

# **Screening Test of Gilsonite Application**

A report submitted to

SealMaster

by

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### **SUMMARY**

The major objective of this study is to investigate the properties of asphalt binder modified with different percentages of gilsonite over a wide range of climatic conditions. It was addressed by determining the performance grade (PG) of gilsonite-modified binders according to Superpave specifications. The base asphalt used in this study was the neat asphalt with PG 52-28, and 5 different percentages of gilsonite (0, 3%, 6%, 10%, and 12% of total binder content) were introduced.

With the increase of gilsonite content from 0% to 12%, the PG high temperature increased from 52°C to 70°C. However, the PG low temperature also increased from -28°C to -22°C. The results indicate that the addition of gilsonite tends to improve the rutting resistance of asphalt binders, however, increases the tendency for fatigue cracking and low temperature cracking. Adding low content of gilsonite (i.e. 3% within the scope of this study), the modified binder presents improved rutting resistance without any compromise of resistance to low temperature cracking.

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# Screening Test of Gilsonite Application

## INTRODUCTION

The use of additives to improve the performance of hot mixes continues to generate worldwide interest and attention. Among many modifiers, gilsonite is known for its easy use and good affinity with asphalt. An area of major interest is the development of high stability asphalt binders that maintain good performance at low temperatures with addition of gilsonite.

## RESEARCH OBJECTIVE

The major objective of this study is to investigate the properties of asphalt binder modified with different percentages of gilsonite over a wide range of climatic conditions through the determination of performance grade (PG) of gilsonite-modified binders according to Superpave criteria.

## EXPERIMENTAL WORK

This study conducted by the UAF determined the performance grade (PG) of the gilsonite-modified binders to assess the high and low temperature limits for adequate performance (i.e. resistances to rutting, fatigue cracking and thermal cracking) according to Superpave criteria. The base asphalt used in this study was the neat asphalt with PG 52-28, and the granulated gilsonite used was by SealMaster Company. 5 different percentages of gilsonite (0, 3%, 6%, 10%, and 12% of total binder content) were introduced to produce modified asphalt binders, and effect of gilsonite content on the properties of modified asphalt binders were evaluated.

## Test Procedures

Table 1 summarizes the tests conducted in the laboratory. For each test, three replicates were provided for each temperature measured. All tests were performed according to AASHTO PP6 (Standard Practice for Grading or Verifying Performance Grade of an Asphalt Binder). Characterization tests were performed on original (unaged binder), Rolling Film Oven aged residue (i.e. Rolling Film Oven Test – RTFOT), and Pressure Aging Vessel (PAV) residue. RTFOT simulates short-term aging that occurs at a hot mix production plant and also during the construction phase. The PAV test is supposed to simulate long term aging for pavements in service.

Table 1. Superpave Tests for Gilsonite-modified Binders.

Binder aging	Tests	Performance parameter	Test equipment/model
Original binder	Flash point (AASHTO T48)	safety	Gilson/PT-6
	Rotational viscosity (ASTM D4402)	Handling and pumping	Brookfield/RV DV-III
	DSR test (AASHTO TP5)	$G^*/\sin\delta$ - resistance to rutting	Rheometric Scientific/ARES-RAA
Short term aging (RTFO)	DSR test (AASHTO TP5)	$G^*/\sin\delta$ - resistance to rutting	Rheometric Scientific/ARES-RAA
	Mass loss	the amount of volatiles evaporating during the mixing and construction process	Mettler Toledo Balance
Long term aging (PAV residue)	DSR test (AASHTO TP5)	$G^*(\sin\delta)$ - resistance to fatigue cracking	Rheometric Scientific/ARES-RAA
	BBR test (AASHTO TP1)	Creep stiffness and m-value – resistance to thermal cracking	Cannon Instrument/TE-BBR
	Direct tension (AASHTO TP3)	Resistance to thermal cracking	Instron/BTI-3

*Testing of Original Binder*

Testing of original binder includes flash point test (AASHTO T48), rotational (Brookfield) viscosity test (ASTM D4402), and Dynamic shear rheometer (DSR) test (AASHTO TP5). The flash point test ensures safety by measuring the temperature to which the binder could be heated without flashing in the presence of a flame. The specification for all binders is a minimum temperature of 230 °C. The rotational (Brookfield) viscosity test utilizes a Brookfield viscometer to ensure ease of pumping and handling of the binder at the hot mix production plant. This is attained by specifying a maximum viscosity of 3 Pas ( $\approx 3000\text{mm}^2/\text{s}$ ) at 135 °C. The DSR is conducted on both the original and aged binder. A controlled-strain DSR is used to measure the viscoelastic behavior at different temperatures of the binder in terms of complex modulus ( $G^*$ ) and phase angle ( $\delta$ ). The DSR applies a torque to a thin film of binder specimen placed between two plates at a frequency of 10 radians per second. The applied torque and resulting shear strain are used in the computation of the complex modulus and phase angle. The specification requires determining the temperature that corresponds to a minimum value of 1.0 kPa for  $G^*/\sin(\delta)$ .

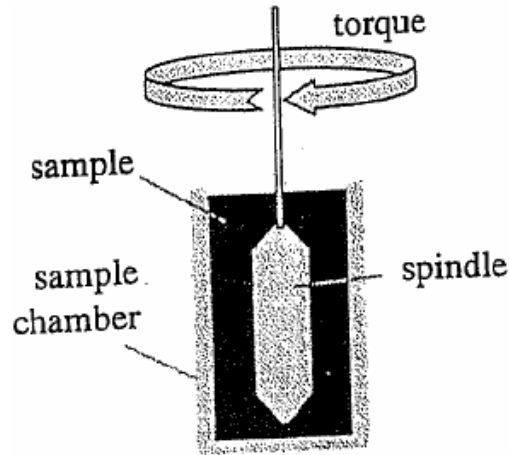


Figure 1. Principle of rotational viscometer.

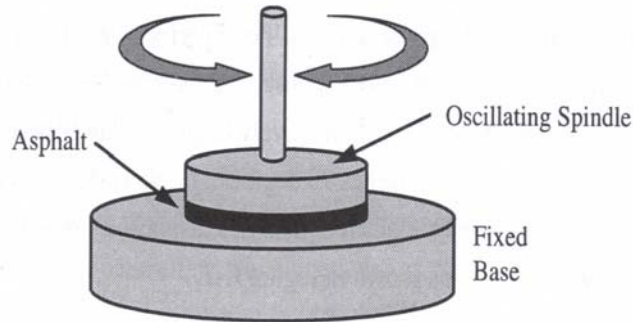


Figure 2. Principle of DSR.

### *Testing of RTFO Aged Residue*

RTFO test (AASHTO T240) exposes fresh thin films of binder to heat (163 °C) and air for 85 minutes by rotating coated bottles (15 revolutions/minute) and blowing air into the bottles (4000 ml/minute). The average percent mass loss is calculated after testing. The specification of 1% maximum mass loss guards against binders that age excessively. The RTFO residue is tested again using the DSR. In this case the limit on  $G^*/\sin\delta$  required is 2.2 kPa for a loading rate of 10 radians/second. DSR tests on the original and RTFO aged binders are supposed to evaluate the binder's resistance to rutting.

### *Testing of PAV Aged Residue*

The RTFO residue is aged again in a PAV (AASHTO PP1) to simulate long term aging. In this case, the binder is subjected to high temperature (90°C, 100°C, or 110°C) and pressure of 2070 kPa for 20 hours. The PAV residue is then tested using DSR to evaluate the fatigue resistance of the binder. The specification in this case requires determining the temperature associated with a maximum  $G^*\sin\delta$  of 5000 kPa for a loading rate of 10 radians/second. Bending Beam Rheometer (BBR) Test (AASHTO TP1) is used to evaluate the stiffness of the PAV aged binder at low temperatures. The BBR subjects a small beam of binder to a constant creep load and measures the resulting deflection at a temperature related to the

anticipated lowest pavement service temperature. Using simple beam theory, the binder stiffness is calculated. The creep rate (m-value) defined as the rate of change of stiffness with time is also determined. The stiffness at 60 seconds must be less than 300 MPa, and the m-value at this time of loading must be at least 0.30 in order to meet the binder specification. If the stiffness is between 300 MPa and 600 MPa, then the direct tension test (DTT) (AASHTO TP3) should be used. In this test, a dog-bone shaped sample of binder is pulled at a slow rate of 1 mm/minute at low temperatures. The failure strain (defined at the maximum recorded load during the test) is determined. The specification requires that the failure strain be at least 1%. The m-value requirement must be satisfied in both cases.

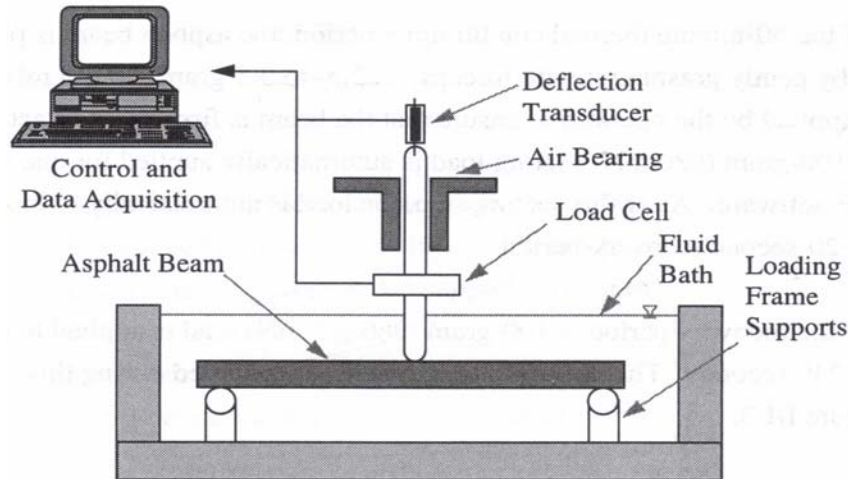


Figure 3. Principle of BBR.

Using the suite of tests described above, the PG grade of gilsonite modified binders was determined according to AASHTO MP1 (Standard Specification for Performance Graded Asphalt Binder) shown in Table 2.

Table 2. Performance Grade Asphalt Binder Specifications (AASHTO MP1).

Performance Grade	PG 46				PG 52				PG 58				PG 64									
	-34	-40	-46	<46	-10	-16	-22	-28	-34	-40	-46	-16	-22	-28	-34	-40	-10	-16	-22	-28	-34	-40
Average 7-day Maximum Pavement Design Temperature, °C <sup>a</sup>	<64																					
Minimum Pavement Design Temperature, °C <sup>a</sup>	<58																					
	Original Binder																					
Flash Point Temp, T <sub>48</sub> : Minimum °C	230																					
Viscosity, ASTM D 4402: b Maximum, 3 Pa·s (3000 cP), Test Temp, °C	135																					
Dynamic Shear, TP5: c G*/sin δ, Minimum, 1.00 kPa Test Temperature @ 10 rad/s, °C	46				52				58				64									
	Rolling Thin Film Oven (T 240) or Thin Film Oven (T 179) Residue																					
Mass Loss, Maximum, %	1.00																					
Dynamic Shear, TP5: G*/sin δ, Minimum, 2.20 kPa Test Temp @ 10 rad/sec, °C	46				52				58				64									
	Pressure Aging Vessel Residue (PP1)																					
PAV Aging Temperature, °C <sup>d</sup>	90				90				100				100									
Dynamic Shear, TP5: G*/sin δ, Maximum, 5000 kPa Test Temp @ 10 rad/sec, °C	10	7	4	25	22	19	16	13	10	7	25	22	19	16	13	10	7	25	22	19	16	16
Physical Hardening <sup>e</sup>	Report																					
Creep Stiffness, TP1: f S, Maximum, 300 MPa m-value, Minimum, 0.300 Test Temp, @ 60 sec, °C	-24	-30	-36	0	-6	-12	-18	-24	-30	-36	-6	-12	-18	-24	-30	0	-6	-12	-18	-24	-30	-30
Direct Tension, TP3: f Failure Strain, Minimum, 1.0% Test Temp @ 1.0 mm/min, °C	-24	-30	-36	0	-6	-12	-18	-24	-30	-36	-6	-12	-18	-24	-30	0	-6	-12	-18	-24	-30	-30



Table 2. (Continued).

Performance Grade	PG 70				PG 76				PG 82							
	-10	-16	-22	-28	-34	-40	-10	-16	-22	-28	-34	-10	-16	-22	-28	-34
Average 7-clay Maximum Pavement Design Temperature, °C <sup>a</sup>	<70															
Minimum Pavement Design Temperature, °C <sup>a</sup>	<76															
Flash Point Temp, T <sub>48</sub> : Minimum °C	Original Binder															
Viscosity, ASTM D 4402: b Maximum, 3 Pa·s (3000 cP), Test Temp, °C	230															
Dynamic Shear, TP5: c G*/sin δ, Minimum, 1.00 kPa Test Temperature @ 10 rad/s, °C	70															
Mass Loss, Maximum, %	1.00															
Dynamic Shear, TP5: G*/sin δ, Minimum, 2.20 kPa Test Temp @ 10 rad/sec, °C	76															
Rolling Thin Film Oven (T 240) or Thin Film Oven (T 179) Residue	82															
PAV Aging Temperature, °C <sup>d</sup>	Pressure Aging Vessel Residue (PP1)															
Dynamic Shear, TP5: G*/sin δ, Maximum, 5000 kPa Test Temp @ 10 rad/sec, °C	100(110)				100(110)				100(110)				100(110)			
Physical Hardening <sup>e</sup>	34	31	28	25	22	19	37	34	31	28	22	40	37	34	31	28
Creep Stiffness, TP1: f S, Maximum, 300 MPa m-value, Minimum, 0.300 Test Temp, @ 60 sec, °C	Report															
Direct Tension, TP3: f Failure Strain, Minimum, 1.0% Test Temp @ 1.0 mm/min, °C	0	-6	-12	-18	-24	-30	0	-6	-12	-18	-24	0	-6	-12	-18	-24
	0	-6	-12	-18	-24	-30	0	-6	-12	-18	-24	0	-6	-12	-18	-24

## Test Results and Discussion

Results of Superpave binder tests are summarized in Table 3. Detailed testing results are presented in the Appendix. DTT strain data were not available, because based on the specification, the DTT strain is only necessary when m-value requirement is satisfied but the creep stiffness requirement is not. The DTT is not required if the creep stiffness is less than 300 MPa. As shown in Table 3, for different gilsonite additions, the PG high temperature ranges from 52°C to 70°C, while low temperature ranges from -28°C to -22°C. According to Superpave specification, a PG 52-28 grade is intended for use in an environment where an average seven-day maximum pavement temperature of 52°C and a minimum pavement design temperature of -28°C, are likely to be experienced. A PG 70-22 grade is intended for use in an environment where an average seven-day maximum pavement temperature of 70°C and a minimum pavement design temperature of -22°C, are likely to be experienced, etc.

Figure 4 illustrates PG grading of gilsonite-modified asphalt binders. Since the Superpave asphalt binder specification is meant to be performance based, it addresses three primary performance parameters of asphalt pavements: permanent deformation (rutting), fatigue cracking, and low temperature (thermal) cracking.

Table 3. Summary of Superpave Binder Test Results.

Gilsonite content, %	Flash point, °C	Mass loss, %	Viscosity @ 135°C	Grade Temp. at which specified criterion is satisfied, °C						PG grade	
				DSR origin.	DSR RTFO	DSR PAV	BBR S	BBR m-value	DTT strain	High	Low
0	310	0.59	0.2168	52	52	16	-18	-18	-	52	-28
3	316	0.61	0.2555	58	58	19	-18	-18	-	58	-28
6	310	0.21	0.3400	58	58	22	-12	-12	-	58	-22
9	316	0.24	0.3842	64	64	25	-12	-12	-	64	-22
12	316	0.23	0.5208	70	70	25	-12	-12	-	70	-22

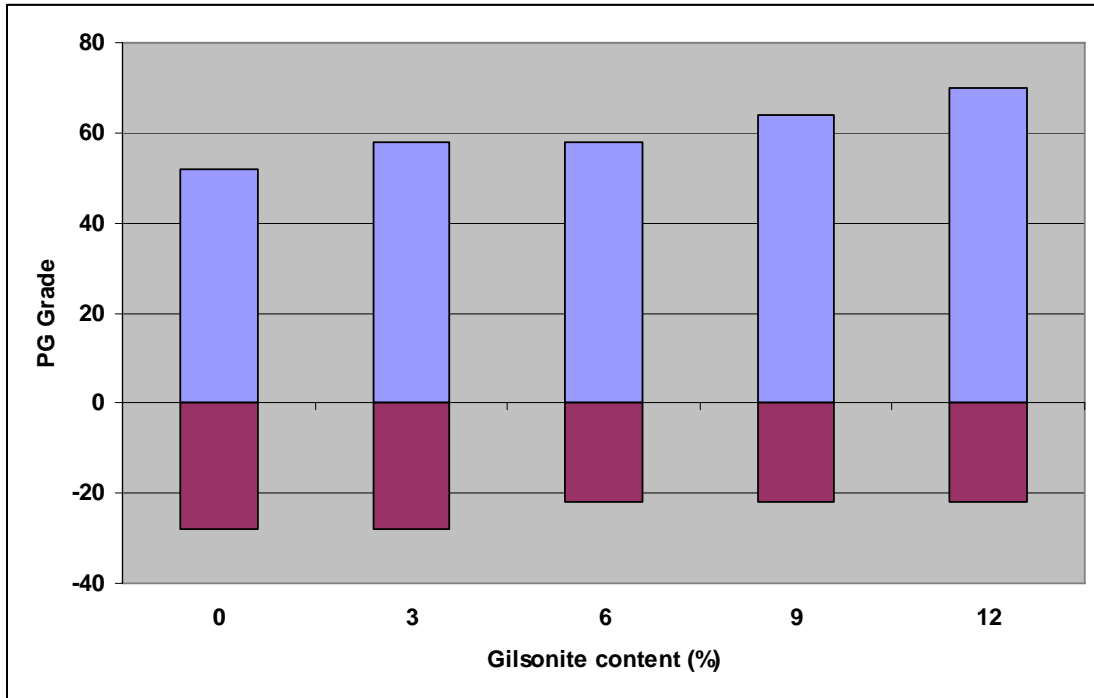


Figure 4. PG Grading of Gilsonite-modified binders.

*Effects of Gilsonite on Rutting Performance of Asphalt Binders*

It can be seen from Table 1 that the addition of gilsonite increases the viscosity of an asphalt at high pavement temperatures. This effect increases as the level gilsonite addition increases. The Superpave binder specification uses a rutting factor,  $G^*/\sin\delta$ , which is a measure of asphalt binder's stiffness or rut resistance at high pavement service temperatures. Figure 5 illustrates  $G^*/\sin\delta$  measured at 58°C for both original binder and RTFO aged binders. For both conditions, rutting factor increases with the increase of gilsonite addition. The high temperature of PG grade is determined based on that the rutting factor must be at least 1.00 kPa for the original asphalt binder and a minimum of 2.20 kPa for the RTFO aged asphalt binder when tested by DSR. According to the PG grading, with the increase of gilsonite addition from 0 to 12% of total binder weight, the PG high temperature increases from 52°C to 70°C, indicating a higher rutting resistance of the binder.

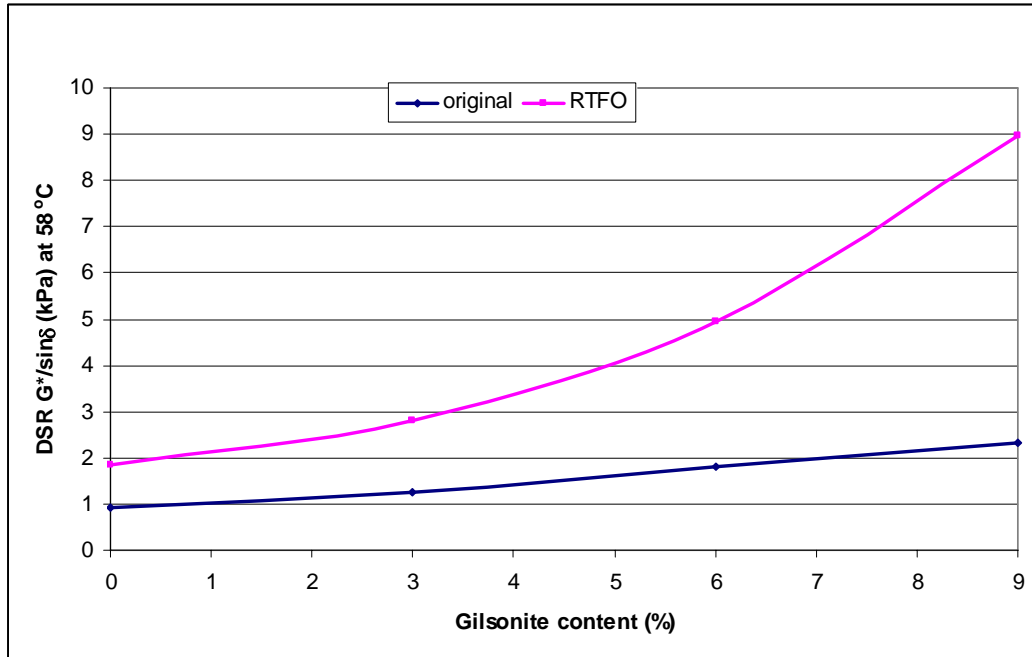


Figure 5. Effects of Gilsonite Addition on Rutting Factor.

*Effects of Gilsonite on Fatigue Resistance of Asphalt Binders*

The specification uses a fatigue factor,  $G^*\sin\delta$ , which represents asphalt binder's resistance to fatigue cracking. The specification has a maximum limit of 5000 kPa for  $G^*\sin\delta$  for the binder subjected to PAV aging, and tested at intermediate pavement service temperature. As shown in Figure 6, the intermediate pavement service temperature increases with the increase of gilsonite addition, which implies higher fatigue factor at same intermediate temperature, and associated reduced resistance to fatigue cracking.

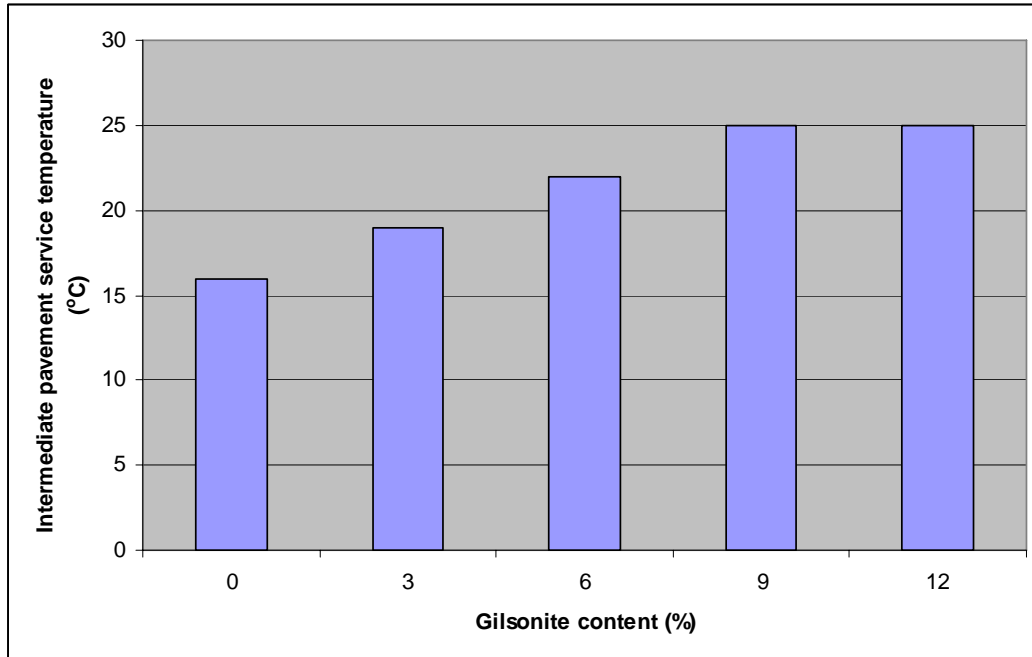


Figure 6. Effects of Gilsonite Addition on Fatigue Resistance.

*Effects of Gilsonite on Resistance to Low Temperature Cracking of Asphalt Binders*

A lower creep stiffness and a higher m-value of PAV aged binders at a low temperature usually mean a higher resistance to low temperature cracking of pavement materials. Figure 7 illustrates creep stiffness and m-value of PAV age binders with different additions of gilsonite measured at  $-18^{\circ}\text{C}$  by BBR test. With the increase of gilsonite content, creep stiffness increases while m-value decreases. In addition, according to the PG grading (Table 3 and Figure 4), the PG low temperature increases from  $-28^{\circ}\text{C}$  to  $-22^{\circ}\text{C}$ , indicating that gilsonite addition increases the tendency for low temperature cracking of the base asphalts.

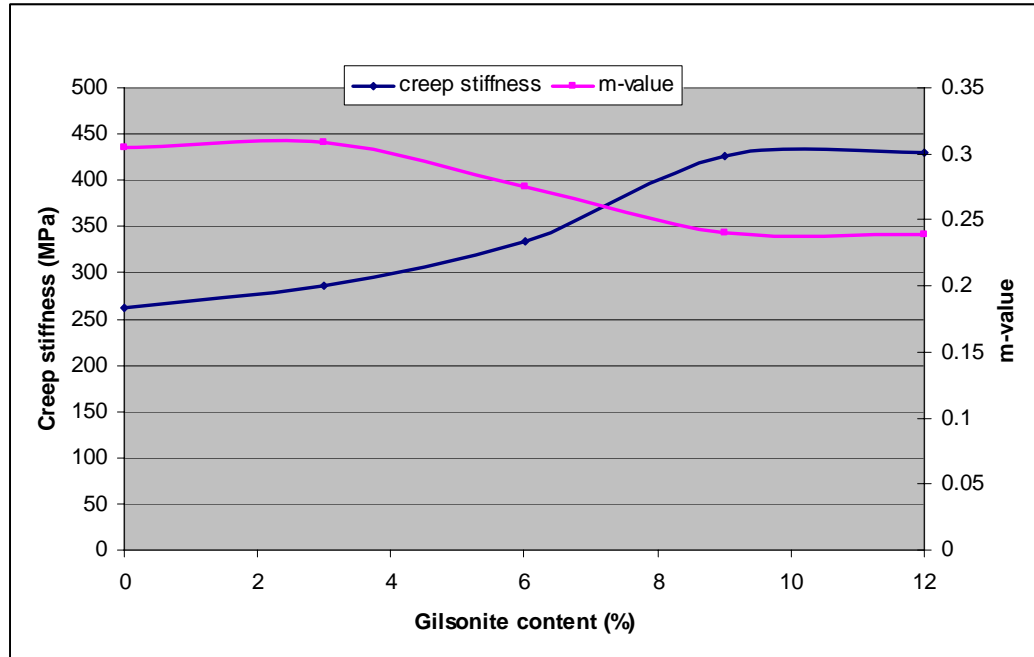


Figure 7. Effects of Gilsonite Addition on Creep Stiffness and m-value.

## CONCLUSIONS

In this study, the properties of asphalt binder modified with different additions of gilsonite over a wide range of climatic conditions were investigated by determining the PG of the gilsonite-modified binders according to Superpave specifications. The base asphalt used in this study was the neat asphalt with PG 52-28, and 5 different percentages of gilsonite (0, 3%, 6%, 10%, and 12% of total binder content) were introduced.

With the increase of gilsonite content from 0% to 12%, the PG high temperature increased from 52°C to 70°C. However, the PG low temperature also increased from -28°C to -22°C. The results indicate that the addition of gilsonite tends to improve the rutting resistance of asphalt binders, however, increases the tendency for fatigue cracking and low temperature cracking. Adding low content of gilsonite (i.e. 3% within the scope of this study), the modified binder presents improved rutting resistance without any compromise of resistance to low temperature cracking.

## APPENDIX A- TEST RESULTS SUMMARY

Table A-1. Results summary of binder with 0% gilsonite.

Binder	Parameters	Specification	Temperature measured	Measured parameters	Pass/Fail
Original	Flash point temperature	230°C, Min.	-	310°C	Pass
	Viscosity at 135 °C	3 Pa·s, Max.	-	0.2168 Pa·s	Pass
	DSR $G^*/\sin\delta$ at 10 rad/s	1.00 kPa, Min.	52	2.1186 kPa	Pass
58			0.9357 kPa	Fail	
RTFO aged	Mass loss	1%, Max	-	0.59%	Pass
	DSR $G^*/\sin\delta$ at 10 rad/s	2.20 kPa, Min.	52	4.3360 kPa	Pass
			58	1.8562 kPa	Fail
			64	0.8350 kPa	Fail
PAV aged	DSR $G^*\cdot\sin\delta$ at 10 rad/s	5000 kPa, Max.	16	3100 kPa	Pass
	BBR- Creep stiffness S	300 MPa, Max.	-18	261.33 MPa	Pass
			-24	583.4 MPa	Fail
	BBR- m-value	0.300, Min.	-18	0.3043	Pass
-24			0.238	Fail	

Table A-2. Results summary of modified binder with 3% gilsonite.

Binder	Parameters	Specification	Temperature measured	Measured parameters	Pass/Fail
Original	Flash point temperature	230°C, Min.	-	316°C	Pass
	Viscosity at 135 °C	3 Pa·s, Max.	-	0.2555	Pass
	DSR G*/sinδ at 10 rad/s	1.00 kPa, Min.	52	2.9232	Pass
			58	1.2381	Pass
			64	0.5840	Fail
RTFO aged	Mass loss	1%, Max	-	0.61%	Pass
	DSR G*/sinδ at 10 rad/s	2.20 kPa, Min.	58	2.8071	Pass
			64	1.2029	Fail
			70	0.5745	Fail
PAV aged	DSR G*·sinδ at 10 rad/s	5000 kPa, Max.	19	4535	Pass
			16	6718	Fail
			13	10347	Fail
	BBR- Creep stiffness S	300 MPa, Max.	-18	286.33	Pass
			-24	589.33	Fail
	BBR- m-value	0.300, Min.	-18	0.3087	Pass
-24			0.2263	Fail	



Table A-3. Results summary of modified binder with 6% gilsonite.

Binder	Parameters	Specification	Temperature measured	Measured parameters	Pass/Fail
Original	Flash point temperature	230°C, Min.	-	310°C	Pass
	Viscosity at 135 °C	3 Pa·s, Max.	-	0.340	Pass
	DSR G*/sinδ at 10 rad/s	1.00 kPa, Min.	58	1.8195	Pass
			64	0.8434	Fail
RTFO aged	Mass loss	1%, Max	-	0.21%	Pass
	DSR G*/sinδ at 10 rad/s	2.20 kPa, Min.	58	4.9273	Pass
			64	2.1957	Fail
PAV aged	DSR G*·sinδ at 10 rad/s	5000 kPa, Max.	22	3629.8	Pass
			19	5268.2	Fail
			16	9132.2	Fail
	BBR- Creep stiffness S	300 MPa, Max.	-12	168.67	Pass
			-18	334.0	Fail
			-24	799.5	Fail
	BBR- m-value	0.300, Min.	-12	0.3357	Pass
			-18	0.2750	Fail
			-24	0.1715	Fail

Table A-4. Results summary of modified binder with 9% gilsonite.

Binder	Parameters	Specification	Temperature measured	Measured parameters	Pass/Fail
Original	Flash point temperature	230°C, Min.	-	316°C	Pass
	Viscosity at 135 °C	3 Pa·s, Max.	-	0.3842	Pass
	DSR G*/sinδ at 10 rad/s	1.00 kPa, Min.	58	2.3334	Pass
			64	1.0759	Pass
70			0.5383	Fail	
RTFO aged	Mass loss	1%, Max	-	0.24%	Pass
	DSR G*/sinδ at 10 rad/s	2.20 kPa, Min.	58	8.9769	Pass
			64	4.1223	Pass
			70	1.8890	Fail
PAV aged	DSR G*·sinδ at 10 rad/s	5000 kPa, Max.	25	3488.2	Pass
			22	5224.1	Fail
			19	7030.4	Fail
	BBR- Creep stiffness S	300 MPa, Max.	-12	203.33	Pass
			-18	426.50	Fail
	BBR- m-value	0.300, Min.	-12	0.3067	Pass
-18			0.2405	Fail	

Table A-5. Results summary of modified binder with 12% gilsonite.

Binder	Parameters	Specification	Temperature measured	Measured parameters	Pass/Fail
Original	Flash point temperature	230°C, Min.	-	316°C	Pass
	Viscosity at 135 °C	3 Pa·s, Max.	-	0.5208	Pass
	DSR $G^*/\sin\delta$ at 10 rad/s	1.00 kPa, Min.	64	3.3029	Pass
			70	1.6144	Pass
76			0.8749	Fail	
RTFO aged	Mass loss	1%, Max	-	0.23%	Pass
	DSR $G^*/\sin\delta$ at 10 rad/s	2.20 kPa, Min.	64	5.6705	Pass
			70	2.5659	Pass
			76	1.2618	Fail
PAV aged	DSR $G^*\cdot\sin\delta$ at 10 rad/s	5000 kPa, Max.	25	4345.43	Pass
			22	5957.9	Fail
	BBR- Creep stiffness S	300 MPa, Max.	-12	197.33	Pass
			-18	429.67	Fail
	BBR- m-value	0.300, Min.	-12	0.3010	Pass
			-18	0.2393	Fail