



TECHSUMMARY December 2014

SIO No. 30000163 / LTRC Project No. 11-2B

Evaluation of Dynamic Shear Rheometer Tests for Emulsions

INTRODUCTION

Currently, the Louisiana Department of Transportation and Development (DOTD) requires ductility tests at 25°C according to AASHTO T51 for emulsions, force ductility ratio at 4°C, and elastic recovery at 10°C according to AASHTO T300 and AASHTO T301, respectively, for polymer modified emulsions. The fact that polymer-modified asphalt is strain dependent creates questions regarding purchase specifications based on constant strain controlled tests. In developing performance grade (PG) specifications for emulsions, one of the tests proposed in several reports is the multiple stress creep recovery (MSCR) test according to ASTM D7405. This test determines percent elastic recovery and non-recoverable creep compliance and measures the damage behavior in the linear and nonlinear range. Compared with the current binder protocol, a repeated creep test protocol (or some other method that varies the frequency and strain and measures the accumulated permanent strain of the binder) represents an improvement in the theoretical and practical concepts that will better rate the binder properties related to performance.

OBJECTIVE

The main objective of this study was to evaluate the dynamic shear rheometer (DSR)-based test parameters to replace the elastic recovery test (AASHTO T301) and the force ductility test (AASHTO T300) using a ductility bath for emulsified asphalt residues. It compared MSCR test results at various applicable stresses with elastic recovery and force ductility test results. One of the important objectives was to develop a new residue recovery method that will need shorter time periods and lower temperatures. Also, this study evaluated the use of a rotational viscometer replacing the Saybolt Furl viscometer for the viscosity of emulsion state.

SCOPE

Temperature sweep, strain sweep, frequency sweep, and multiple stress creep and recovery tests of commonly used emulsion residues were performed. The DSR-based test parameters were evaluated to replace the elastic recovery test (AASHTO T301) and force ductility test (AASHTO T300). Residues were obtained using five different methods to develop a lower duration and lower temperature recovery method. Rotational viscometer was used to develop emulsion state viscosity test specifications. Twenty different (by source and type) asphalt emulsions including anionic, cationic, slow setting, rapid setting, polymer-modified, latex-modified, and neat emulsions were used. In addition to that, un-aged bases of several emulsions were tested.

METHODOLOGY

A strain sweep test was performed between 2 percent and 52 percent strain at 10 percent intervals at 52°C and at 70°C. A frequency sweep test was performed between 0.1 rad/s and 100 rad/s angular frequency at 10°C, 25°C, 52°C, and 70°C to draw rheological master curves at 25°C. A temperature sweep test was performed between 52°C and 94°C at 6°C intervals using an angular frequency of 10 rad/s and 12 percent strain to develop a four-element Burgers model. Multiple stress creep and recovery testing was performed at 10°C, 25°C, 58°C, 70°C, and one equal-stiffness (2.2 kPa) temperature according to AASHTO TP70 method. At 2.2 kPa stiffness temperature, MSCR was also performed at 0.2 and 0.5 kPa stresses in addition to the standard 0.1 and 3.2 kPa stresses. The elastic recovery test was performed at 10°C and 25°C according to AASHTO T301 and the force ductility test was performed at 4°C according to AASHTO T300. In order to develop a low temperature low duration recovery method, a comparative study among ASTM D7497 (24 hr. at 25°C and 24 hr. at 60°C), ASTM D6934 (3 hr. at 163°C), Texas DOT method (6 hr. at 60°C), vacuum dry method [6 hr. at 60°C (a method developed in this study)] and field curing were performed. For viscosity in the emulsion state, a multiple step shear method was used. The rotation was started at 2 rpm and was increased gradually from 20 to 100 rpm and 20, 30, 50, 60, and 100 was selected for this study.

LTRC Report 519

Read online summary or final report:
www.ltrc.lsu.edu/publications.html

PRINCIPAL INVESTIGATOR:

N. "Wasi" Wasiuddin
Louisiana Tech University

LTRC CONTACT:

Bill King, P.E.
225-767-9129

FUNDING:

State: TT-Reg

Louisiana Transportation Research Center

4101 Gourrier Ave
Baton Rouge, LA 70808-4443

www.ltrc.lsu.edu

CONCLUSIONS

Findings on Elastic Recovery and Force Ductility Relationships

It was observed that emulsions prepared of hard pen base asphalt have relatively lower strain tolerance, i.e. relatively higher drop from initial complex modulus.

Complex modulus master curves constructed at 25°C demonstrate that all of the 20 emulsion residues possessed significant differences in complex modulus at lower frequencies (or higher temperatures).

$G^*/\sin\delta$ curves drawn at temperatures from 52°C to 94°C show that hard pen base asphalt emulsions have higher stiffness than all other emulsions. One of the findings of this study is that as temperature increases, phase angle of an emulsion residue increases, reaches a maximum, and then decreases. It was observed that the maximum phase angle of all the non-hard pen polymer-modified asphalt emulsion residue falls between 75° to 85°. Therefore, a maximum phase angle in the range of 75° to 85° may be used as a criterion for polymer identification as observed from the 20 emulsions tested in this study.

A general trend in MSCR test results is that as temperature increases, percent recovery decreases. All non-polymer modified emulsions follow the trend. For example, the average percent recoveries of CHFRS-2P at 0.1 kPa creep stress are 72.7, 67.5, 36.8, and 38.1, respectively, at 10°C, 25°C, 58°C, and 70°C.

In this study, it was observed that the effect of creep stress on percent recovery (MSCR) is logarithmic. A general trend observed is that as the creep stress increases, the percent recovery decreases.

The Burgers model prepared for 0.1kPa creep stress and recovery at 2.2 kPa stiffness temperatures demonstrate that average Maxwell viscosity, n_o , values of polymer modified emulsions are higher than those of non-polymer modified and non-hard pen base asphalt emulsions. In contrast, the average Kelvin viscosity, n_i , values of polymer modified emulsions are significantly lower than those of non-polymer modified and non-hard pen base asphalt emulsions.

A significant finding of this study is that higher percent recovery values of MSCR indicate the presence of polymer and MSCR percent recovery is correlated with elastic recovery of AASHTO T301. It was observed that the highest coefficient of determination of linear correlation was obtained between MSCR percent recovery at 10°C and elastic recovery (AASHTO T301) at 58°C. For this strongest relationship, R2 values for 0.1 kPa and 3.2 kPa creep stresses are 0.961 and 0.938, respectively.

It was concluded from this study that percent recovery of MSCR and phase angle can be used to replace force ductility requirements (AASHTO T300). It is recommended that at 58°C, a maximum phase angle of 81° and a minimum MSCR percent recovery (at 0.1kPa creep stress) of 30 are specified to replace force ductility test (AASHTO T300). These criteria are applicable for emulsion residues prepared according to the low temperature evaporative method specified in ASTM D7497.

Findings on Recovery Methods

The new recovery process proposed in the study using a vacuum drying oven is the most efficient low-temperature method to ensure complete drying of the sample within a short period of time. This method ensures two benefits at the same time. One is higher drying of the sample relative to any

other methods currently in practice and the other is the system which is totally void of air; theoretically, this method produces a residue that should not experience any oxidative aging. For neat emulsions, this method produces residue that is not unnecessarily aged and similar to base binder in stiffness which validates the previous statement.

Curing time can be reduced to six hours using this method. Six hours of curing time successfully identified the improvement of polymer and latex modification. It can also differentiate between polymer, latex, and neat emulsion within this period of time.

It can be concluded that the proposed low-temperature vacuum drying method allows for the recovery of modified binders without degrading the polymer network.

Findings on Rotational Viscosity

This study proposes the use of a rotational viscometer to find the viscosity of the emulsion in the emulsion state. Two ideal test conditions have been found to evaluate the viscosity of the emulsion in the emulsion state based on data repeatability: 50 rpm at 30°C and 30 rpm at 60°C.

A strong correlation has been found between water content and emulsion viscosity at 30°C temperature with an R2 value of more than 0.94.

Current specification of viscosity range for low and high viscous emulsions using the Saybolt Furol viscometer to ensure quality control and quality assurance can be replaced by the rotational viscometer. The ranges recommended are 220 - 730 cP and 5 - 90 cP with 98 percent probability at 50 rpm and 30°C for high and low viscous emulsions, respectively.

RECOMMENDATIONS

It is recommended that MSCR (AASHTO TP70) at 58°C be adopted to replace the elastic recovery test (AASHTO T301) for modified emulsions. At 0.1kPa creep stress, a minimum percent recovery of 25 and at 3.2kPa, a minimum percent recovery of 9 are recommended to identify the presence of polymer replacing the elastic recovery test (AASHTO T301). It is recommended that at 58°C, a maximum phase angle of 81° and a minimum MSCR percent recovery (at 0.1kPa creep stress) of 30 are specified to replace force ductility test (AASHTO T300). These criteria are applicable for emulsion residues prepared according to the low temperature evaporative method specified in ASTM D7497. However, further research is recommended for a better DSR-based parameter to replace the force ductility test. Also, the possibilities of using an extensional rheometer for the replacement of force ductility should be investigated.

A new low-temperature, low-duration residue recovery method called "vacuum dry method" has been developed and proposed. Although in this study only rapid setting emulsion was used, further research is needed to verify the time reduction possibility and the rate of the polymer network development using slow setting emulsions.

A multiple step shear method as well as new specifications for emulsion state viscosity have been proposed using a rotational viscometer.