Technical White Paper

Characterization of the Effects of Wax (Sasobit®) on Asphalt Binder

Fundamental Properties of Asphalts and Modified Asphalts III Product: FP 13

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INTRODUCTION

Warm mix asphalt (WMA) utilizes a broad range of processes and a number of additives, some of which are proprietary, that allow production of asphalt mix at significantly lower temperature than hot mix asphalt (HMA). The lower production temperatures substantially reduce asphalt fumes and plant emissions, and burner fuel requirements.

This report is concerned with Sasobit®, which is one of the more well known WMA additives. Sasobit® is manufactured by Sasol Wax in South Africa. It is a long-chain aliphatic hydrocarbon with a melting point of about 100ºC and is completely soluble in asphalt above 140ºC at typical dosage concentrations. At temperatures above the melting point it acts as a flow improver by reducing the viscosity of the asphalt and enabling mixing and compaction temperatures to be reduced by 10 to 30ºC.

Sasobit® is produced using the Fischer-Tropsch (F-T) process where hydrocarbons are synthesized from hydrogen and carbon monoxide. For a good summary of the F-T process see Schulz [1999]. Its molecular chain length ranges from 40 to more than 115 carbons atoms [Sasol Wax 2008]. By comparison, macrocrystalline bituminous paraffin waxes have carbon chain lengths ranging from C_{25} to C_{60}.

Although Sasobit® is the most common wax additive in WMA in the United States, it isn't the only wax additive available. For example, Asphaltan B®, which is a Montan wax, is naturally occurring and found in brown or lignite coal deposits. It is derived by solvent extraction [Romonta 2007]. Another example, is Isomerized Paraffin, which is a wax additive obtained by hydroisomerization of the substantially linear paraffin wax as obtained in an F-T process [Bobee et al. 2006].

In a study by Butz et al. [2000], six compositionally diverse asphalts ranging in asphaltene content from 7 to 20.6 percent (by weight) were modified with 3 and 4 percent F-T paraffin. The modified binders stored at 160°C remained stable, and the colloidal stability was not adversely affected. FT-paraffin structures were identified in thin films of the modified binders under an optical microscope at 400X. The observed structures were uniformly distributed and exhibited thread- and rod-shaped forms. Ring & Ball softening point increased by about 40 to 50°C, and at 4% FT-paraffin, was about the same as the softening point of the pure FT-paraffin. The Fraass break point remained practically unchanged. Addition of FT-paraffin led to a reduction in phase angle (δ) and an increase in the storage modulus (G').

A study by Kanitpong et al. [2007] on the properties and performance of F-T paraffin (Sasobit®) modified binders and mixes found: (1) the mix workability was improved, particularly with polymer modified binders, (2) higher resistance to densification under simulated traffic, and (3) a "neutral" effect on the resistance of asphalt mixes to moisture damage with relatively high mix temperatures (however, at relatively low mix temperatures some damage was observed, possibly due to the detrimental effects of trapped moisture in the mix).

More recent studies on Sasobit® modified binders include its impact on performance grade [Wu and Zeng 2011], aging behavior [Lee et al. 2009; Jamshidi et al. 2012; Banerjee et al. 2012] and rheological properties at high or low temperatures [Lui et al. 2011; Merusi and Giuliani 2011; Edwards et al. 2006; and Xiao et al. 2012]. It has been found that Sasobit® modified binders have higher complex moduli, lower creep compliances and phase angles than the control binders and the binders with other Warm Mix Asphalt (WMA) additives. An improvement in rutting resistance was reported regardless of asphalt types and grade [Xiao et al. 2012]. Wu and Zeng [2011] also found the performance grade of asphalt binder can be expanded by addition of Sasobit®, with the upper temperature increased by up to 13.5°C and lower temperature increased by up to 6.9°C for 3% Sasobit® modification. In addition, resistance to oxidative aging was observed for Sasobit® modified WMA [Banerjee et al. 2012].

In spite of a large body of research on Sasobit® modified binders, only limited systematic studies on the correlation between structure and viscoelasticity have been performed. Recently, Polacco and coworkers performed a comprehensive study regarding the effect of different types of waxes on asphalt morphology, residual crystallinity, and mechanical properties [Polacco et al. 2012]. A gel structure of asphalt blends with Fischer-Tropsch (FT) type waxes was suggested based on the observation of a high penetration index and low values of phase angle (55° – 65°).

The focus of the present study is to evaluate the effect of increased wax (Sasobit®) concentration on asphalt binder structure and rheology, and glass transition temperatures and whether time-temperature superposition (TTS) holds. The study involves four unaged binders with varying Sasobit® concentrations. One of the four asphalts was polymer modified.

Background - Warm Mix Asphalt (WMA)

Strong interest in WMA in North America dates back to a presentation at the January 2003 annual meeting of the National Asphalt Pavement Association (NAPA) [Kuennen 2004]. However since the mid to late 1990‘s, WMA has been a major topic of interest in Europe and South Africa.

The key to the production of hot mix is providing sufficient temperature to reduce the viscosity of the asphalt to adequately coat the aggregate and enable compaction of loose mix. WMA achieves adequate coating by reducing the asphalt viscosity in a number of innovative ways, and a number of WMA additives and processes have been developed over the last 15 years. A 2002 scan tour of Europe by representatives of Federal Highway Administration (FHWA), National Asphalt Pavement Association (NAPA), Asphalt Institute (AI), and contractors and consultants identified four WMA technologies: Aspha-Min, Sasobit®, Asphaltan-B, and WAM-Foam. A follow up WMA scan tour of Europe in 2007 identified at least 12 technologies [Prowell 2007]. Sasobit® was one of the 12 technologies/additives identified.

The number and type of WMA additives commercially available is constantly changing as new additives come on the market and others drop out. The latest preapproved list for WMA additives by the Texas Department of Transportation [2013] has 16 additives, one of which is Sasobit®.

EXPERIMENTAL

Materials

Asphalt Binders

Four asphalt binders, listed in table 1, with different types and grades, were used as control binders in this study. Among the four binders, MN1-3, MN1-4, and MB are PG 58-28 unmodified and from different crude sources. YNP is a styrene-butadiene-styrene (SBS) PG 58-34 modified binder.

Table 1. Sasobit® modified asphalt binders.

Asphalt Binder	PG grade	Description
MB	PG 58-28	Unmodified binder from Manitoba, Canada, 150/200 pen, Canadian blend. Manitoba comparative pavement section R1.
MN1-3	PG 58-28	Unmodified binder from Minnesota, Canadian blend, Minnesota comparative pavement section 1-3
MN1-4	PG 58-28	Unmodified binder from Minnesota, Blend: Arab heavy, Arab medium, and Kirkuk. Minnesota comparative pavement section 1-4.
YNP	PG 58-34	SBS modified binder from Yellow Stone National Park. YNP project constructed 2007.

One and 3 percent Sasobit® loading concentrations (by weight) were selected for this study because 3% is typically the maximum loading concentration, above which the low temperature performance of Sasobit® modification may be negatively affected [Hurley and Prowell 2005a&b; Butz et al. 2001]. Sasobit®, which is manufactured by Sasol, recommends a Sasobit® concentration close to 0.8%, but no more than 3%.

The Sasobit® modified asphalts binders were prepared by heating the asphalt to 150°C and adding Sasobit® pellets with gentle stirring. The modified asphalt was kept at 150°C for approximately an hour with occasional stirring.

Sasobit®

The Sasobit® used in this study was manufactured by SasolWax, South Africa, and acquired on a HMA/WMA project in Yellowstone National Park in 2007.

Test Methods

Nuclear Magnetic Resonance (NMR)

Solid-state ^{13}C NMR measurements were made on an NMR solids spectrometer using cross polarization and magic-angle spinning to characterize the molecular structure of Sasobit® in asphalt.

Differential Scanning Calorimetry (DSC)

DSC measurements were performed on a TA Instruments Q2000 Differential Scanning Calorimeter. Samples of the asphalt of 3-8 mg were weighed into covered and crimp-sealed aluminum pans. The method employed for DSC experiments, unless otherwise specified, used both standard and modulated elements:

Standard

1. Equilibrate at 165.00°C
2. Isothermal for 5.00 min
3. Ramp from 165.00°C to -90.00°C at 5.00°C/min
4. Isothermal for 5.00 min
5. Ramp to 165.00°C at 10.00°C/min

Modulated

6. Modulate +/- 0.50°C every 80 seconds
7. Isothermal for 5.00 min
8. Ramp to -90.00°C at 2.00°C/min
9. Isothermal for 5.00 min
10. Ramp to 165.00°C at 2.00°C/min

The data from the standard portion of the method are used to calculate crystallization onset temperatures and crystallization and melting energies. The modulated portion of the method provides information on the glass transition, including onset, endpoint, height, width, and temperature.

Dynamic Shear Rheometry (DSR)

Rheology was performed on a Malvern Kinexus stress-control rheometer, and in a few cases on a TA Instruments ARES strain-control rheometer. The low and intermediate temperature (-30 to 30°C) rheological properties of the recovered binders were measured with 4 mm diameter parallel plate geometry DSR (typically referred to as simply 4-mm DSR). High temperature rheological properties were measured with 25 mm diameter plate geometry.

The 4-mm DSR test method is described elsewhere by Sui et al. [2010]. The method corrects for machine compliance and allows testing at temperatures as low as -40°C. Low and intermediate temperature frequency sweeps were typically performed at 20°C intervals over a temperature range of -30 to 30°C and an angular frequency range of 0.1 to 50 rad/sec. High temperature frequency sweeps were typically performed at 50 and 70°C. The samples were annealed at 60 °C for about 10 minutes before loading onto the rheometer with loading temperature of 60°C. The gap and normal force were properly adjusted at test temperatures other than 60°C.

RESULTS AND DISCUSSIONS

Sasobit® ^{13}C NMR Spectra

The ^{13}C NMR spectra of the Sasobit wax is presented in figure 1. The data are consistent with referring to the Sasobit® as a wax in that the major resonance at ~33.6 ppm is narrow, characteristic of microcrystalline methylene carbons (CH_2) in long chains [Michon et al. 1999]. Resonances due to terminal methyl groups on the hydrocarbon chain are at ~15.2 ppm. The difference in chemical shifts between the methylene carbons and the terminal methyl carbons can be used to determine the crystal nature of long-chain waxes [Vanderhart 1981]. For Sasobit®, this difference is ~18.4 ppm, which is characteristic of a monoclinic crystalline environment.

The average carbon chain length in Sasobit® can be obtained from the ^{13}C NMR spectrum [Reynhardt 1985]. By taking the ratio of the intensity of the methylenes (CH_2) to the methyl (CH_3) groups, an estimate of the average carbon chain (*m*) length can be obtained. The formula is:

$$m = 2\left(\frac{I_{CH\,2}}{I_{CH\,3}} + 1\right) \qquad (1)$$

where $I_{CH\,2}$ and $I_{CH\,3}$ are the intensities of the methylene and methyl groups, respectively. For Sasobit®, an average chain length of 78 carbons was calculated using a pulse delay of 10 s to acquire the spectrum.

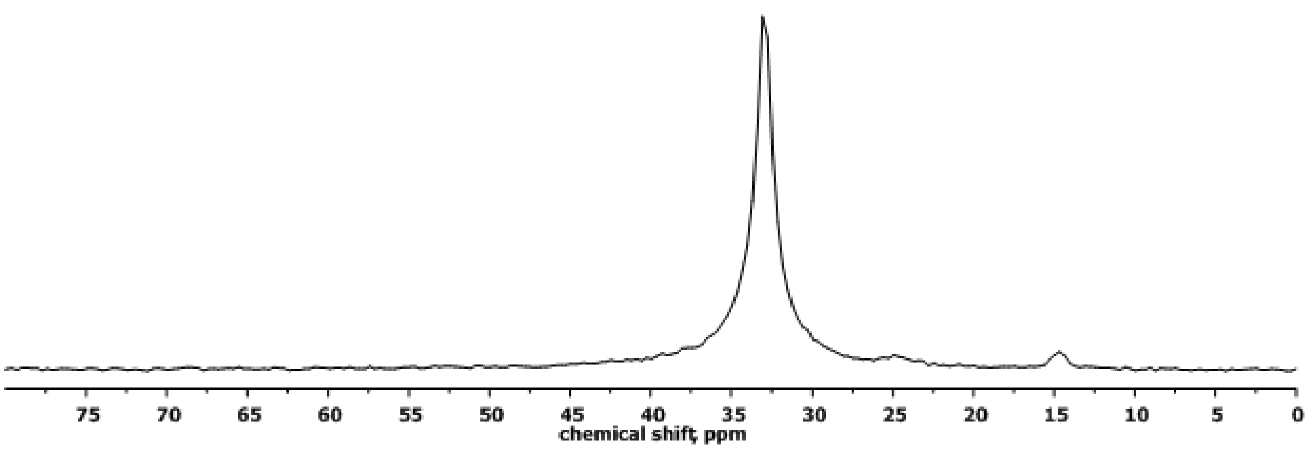

Figure 1. Cross polarization, magic-angle spinning (CP/MAS) ^{13}C NMR spectra of Sasobit® warm mix additive illustrating the long chain aliphatic character of the Sasobit®.

Glass Transition Temperature

Modulated differential scanning calorimetry (MDSC) measurements were made on the four study asphalts doped with 0, 1, and 3 weight percent Sasobit®.

Changes in the glass transition onset, end, and inflection (Tg) temperatures are shown in figures 2 through 5.

Negative effects that might be expected from addition of materials to asphalts would be increases in the onset, end, or Tg. In only one case, MB (figure 5), is there an increase in one of the values, but this is less than two degrees Celsius. The end temperature decreases in all cases from two to eight degrees Celsius. This suggests that structuring due to Sasobit® may not be a major factor in terms of the transition from a more viscous state to a more stiff relatively brittle state. Furthermore, the ability of the asphalts at low temperature to relax is probably unaffected or possibly improved.

It's important to keep in mind the above comments are solely in regard to the addition of Sasobit. The research on "waxy" asphalts where the wax is naturally occurring has been shown by some to be detrimental in terms of physical hardening. For example, Anderson and Marasteanu [1999] concluded that physical hardening is caused by the formation of crystalline wax in addition to free volume collapse, both above and below the glass transition temperature.

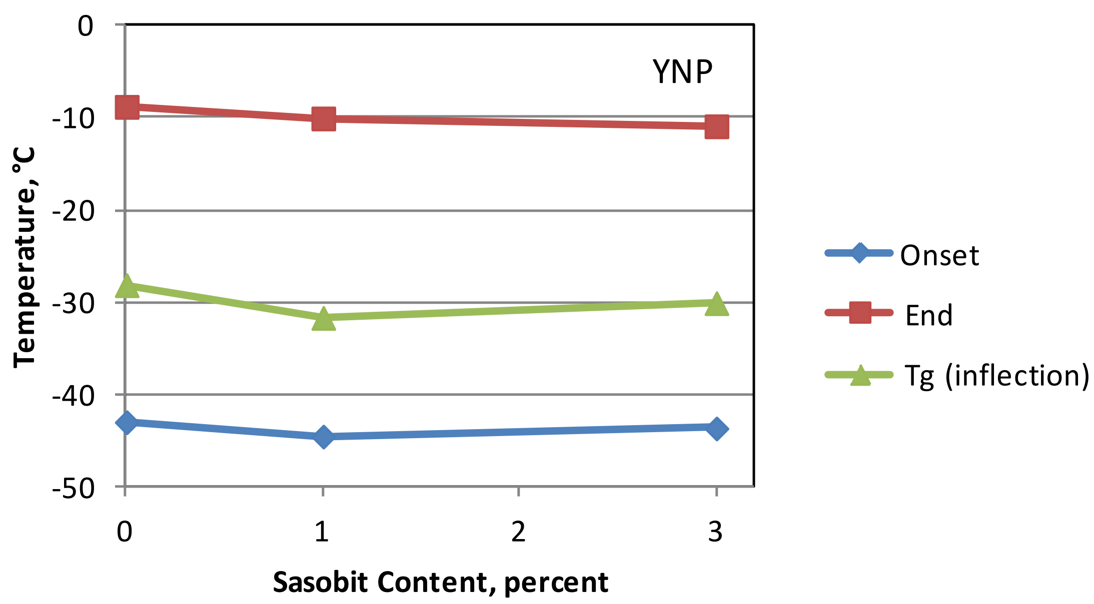

Figure 2. Changes in YNP glass transition characteristics with addition of Sasobit®.

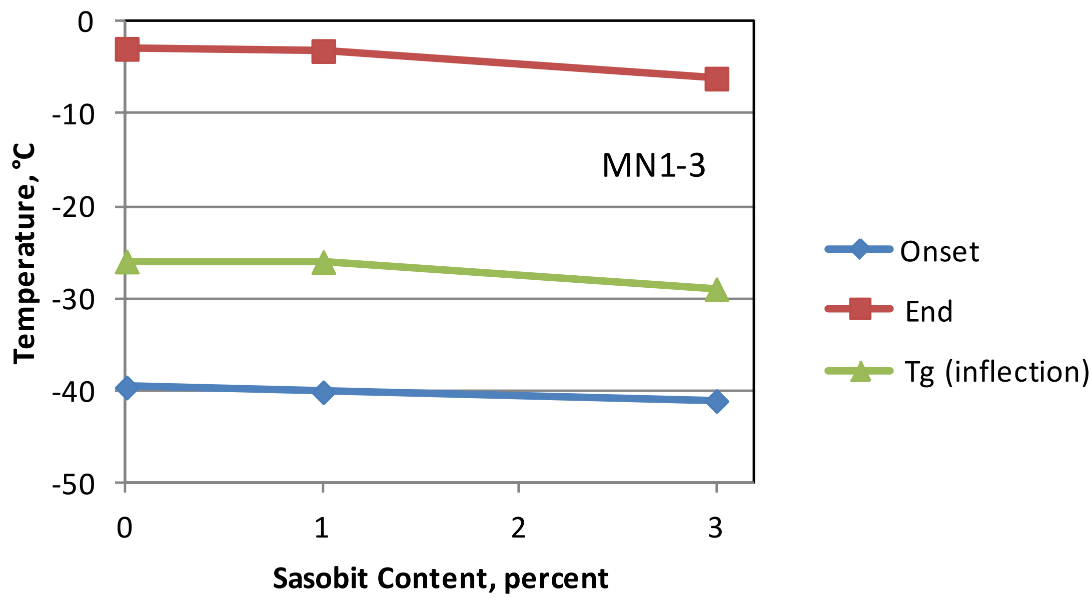

Figure 3. Changes in MN1-3 glass transition characteristics with addition of Sasobit®.

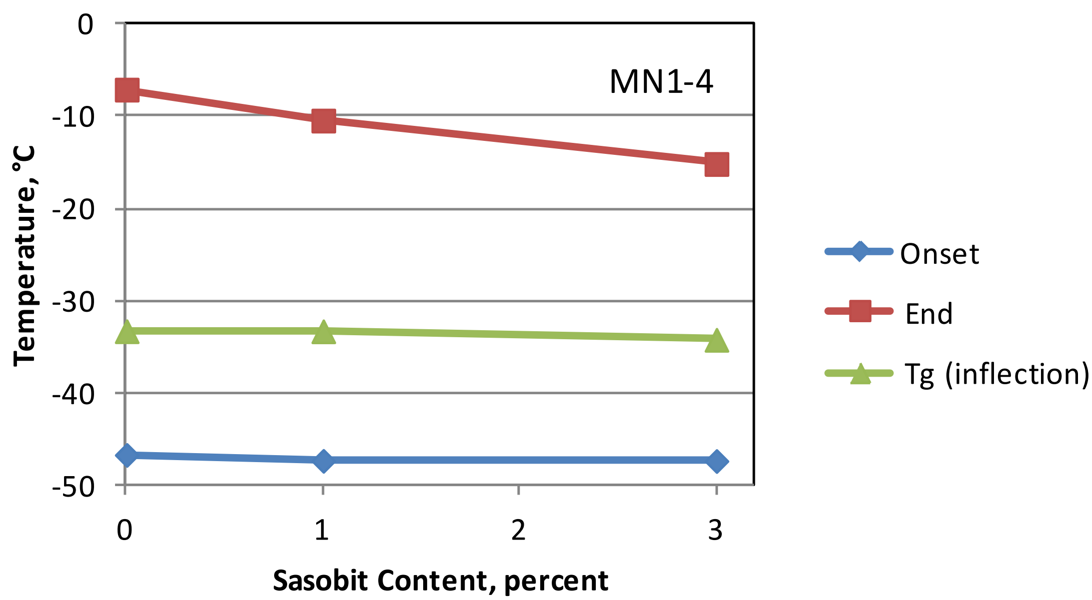

Figure 4. Changes in MN1-4 glass transition characteristics with addition of Sasobit®.

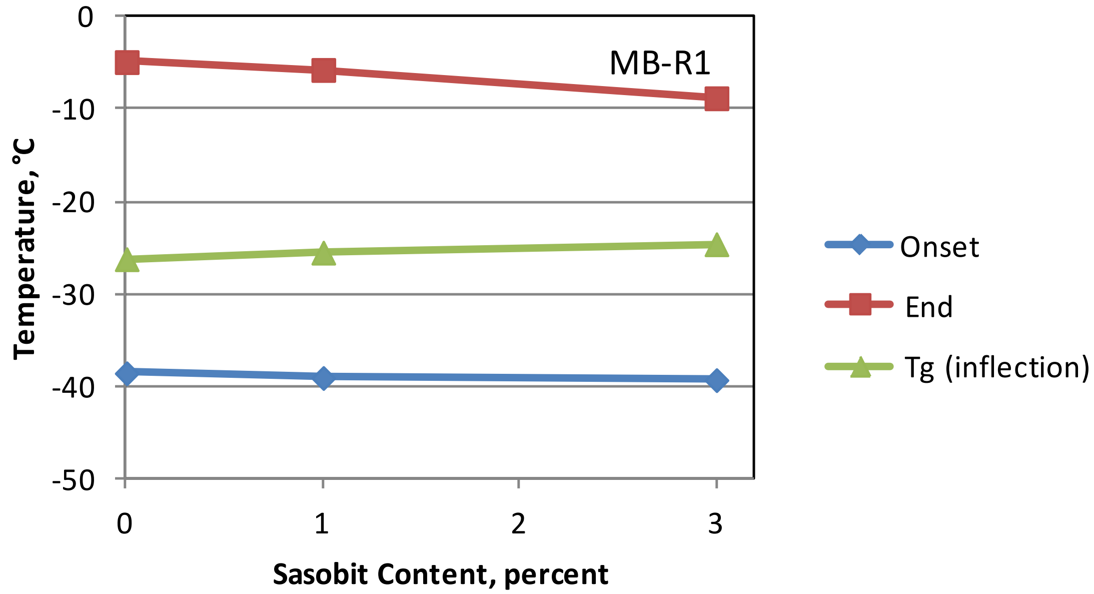

Figure 5. Changes in MB-R1 glass transition characteristics with addition of Sasobit®**.**

Rheology

Time Temperature Superposition(TTS) and Thermorheological Simplicity

To determine if TTS holds for Sasobit® modified asphalts, black space plots were prepared using the frequency sweep data. A black space plot depicts the phase angle versus the corresponding absolute value of the complex shear modulus from the dynamic rheological data. It is also known as the van Gurp–Palmen plot [van Gurp and Palmen 1998]. The plot is typically used to measure of the validity of time-temperature superposition (TTS) and thermorheological simplicity. The black space plot is one of a family of similar plots such as the well known Cole-Cole, Han, and Wicket plots which are independent of reduced frequency and hence temperature.

Figures 6 through 9 are black space plots for the four base binders in this study and include data for 1% and 3% Sasobit® modified binders.

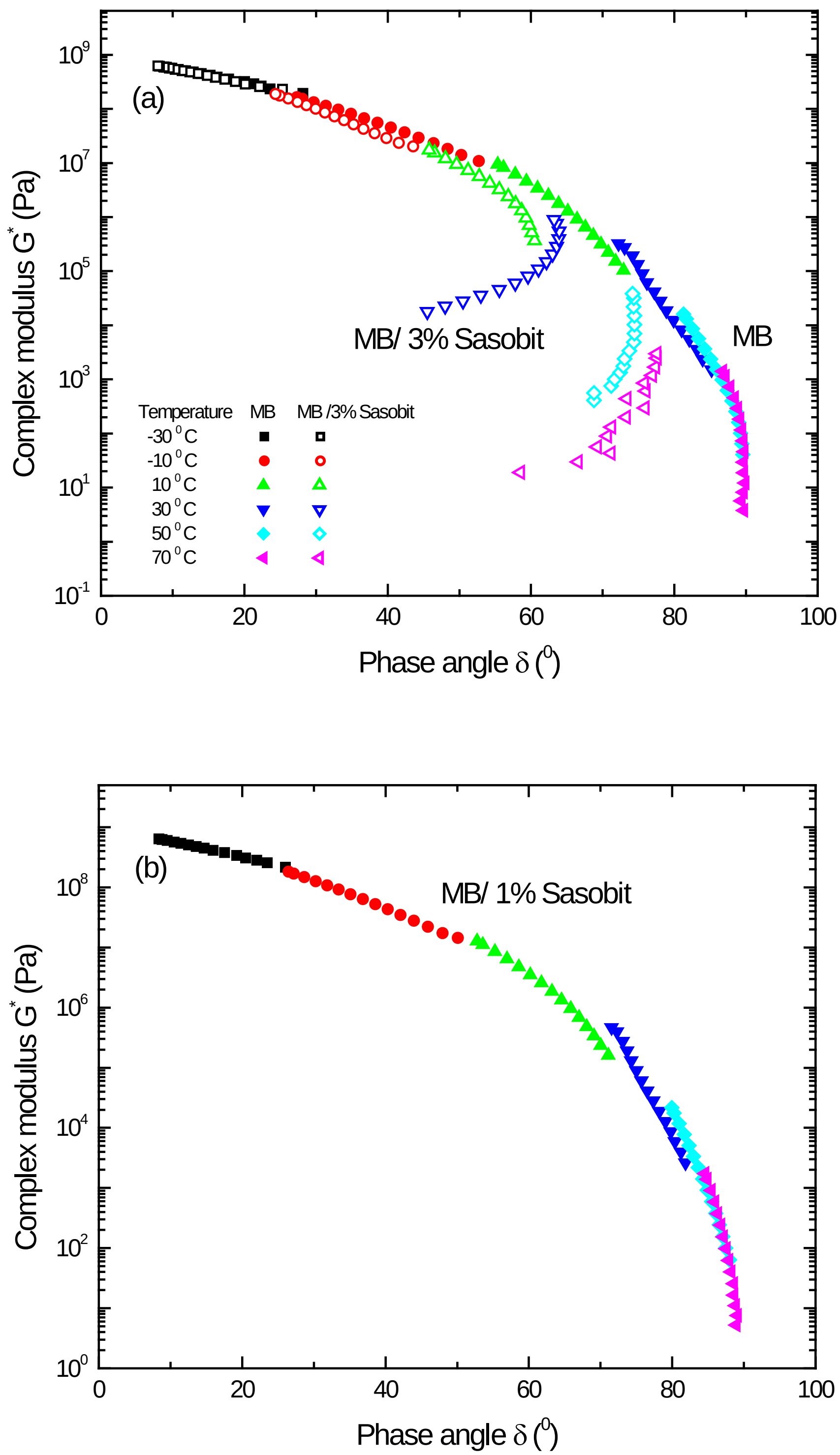

Figure 6. Black space diagrams of (a) MB binder and MB/3% Sasobit®; (b) MB/1% Sasobit®. The frequency sweep temperatures shown in figure (a) also apply to figure (b).

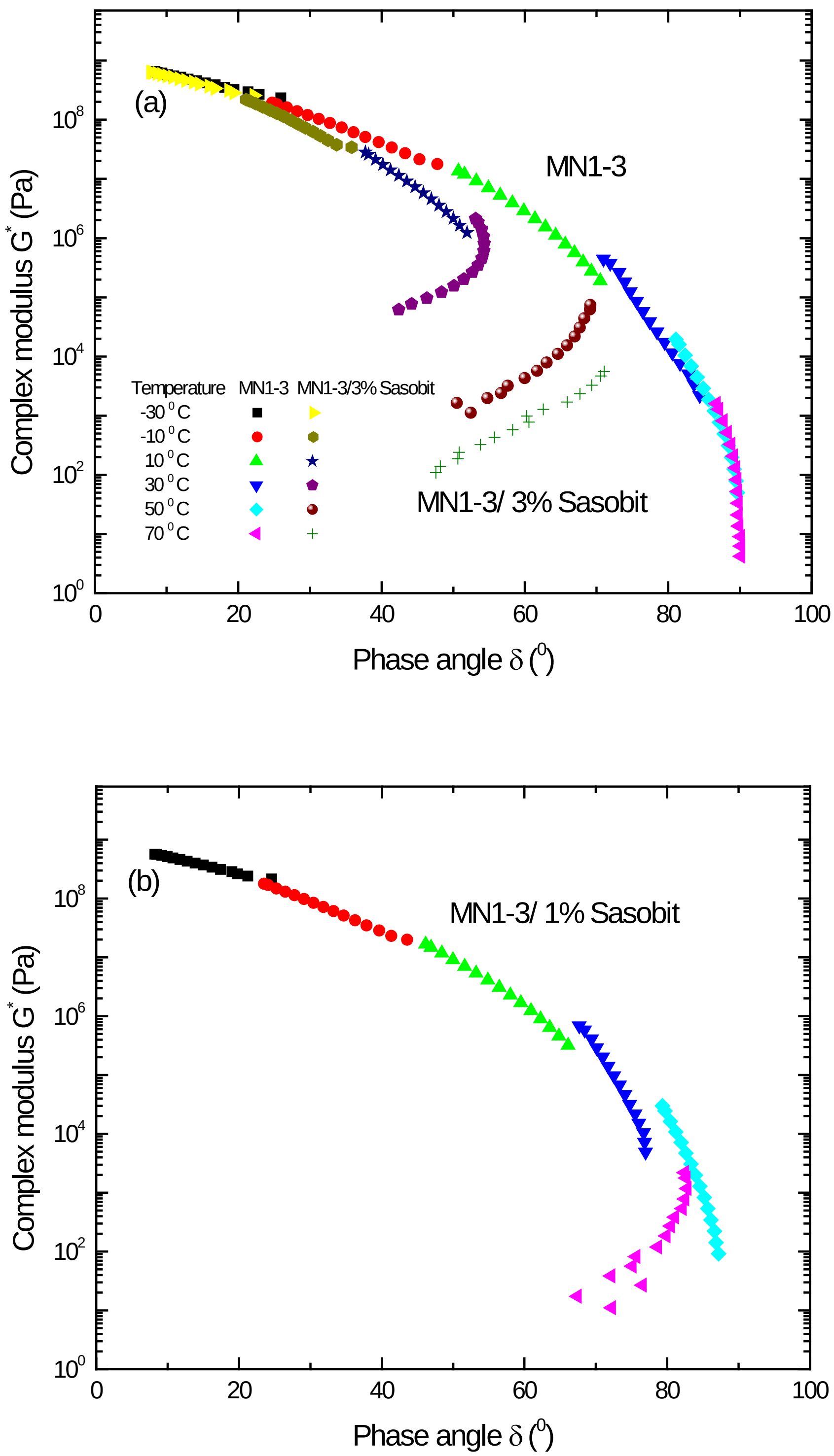

Figure 7. Black space diagrams of (a) MN1-3 binder and MN1-3/3% Sasobit®; (b) MN1-3/1% Sasobit®.

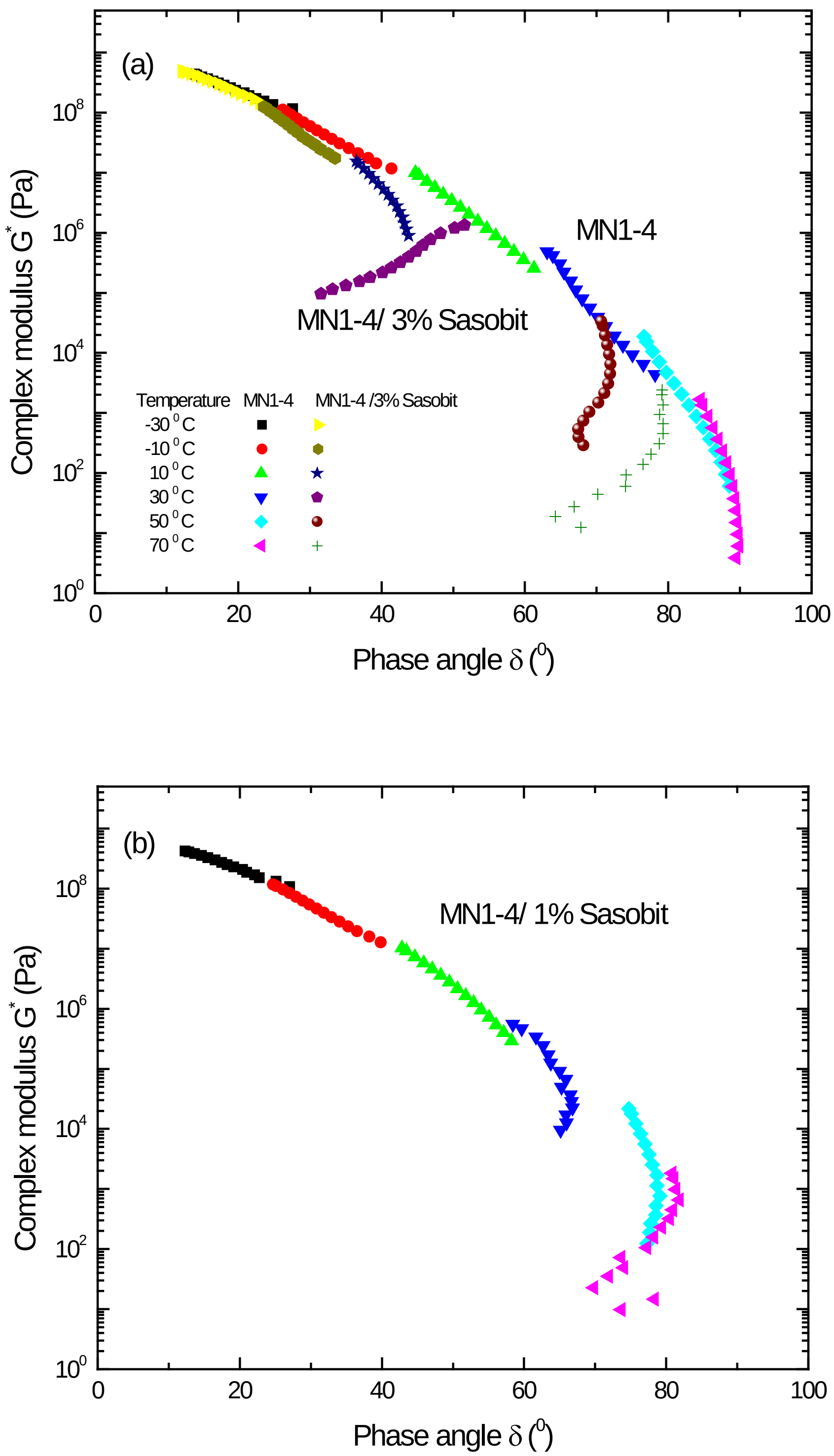

Figure 8. Black space diagrams of (a) MN1-4 binder and MN1-4/3% Sasobit®; (b) MN1-4/1% Sasobit®.

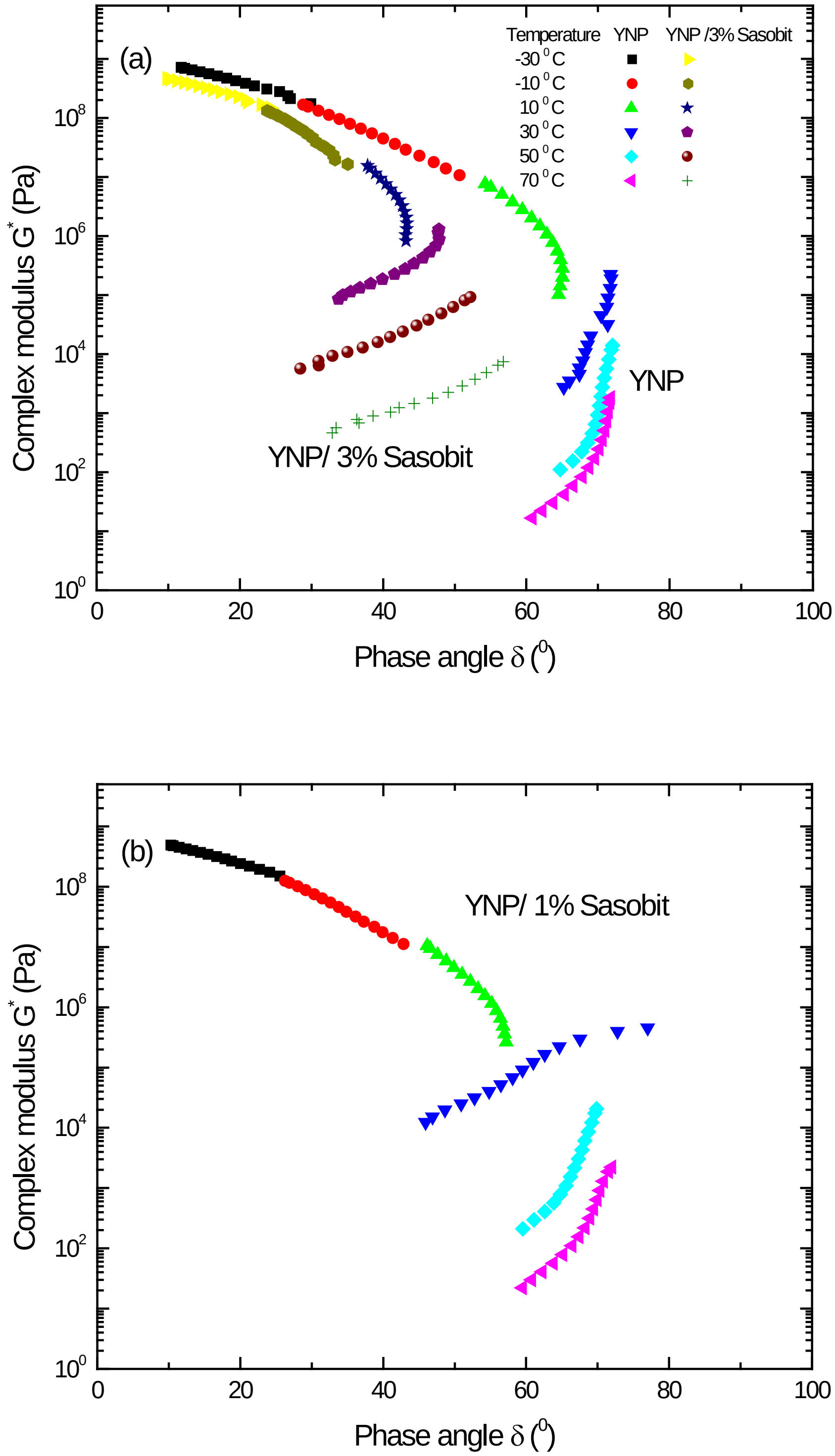

Figure 9. Black space diagrams of (a) YNP binder and YNP/3% Sasobit®; (b) YNP/1% Sasobit®.

Except for YNP, which is a SBS modified binder, single curves at all frequencies and temperatures of interest are obtained for the base binders (MB, MN1-3 and MN1-4). The single curves presented in the black space plots indicate time-temperature superposition (TTS) holds reasonably well. The deviation from TTS for YNP at high temperature, i.e., above 30°C as shown in figure 9, is attributed to the presence of SBS in the asphalt binder matrix, which not only changes the absolute value of the relaxation times, but also alters the relaxation function of the binder matrix.

Upon the additon of 3% Sasobit®, the black space plots of Sasobit® modified binders lose their single curve characteristics. TTS appears to be lost at temperatures above 30°C for 3% Sasobit®. The deviation from TTS is speculated to be from microstructural changes caused by the incorporation of Sasobit® into asphalt binders leading to different stress relaxation functions within the binder. This concept is discussed in more detail later in this report.

Figures 6 through 9 indicate TTS holds for 1% Sasobit® blends at higher temperature than 3% blends. For the 1% Sasobit®/MB blend, the TTS holds for the entire temperature range investigated. That observation is difficult to explain based entirely on rheological analysis, and additional investigation in terms of the microstructure and morphology of Sasobit® modified asphalt binders is suggested.

Rheological Properties: Low Temperature

The complex shear modulus and complex viscosity at -30°C (frequency sweep) of the base binders and their corresponding blends with Sasobit® are shown in figures 10 through 13.

With the addition of up to 3% Sasobit®, no signficant changes in binder stiffness in terms of $G^*(\omega)$ or $\eta^*(\omega)$ are observed. It appears that the low temperature viscoelasticity of Sasobit® modified MB, MN1-3 and MN1-4 is dominated by the asphalt binder matrix at Sasobit® concentrations less than 3%. Three percent Sasobit® is not sufficient to exert a significant low temperature rheological impact, at least for the binders considered in this study. Further, it is observed the YNP Sasobit® blends actually become softer at -30°C as shown in figure 13. The exact reason for the softening is not well understood and needs further investigation, especially in terms of possible structural changes or associations between the Sasobit® and SBS present in YNP and the fact that the DSC did not indicate any significant changes in Tg with the addition of Sasobit.

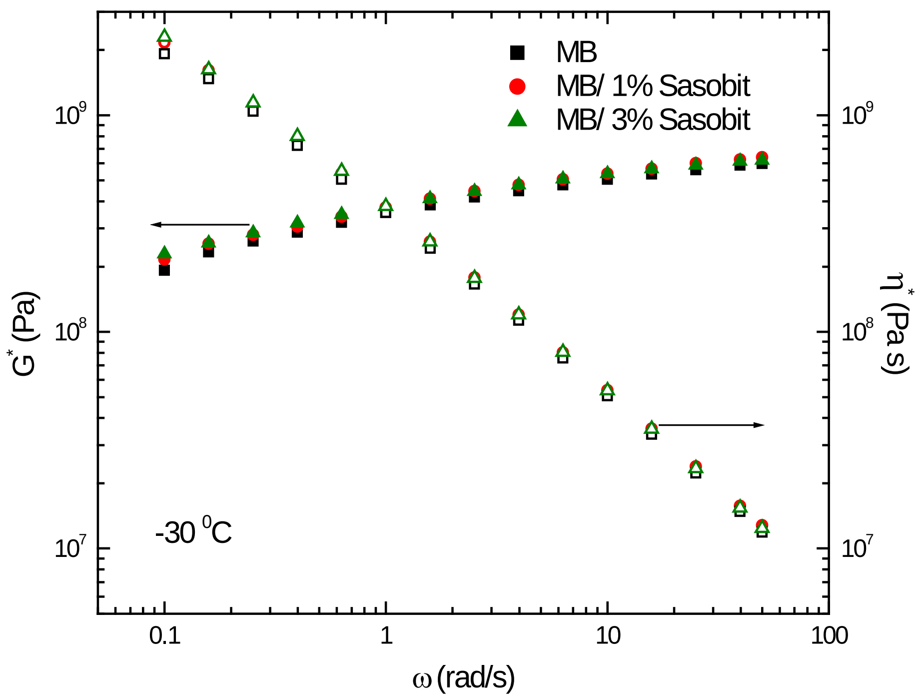

Figure 10. Complex shear modulus and viscosity at -30°C for MB and Sasobit® modified MB binders.

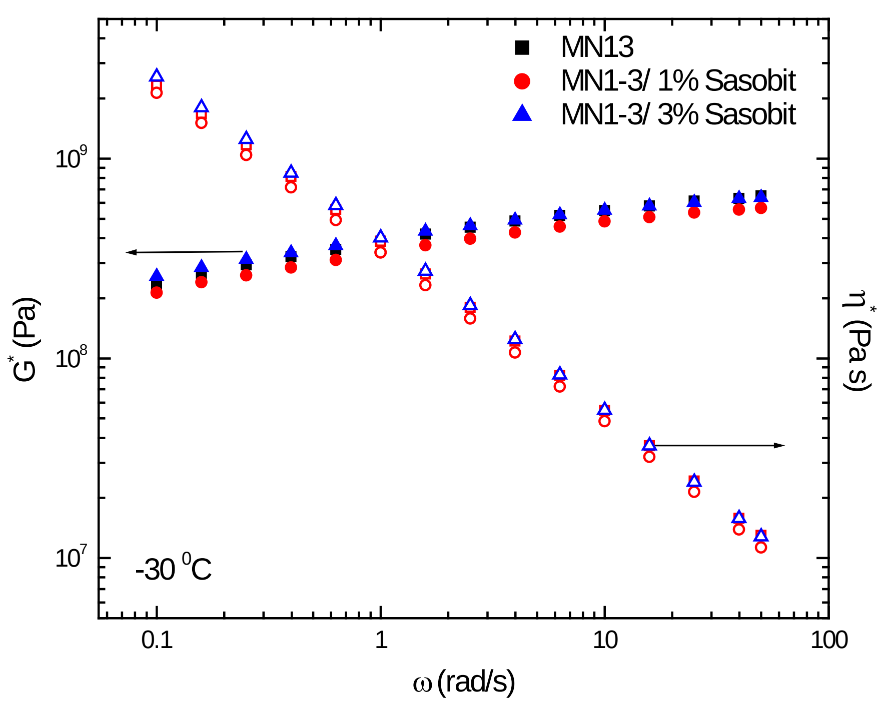

Figure 11. Complex shear modulus and viscosity at -30°C for MN1-3 and Sasobit® modified MN1-3 binders.

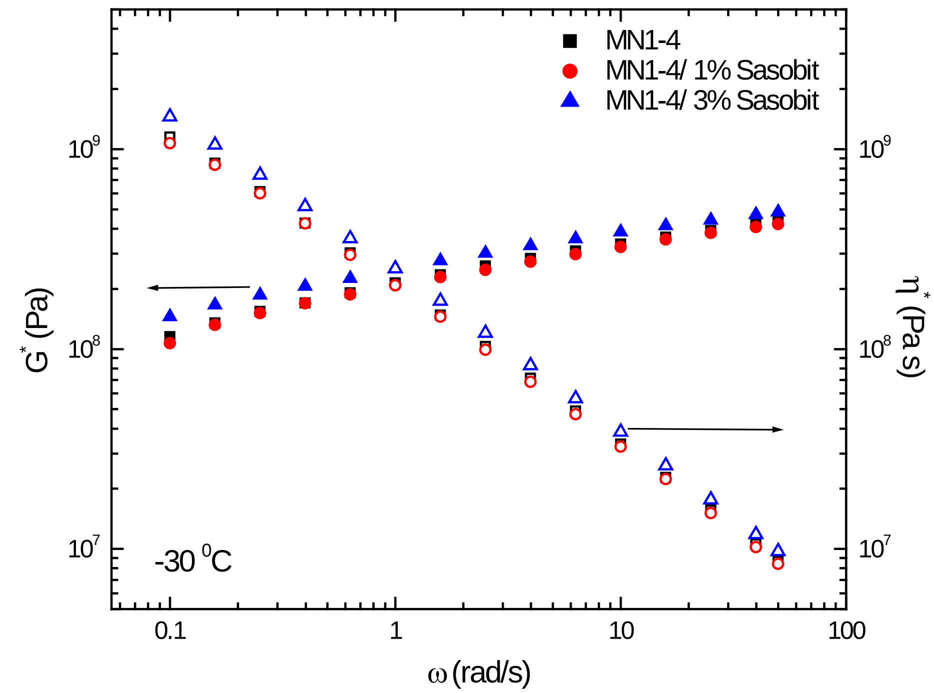

Figure 12. Complex shear modulus and viscosity at -30°C for MN1-4 and Sasobit® modified MN1-4 binders.

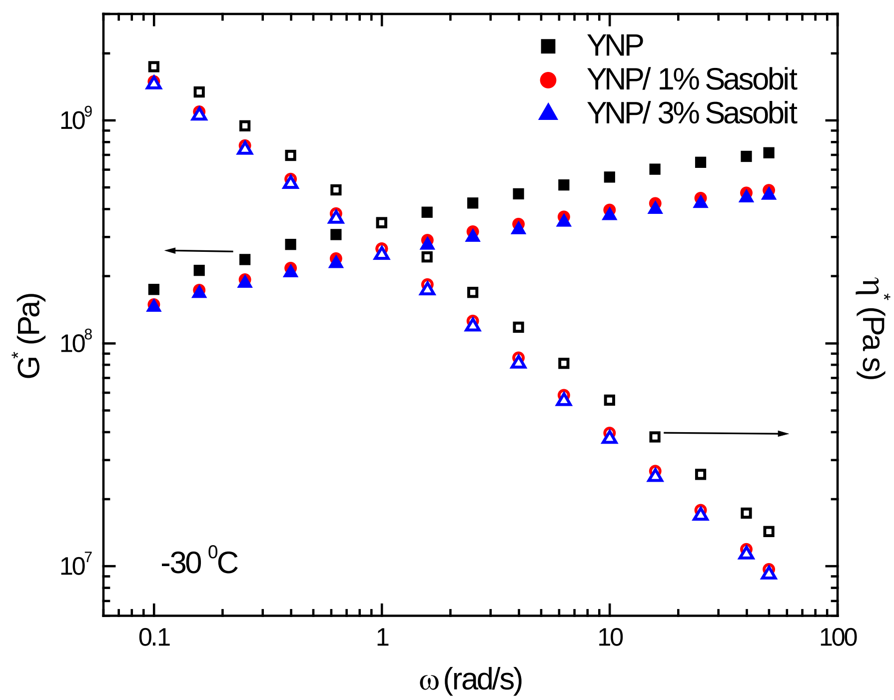

Figure 13. Complex shear modulus and viscosity at -30°C for YNP and Sasobit® modified YNP binders.

Figure 14 shows the column plots of “limiting low temperaures” for base binders and Sasobit® modified binders. These results were estimated based on the criteria of creep stiffness (S value) and creep rate (m value). It should be mentioned that all the S and m values were calculated from low temperature 4 mm DSR results rather than BBR according to [Sui et al. 2011]. The “limiting low temperature” of base binders appear lower than the corresponding PG grade described in table 1. That is because these binders did’t undergo the standard rolling thin film oven (RTFO) and pressure aging vessel (PAV) oxidative aging processes. The figure 14 “limiting low temperatures” are used to consider the effect of the Sasobit® on low temperature performance. Consistent with our findings in figure 1-13, there is no significant increase in limiting low temperature. The slight increase in “low limiting temperature” of 3% Sasobit/YNP binder was found to be due to its slow relaxation even though the sample exhibits smaller creep stiffness at low temperatures.

Overall, the DSC and DSR results indicate that Sasobit® (with addition of no greater than 3% by weight) does not negatively influence the low temperature physical properties of the binders investigated.

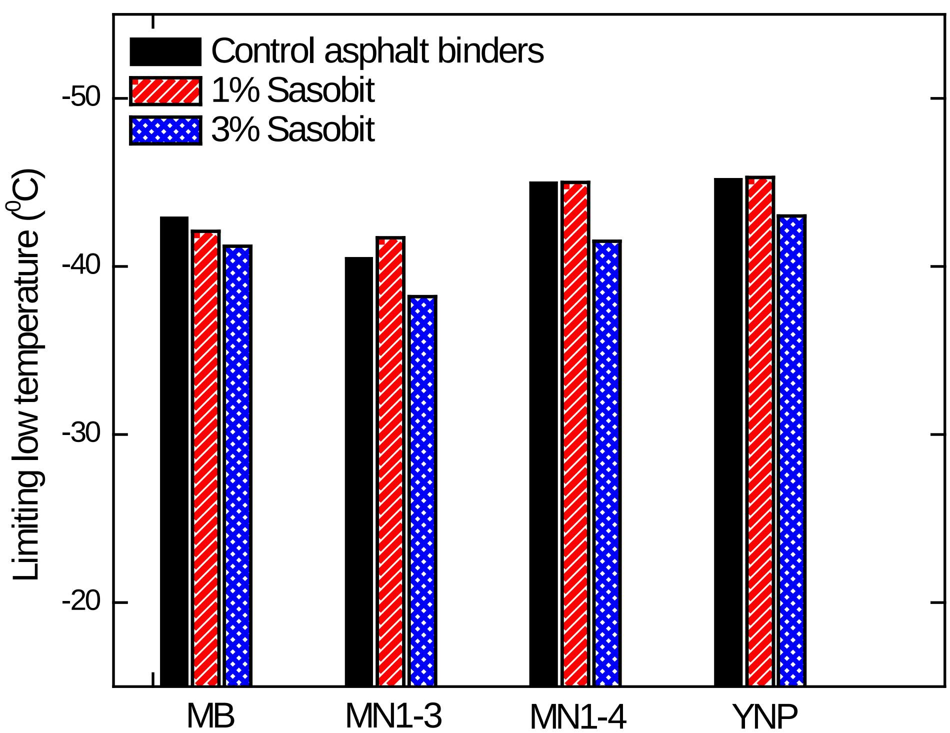

Figure 14. The “limiting low temperature” or continuous grade temperature of base binders and Sasobit® modified binders based on the critiria of creep stiffness S=300 MPa and creep rate m= -0.3.

Rheological Properties: Intermediate Temperature

Among the four binders investigated, MN1-4 exhibits the most rigid response at intermediate temperature (10°C) as implied by its high zero shear viscosity and complex modulus relative to the other three asphalts (see figure 15). MB, MN1-3 and YNP appear to have similar rheological properties at 10°C.

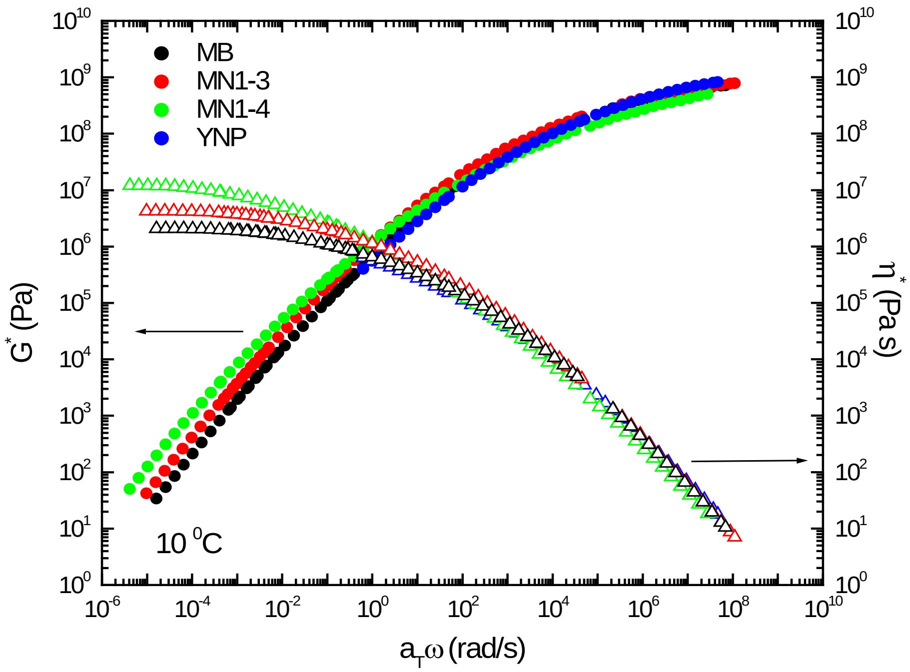

Figure 15. Dynamic rheological properties of MB, MN1-3, MN1-4 and YNP binders at 10°C. Note: $a_T\omega$ refers to reduced frequency, and the closed symbols refer to G* and the open symbols refer to η*.

The complex shear modulus and complex viscosity at 10°C (frequency sweep) of the base binders and their corresponding blends with Sasobit® are shown in figures 16 through 19.

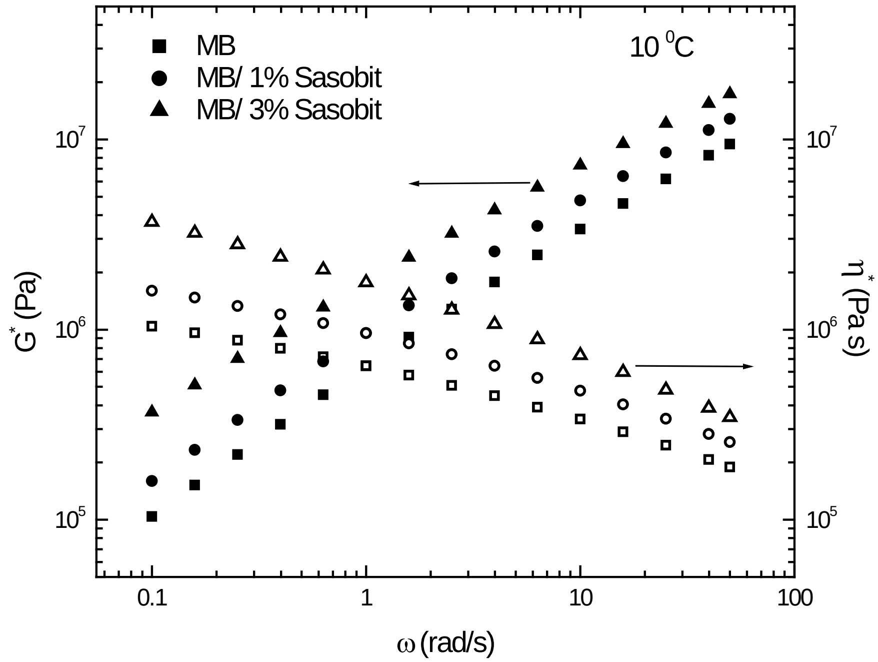

Figure 16. Complex shear modulus and viscosity at 10°C for MB and Sasobit® modified MB binders. Note: The closed symbols refer to G* and the open symbols refer to η*.

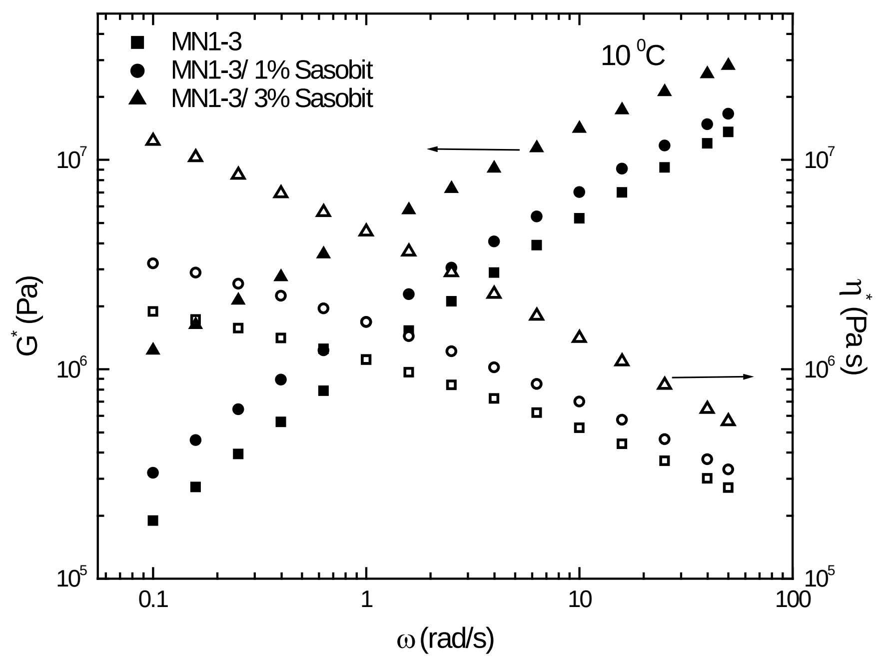

Figure 17. Complex shear modulus and viscosity at 10°C for MN1-3 and Sasobit® modified MN1-3 binders. Note: The closed symbols refer to G* and the open symbols refer to η*.

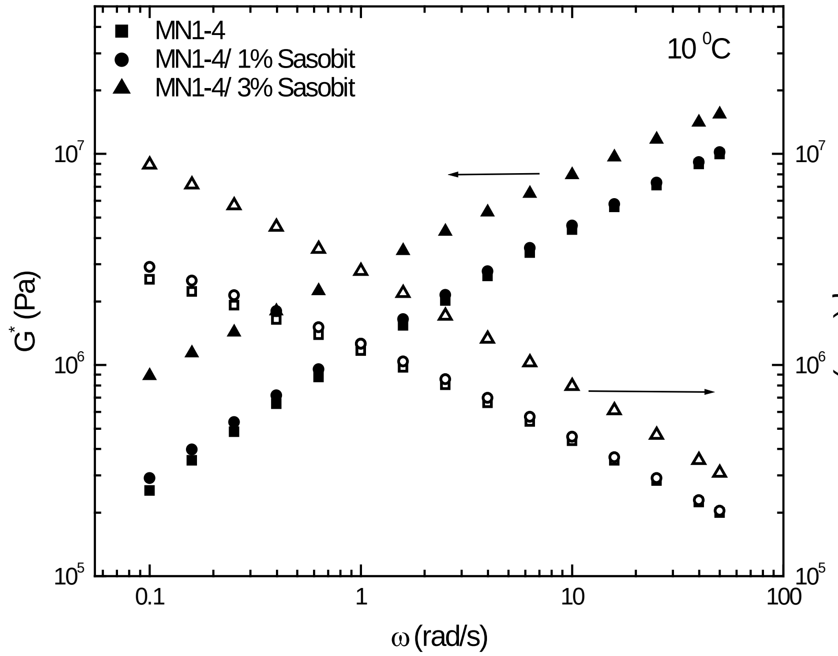

Figure 18. Complex shear modulus and viscosity at 10°C for MN1-4 and Sasobit® modified MN1-4 binders.

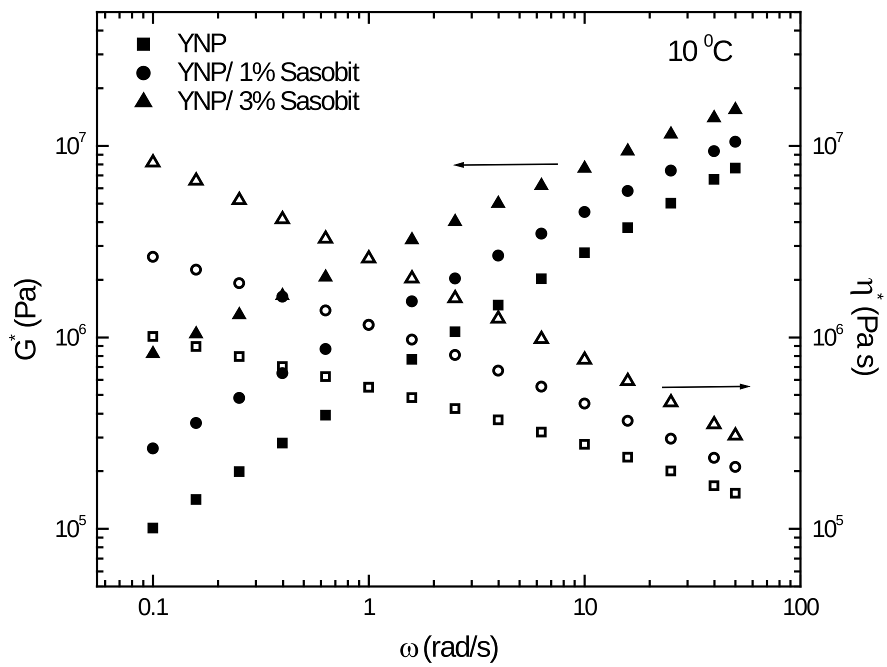

Figure 19. Complex shear modulus and viscosity at 10°C for YNP and Sasobit® modified YNP binders.

Both the complex modulus and complex viscosity increase by 4 - 5 times at low freqencies for the MB, MN1-3, MN1-4 binders with 3% Sasobit® at 10°C. For the YNP 3% Sasobit® blend binder, the modulus and viscosity increase even more dramatically as shown in figure 19. At this intermediate temperature, not only the interaction between Sasobit® crystals, but also the interaction between Sasobit® and thermoplastic elastomer SBS is expected to play an important role in reinforcing the binders, but not necessarily causing brittleness which would lead to more fatigue cracking. This is an interesting and complex phenomena and deserves further research. It appears there is a synergistic effect between the SBS and Sasobit that stiffens the binder at high temperature and yet makes the binder softer at low temperature.

Rheological Properties: High Temperature

Figure 20 compares the high temperature performance grade where G*/sin δ = 1.0 kPa. There is an approximate linear increase in the continuous grade temperature for two of the asphalts (MB MN1-4) with the addition of Sasobit® and a non-linear increase for the other two (MN1-3 and YNP).

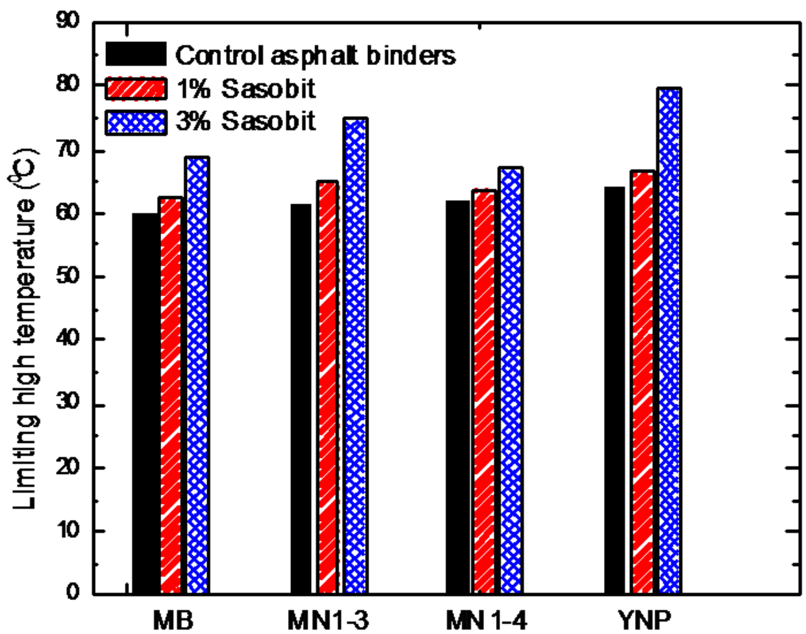

Figure 20. The continuous grade temperatures corresponding to G*/sin δ = 1.0 kPa at frequency of 10 rad/s.

To further evaluate the trends in continous grade, the rheological properties at 50°C were investigated for all four base binders and their 1 and 3% blends. Figure 21 shows the complex storage and loss moduli of MB base asphalt and MB Sasobit® blends.

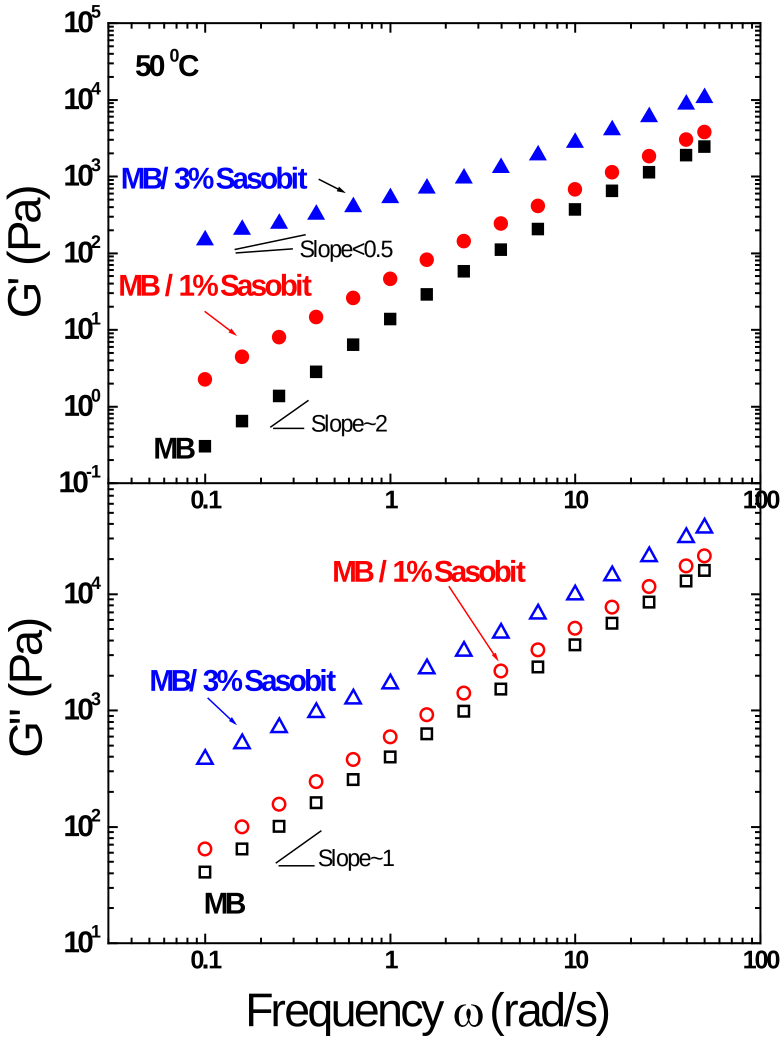

Figure 21. Storage and loss moduli of MB and MB Sasobit® blends at 50°C.

At 50°C, which is far above the glass transition temperature, the MB base asphalt is well below the point where G' and G" cross in terms of frequency. In general, asphalt binder dynamic moduli at frequencies below the crossover frequency display viscous dominated behavior and above the crossover frequency elasticity dominates. Addition of Sasobit®, especially 3% Sasobit® has a dramatic effect on the complex viscosity at a test temperature of 50°C as shown in figure 22. The MB 3% Sasobit® blend exhibits a pseudo-solid or semi-solid behavior and the complex viscosity starts to level off at low frequencies with a slope of less than 0.5 for storage modulus versus frequency in the logarithm plot. The behavior suggests the formation of network structure or certain associations in the modified binders leading to the observed semi-solid rheological behavior at 50°C. In addition, after modification with 3% Sasobit® the modified YNP binder exhibits a significant increase in viscosity compared to other binders. That nearly diverging viscosity is attributed not only to the effect of the Sasobit®, but also to the possible associations between Sasobit® and SBS thermoplastic elastomer.

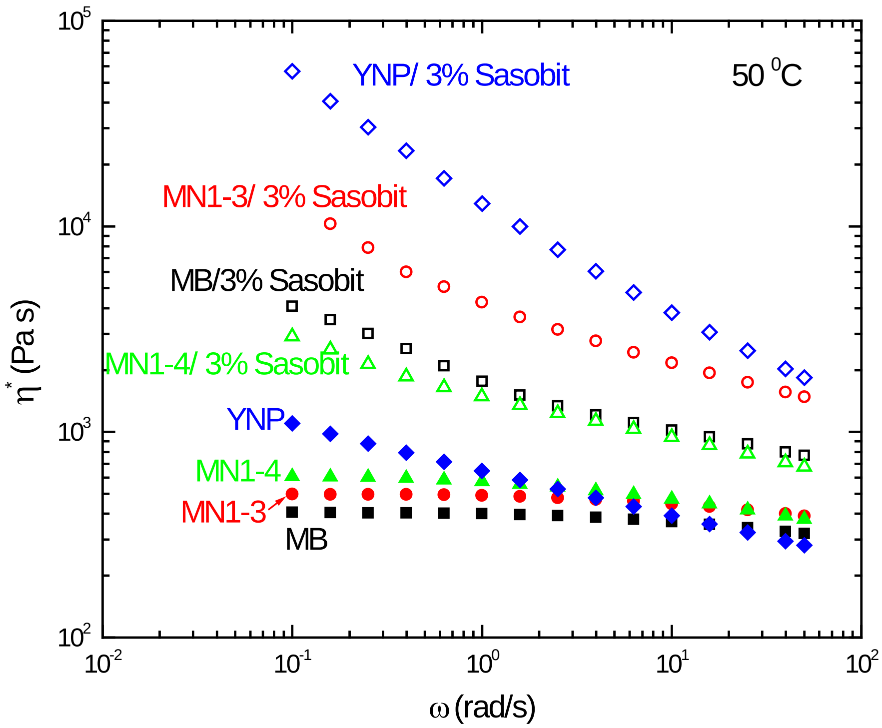

Figure 22. Complex viscosity at 50°C for MB, MN1-3, MN1-4, YNP base asphalts and 3% Sasobit® asphalt blends.

CONCLUSIONS

The average carbon chain length in Sasobit® determined by ^{13}C NMR was 78 carbons.

The glass transition temperature was largely unaffected by the presence of Sasobit®, even at a concentration of 3%.

Time temperature superposition (TTS) appears to be lost at temperatures above 30°C and 3% Sasobit®. At 1% concentration TTS is also lost, but this occurs at significantly higher temperatures.

Overall, the low temperature DSR results tend to reinforce the DSC results and indicate that Sasobit® (with addition of no greater than 3% by weight) does not negatively influence the low temperataure rheological behavior of the binders investigated.

There was an apparent synergistic beneficial softening at low temperature of Sasobit® and SBS modified asphalts which could allow the material to more easily relax and result in less thermal cracking.

At high temperature the formation of network structure or certain associations in the Sasobit® blends suggest a semi-solid rheological behavior which should provide improved resistance to mechanical deformation (rutting).

RECOMMENDATIONS

In the present study the effect of increasing Sasobit® concentration is confined to unaged asphalt binders. The study should be enlarged and include the same asphalt binders but include RTFO and PAV oxidative aging.

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