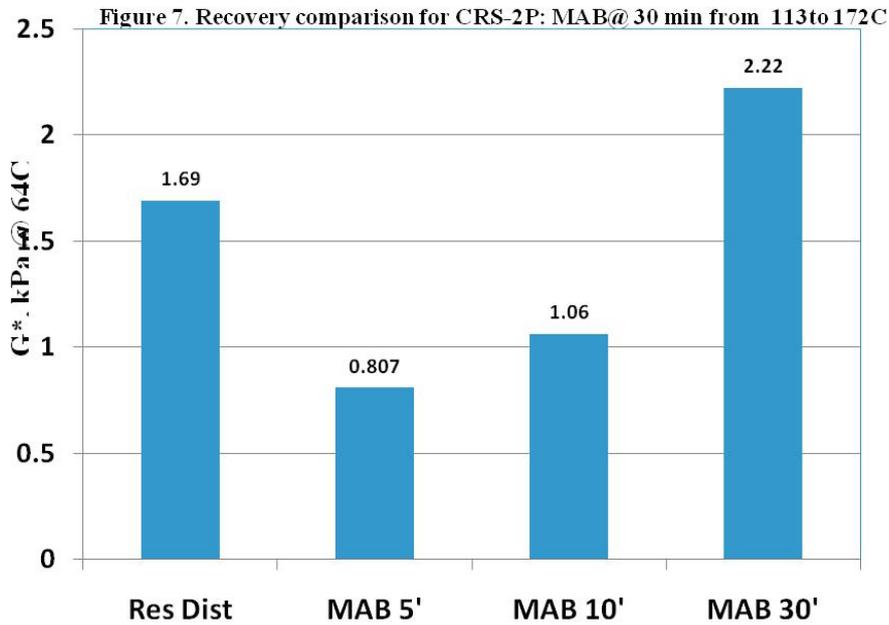
Table I. Complex Modulus, G^* for Residue Recovery Procedures

Emulsion Types/Procedure	Distillation, ASTM D 6997	MAB 5'	MAB 10'	MAB 30'	MAB Temperature profile
CRS-2	0.466	n/d	n/d	0.949	126 to 185°C
CRS-2P	0.825	n/d	n/d	1.375	126 to 185°C
CRS-2	0.547	n/d	n/d	0.805	113 to 172°C
CRS-2P	1.3	n/d	0.759	2.03	113 to 172°C
CRS-2P	1.69	0.807	1.06	2.22	113 to 172°C

The results are best summarized in graphical form and given in Figures 4 through 8. The higher G^* is due to the higher recovery temperatures and/or time of recovery as can be seen in Figure 7 for the CRS-2P residue. For this recovery the G^*_{MAB} increases with time and between 10 and 30 min G^*_{MAB} is comparable to G^*_{DIST} .







5. Conclusions

The MAB-DSR procedure is shown to be a feasible analytical method to obtain an emulsified asphalt residue and its G^* value. The temperatures used in this study were higher than previous work but the purpose here was to show the recovery in a silicone mold. It has been shown that even at these higher temperatures a G^* MAB can be comparable to G^* DIST provided the time is kept under 25 minutes. The higher oxidation of the residue during recovery for the MAB-DSR method is due to the

higher temperatures used and the the static nature of the recovery procedure, where the higher surface-to-volume ratio of the sample is higher than that for the distillation method. The MAB residue determination has a shorter test cycle time and used less material compared to other methods where testing times are at least five times as long and samples size is significantly larger.. Further work will be developed at the lower temperatures now that it has been shown that recovery in a silicone mold is not an issue.

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APPENDIX I

Silicone mold (25 mm) on scale



Silicone mold with weighed emulsified asphalt sample



Silicone mold with emulsified asphalt sample in oven
(if no MAB is available)

