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Chinese Society of Pavement Engineering

International Journal of Pavement Research and Technology 11 (2018) 838-845

http://www.ijprt.org.tw/

Physical and rheological properties of modified sulfur asphalt binder

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Received 5 December 2016; received in revised form 12 June 2018; accepted 30 July 2018

Abstract

In this study, asphalt binder was partially substituted with different percentages of modified sulfur (20%, 30%, and 40 wt%) to give 20/80, 30/70, and 40/60 sulfur asphalt (S/A) mixtures. The physical properties including softening point, viscosity, penetration, and ductility were examined to characterize the consistency of S/A mixtures. Characterizing the rheological properties of modified asphalt binders is also highly recommended for the prediction of the major pavement damages such as rutting, and cracking. The laboratory studies were conducted to examine the viscoelastic properties of modified sulfur asphalt binder using dynamic mechanical analyzer (DMA), and Brookfield rotational viscometer tests. It was concluded that modified sulfur substituted asphalt mixtures had higher resistance against cracking of the pavement at low temperatures, and least permanent (plastic) deformation at high temperatures.

Keywords: DMA; Modified sulfur; Modulus; Rotational viscosity; Loss modulus

1. Introduction

In recent years the availability of large quantities of elemental sulfur in the world has generated a resurgence of interest in the utilization of sulfur in construction materials [13,32,24,10]. Numerous investigations have been undertaken and patents filed on sulfur-asphalt systems [13,32,11,28,23], in which sulfur was distributed as discrete particles acting to improve fluidity for compaction when molten and as filler when solidified. It was considered that a sulfur-asphalt composite could be produced in which the sulfur would form an interconnected network, thus giving the system additional structural rigidity. Such a composite could have interesting possibilities as a repair material for roads damaged by traffic and frost action. It was recognized that for this application, the material should be sufficiently fluid to self-compact and be able to form a good bond with the substrate. Therefore the aim of this research is to study the rheological behavior of modified sulfur asphalt binder to predict the major distresses that could occur in hot mixed asphalt (HMA) pavement such as rutting, and cracking. Recently, several researchers have used DMA to perform the dynamic mechanical analysis of asphalt binders Silvia Caroa et al. [29], asphalt mastics, and HMA mixtures. For example, Hossain and Zaman [16] established a DMA-based protocol to obtain G^* and δ of both unmodified and polymer-modified PG binders. William [35] uses DMA to characterize the aging of asphalt binder. He estimate the critical temperature, T_c from the cross-over temperature at which G'' equals G' at a frequency of 10 rad/s and chosen as a criterion to assess the advancement of hardening (aging). DMA was found to be efficient and reliable in assessing the fatigue and healing characteristics of asphalt mastic and HMA mixtures, and evaluating the rate of damage accumulation in mastics in

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Peer review under responsibility of Chinese Society of Pavement Engineering.

https://doi.org/10.1016/j.ijprt.2018.07.005

1996-6814/

the studies performed by Kim et al. [19] and Lytton et al. [21]. The temperature sensitivity on asphalt binders can be examined by performing temperature sweep tests [7,37]. Temperature sweep tests on an asphalt binder approximate a temperature at which it will satisfy the rutting factor specified by the Superpave. DMA was found to be an efficient tool to perform dynamic mechanical properties of biomodified asphalt binders, Zhai et al. [37] have performed temperature sweep tests to obtain the set points (the temperature at which storage modulus, $G' =$ loss modulus, G'') of emulsified asphalt binders. The temperature susceptibility of selected asphalt binders under short-term and long term aged conditions was predicted in the study performed by Clyne and Marasteanu [7] by using a dynamic shear rheometer (DSR). In this study, asphalt binder was substituted, by a ratio reached to 40%, with modified sulfur. The rheological properties of the prepared mixtures were studied using DMA at which temperature sweep test was conducted from – 40 °C to 80 °C on sulfur asphalt binder to study the behavior of the mixture at high and low temperatures using these parameters complex modulus G^*, G^*/sindelta (Rutting factor) and tan δ. In addition, the conventional tests including softening point, penetration, and ductility were conducted. As well as the rotational viscosity of the mixtures was measured in temperature range of 20–150 °C.

2. Materials and methods

2.1. Materials

In all experimental tests the elementary sulfur was obtained from (E-Chem. Company, Cairo, Egypt) as byproduct with 99.9% purity, bitumen 60/70 paving asphalt was obtained from the Suez Company, and olefinic hydrocarbons C5 byproducts of Alex petroleum distillates company, Egypt.

2.2. Methods

2.2.1. Preparation of modified sulfur

In an oil bath, 10 wt% mix of 7% residual olefinic hydrocarbons obtained from petroleum distillate fractions $C_5 + 3\%$ bituminous residue and 90 wt% of molten sulfur were mechanically mixed at a controlled temperature of 145 °C for almost 3 hours. The modified sulfur was characterized in more details in Souaya [31].

2.2.2. Partial substitution of asphalt binder 60/70 with modified sulfur

Asphalt binder was partially substituted with different percentages of modified sulfur (20%, 30%, and 40 wt%), and each sample was mechanically mixed with high shear rate for at least 30 minutes to attain the required compatibility at a temperature above the melting point of the modified sulfur (120 °C) but that doesn't exceed 140 °C. At that temperature range, the sulfur is completely distributed in asphalt binder, without the evolving of any gases such as H_2S(Hydrogen sulfide gases).

2.2.3. Physical tests of modified sulfur asphalt mixture

2.2.3.1. Penetration test. The standardized procedure for this test can be found in [6]. It is an empirical test which measures the consistency (hardness) of asphalt at a specified test condition. In the standard test condition, a standard needle of a total load of 100 g is applied to the surface of an asphalt sample at a temperature of 25 °C for 5 s. The amount of penetration of the needle at the end of 5 s is measured in units of 0.1 mm (or penetration unit).

2.2.3.2. Ring & ball softening point test. The ring and ball softening point test [3] measures the temperature at which the asphalt reaches certain softness. When the asphalt is at its softening point temperature, it has approximately a penetration of 800 or an absolute viscosity of 13,000 poises. This conversion is only approximate and can vary from asphalt to another, due to the non-Newtonian nature of asphalts and the different shear rates used by these different methods. The softening point temperature can be used along with the penetration to determine the temperature susceptibility of asphalt. Temperature susceptibility of asphalt is often expressed as given in (Eq. (1):

$$M = [\log(P_2) - log(P_1)]/(t_2 - t_1) \quad (1)$$

where: $M =$ temperature susceptibility; t_1, $t_2 =$ temperatures in °C; $p_1 =$ penetration at t_1; $p_2 =$ penetration at t_2.

Since the asphalt has approximately a penetration of 800 at the softening point temperature, the softening point temperature can be used along with the penetration at 25 °C to determine the temperature susceptibility as represented in (Eq. (2)):

$$M = \frac{[\log\left(pen\ at\ 25\hat{A}^{\circ}C\right) - \log(800)]}{25 - S.P.temp.} \quad (2)$$

The M computed in this manner can then be used to compute a Penetration Index (PI) as in Eq. (3).

$$PI = (20 - 500M)/(1 + 50M) \quad (3)$$

The Penetration Index is an indicator of the temperature susceptibility of the asphalt. A high PI indicates low temperature susceptibility. Normal asphalt cements have a PI between −2 and +2. Asphalt cements with a PI of more than +2 are of low temperature susceptibility, while those with a PI of less than −2 are of excessively high temperature susceptibility.

2.2.3.2.1. Flash point test. The flash point test determines the temperature to which the asphalt can be safely heated in the presence of an open flame. The test is performed by heating an asphalt sample in an open cup at a specified rate and determining the temperature at which a small flame passing over the surface of the cup will cause the vapors from the asphalt sample temporarily to ignite or flash. The commonly-used flash point test method is the Cleveland Open Cup [5].

2.2.3.2.2. Ductility test. The ductility test [2] measures the distance a standard asphalt sample will stretch without breaking under a standard testing condition (5) cm/min at 25 °C). It is generally considered that asphalt with a very low ductility will have poor adhesive properties and thus poor performance in service.

2.2.4. Rheological tests of modified sulfur asphalt mixture

2.2.4.1. Brookfield rotational viscometer test. Brookfield rotational viscometer test as specified by ASTM D4402 [4] was used for measuring the viscosity of binders as shown in Fig. 1, the test binder sample is held in a temperature-controlled cylindrical sample chamber, and a cylindrical spindle 25, which is submerged in the sample, is rotated at a specified constant speed. The torque that is required to maintain the constant rotational speed is measured and used to calculate the dynamic viscosity, as compared with the capillary tube viscometers, the rotational viscometer provides larger clearances between the components. Therefore, it can be used to test modified asphalts containing larger particles, which could plug up a capillary viscometer tube. The readings were taken every 0.5 min. The temperature range is starting from 20 °C to 150 °C to study the effect of sulfur on asphalt binder flow at room temperature 25 °C and high temperature 135 °C.

2.2.4.1.1. Dynamic mechanical analyzer (DMA) test. Various forms of (DMA) are used to measure the rheological properties of bituminous binders, usually by means of oscillatory type shear mode. The principle used with the DMA is to apply sinusoidal, oscillatory stresses and strains to a thin disc of bitumen, which is sandwiched between the two parallel plates of the DMA.

The test can be either stress or strain-controlled, depending on which of these variables is controlled by the test apparatus. In this research the tests were carried out in TTDMA apparatus (Triton technology dynamic mechanical analyzer) in which the controlled-strain test is normally used. In TTDMA rheometer test, the clamp components are fitted as shown in Fig. 2. The samples should be mounted in pairs, with a diameter from 2 to 10 mm and thickness should not be greater than 2 mm. The drive shaft is oscillated by a precision motor with a controlled angular velocity, w. The applied force is calculated from recording the input signal to the electro-magnet coils in the driver. The displacement is measured with a Linear Variable differential transformer. From the measured torque and angle of rotation, the shear stress and shear strain can be calculated. The oscillatory strain, γ, is expressed in (Eq. (4).

Fig. 1. Rotational viscometer test.

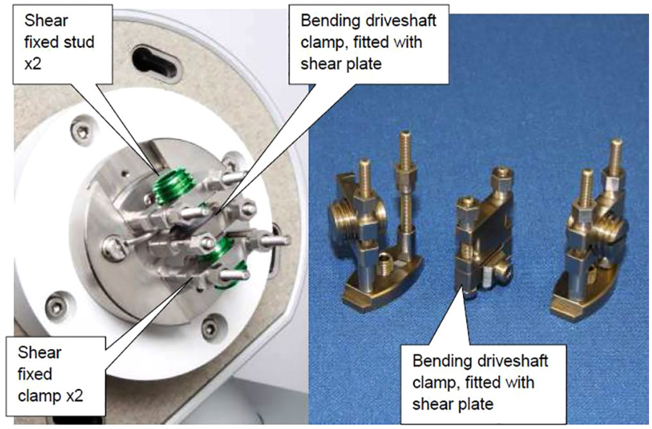

Fig. 2. Shear mode with dual sample.

$$\gamma = \gamma_0 \sin \mathrm{wt} \tag{4}$$

where: γ_o = peak shear strain; w = angular velocity in radian/second; t = time (s).

The shear stress, τ, can be expressed in Eq. (5) as:

$$\tau = \tau_0 \sin (wt + \delta) \tag{5}$$

where: τ_o = peak shear stress; δ = phase shift angle.

SHRP standardized the dynamic shear rheometer test for its usage in measuring the asphalt properties at high and intermediate service temperatures for specification purposes. In the standardized test method [1], the oscillation speed is specified to be 10 (radian/second). The amplitude of shear strain to be used depends on the stiffness of the binder, and varies from 1% for hard materials tested at low temperatures to 13% for relatively softer materials tested at high temperatures.

3. Results and discussion

3.1. Physical properties of sulfur asphalt mixtures

In this study, asphalt binder has been partially substituted with different percentages of modified sulfur and these are: 20%, 30%, and 40 wt% to give 0/100, 20/80, 30/70, and 40/60 sulfur asphalt mixtures. The conventional test methods such as softening point, penetration, and ductility have been examined to characterize sulfur asphalt mixtures performance. The physical properties of the resulting mixtures are summarized in Table 1. According to the obtained data, increasing sulfur content from 20% to 40% causes an increase in softening point, and a decrease in penetration at 25 °C. This is due to the reaction of sulfur with naphthenes-aromatics moiety in asphalt that are partially transformed into polar-aromatics, mainly polysulfides Petrossi et al., [25]. It was stated by Qarles and Vlugter [26] that sulfur reacts not only with hydrocarbons but also with organic compounds. For example, sulfur reacts with indole to simultaneously form polysulfides. Therefore, the change in the chemical structure of asphalt caused by introducing sulfur involves a change of its colloidal structure that has resulted in the increase of the asphaltenes/resins ratio which causes the structure to be changed into the gel structure. It was stated by Lu et al. [20] that lower values of PI indicate higher temperature susceptibility, and asphalt mixtures with higher PI are more resistant to low temperature cracking as well as permanent deformation. According to the obtained PI data shown in Table 1, the PI value of 40/60 sulfur asphalt mixture is 0.9 which is ten times larger than that of the base bitumen, indicating a ten times slower bitumen consistency loss. This behavior stems from the very low temperature susceptibility of the added sulfur which has a polymeric nature. That will result in a higher resistance against thermal cracking of the pavement at low temperatures, and lower permanent (plastic) deformation at high temperatures.

3.2. Brookfield rotational viscosity of sulfur asphalt mixtures

The physical structure of a sulfur/asphalt (S/A) mixture is complex. In addition to asphalt itself consists of a complex colloidal dispersion of resins and asphaltenes in oils, introduction of sulfur, which on cooling congeals into finely dispersed crystalline sulfur particles and in part reacts with the asphalt, necessarily complicates the rheology of such a S/A mixture. Measurements made with Brookfield rotational viscometer allow to predict real field performance of the binder under the operational conditions. The dynamic viscosity of the prepared sulfur asphalt mixtures has been determined over a wide temperature range; 20–150 °C as shown in Fig. 3. A wide range of temperatures was chosen to study the effect of sulfur on workability of binder at 60 °C and above 120 °C to study the rheology of binder at the temperature of mixing with aggregate and above sulfur melting temperature.

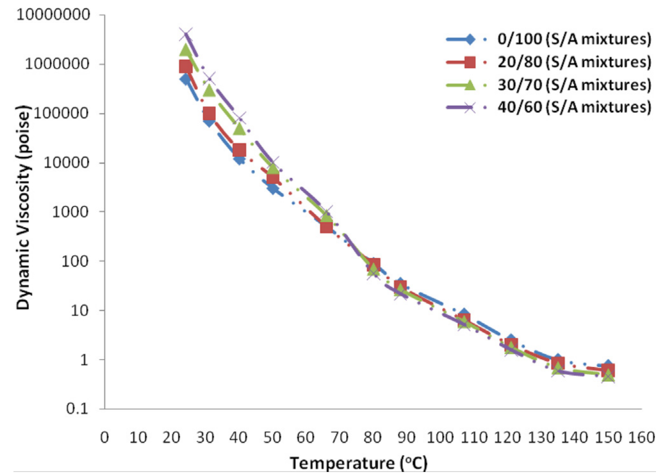

Fig. 3. Brookfield rotational viscosity of sulfur asphalt mixtures at a wide temperature range of 20–150 °C.

At low temperatures, S/A mixtures give higher viscosities above that exhibited by the base asphalt binder. The increase in viscosity appears to be maximized in the temperature range of 25–60 °C below sulfur melting temperature. This is due to the reaction of polymeric sulfur with naphthenes-aromatics moiety in asphalt that is partially transformed into polar-aromatics, mainly polysulfides Petrossi et al. [25].

While at high temperatures >120 °C, the viscosity for binders containing dispersed sulfur has dropped below that of the same asphalt, and the viscosity of the 40/60 (S/A) mixture has been lowered by a factor of 1.6 than that of base asphalt binder, allowing a reduction in power consumption by decreasing temperature of binder heating and asphalt concrete mix preparation. Therefore, 40% sulfur substitution of asphalt allows heat energy saving of paving asphalt without deterioration in the characteristics of the resulting asphalt concrete. These results agreed with the results of the previous work studied by Vitaliy Gladkikh et al. [34] and Deme et al. [9]. The decrease in the kinematic viscosity at 135 °C is due to the partial decomposition of polysulfides to form a polysulfide with a lower degree of polymerization and free sulfur. It has been shown by Qarles and Vlugter [26] that cleavages are

Table 1
Physical properties of base binder and sulfur asphalt mixtures.

Characteristics	ASTM Standard	Sulfur/asphalt mixtures			
Sulfur/Asphalt content (wt%)		0/100	20/80	30/70	40/60
Penetration at 25 °C, 100 g (0.1 mm)	D 5	62	50	45.5	40.1
Softening point (ring and ball) °C	D 36	50	56	59	62
Specific gravity (at 25 °C)	D 2041	1.02	1.025	1.0537	1.071
Flash Point (Cleveland Open Cup) °C	D 92	+250	+250	+250	+250
Ductility (@ 25 °C , 5 cm/min.) cm	D 113	+150	+150	+150	+150
Penetration Index		−0.7	0.309	0.6	0.9

not affecting the newly formed bonds between sulfur and carbon atoms that are investigated by sulfur isotopes.

3.3. Dynamic mechanical analysis (DMA)

In this research, a complete study on the effect of high and low temperatures on the behavior of sulfur asphalt mixtures has been illustrated as follows.

3.3.1. Studying the high temperature behavior of sulfur asphalt mixtures

A temperature sweep has been applied over the range of 20–80 °C at a fixed angular velocity of 10 rad/s and 12.5% strain to ensure measurement in the linear region. All the samples have been held at a defined, constant temperature for 10 min, and then the temperature has been varied in 2 °C increments. Various viscoelastic parameters, such as G' (storage shear modulus), G'' (Loss shear modulus), and tan δ have been collected automatically from DMA test. The complex shear modulus G^* has been calculated from G' and G'' as shown in Eq. (6)

$$G^* = \sqrt{G'^2 + G''^2} \tag{6}$$

The temperature (20–70 °C) dependency of complex shear modulus (G^*) for the base asphalt binder and the different contents of sulfur asphalt mixtures are shown in Fig. 4. The obtained results indicate that sulfur asphalt mixtures show an increase in the G^* compared to the base bitumen.

Measurement of phase angle (delta) is generally considered to be more sensitive to the chemical and physical structure of the modified asphalt binder than complex modulus. The reduction in tan δ value exhibits a more elastic behavior of asphalt binder Jianying et al. [18]. Fig. 5 shows the results of tan δ of sulfur asphalt mixtures against temperature at the frequency of 10 rad/s. It has been noticed that sulfur asphalt mixtures give a more decrease in the value of tanδ at 40 °C than base asphalt binder; the decreasing extent of tan δ becomes greater when sulfur ratio increases. This trend reveals the increase in elastic properties of the sulfur asphalt mixtures. It was stated by Goodrich [14], and Vishnu Radhakrishnan [33] that a low tan δ value of the binder tends to correlate with high rutting resistance. Thus, a low tan δ value of the sulfur asphalt mixture is desirable to reduce rutting potential as stated by Yong-Joon Lee et al. [36].

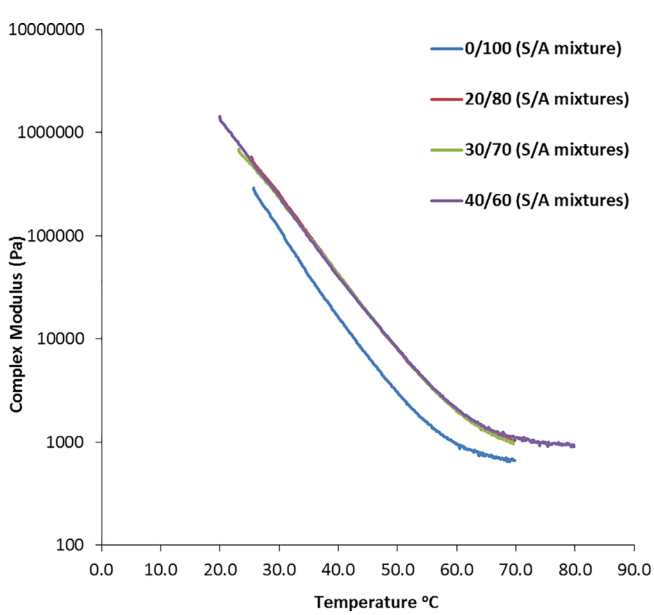

Fig. 4. Complex modulus of sulfur asphalt mixtures at temperature range of 20–80 °C.

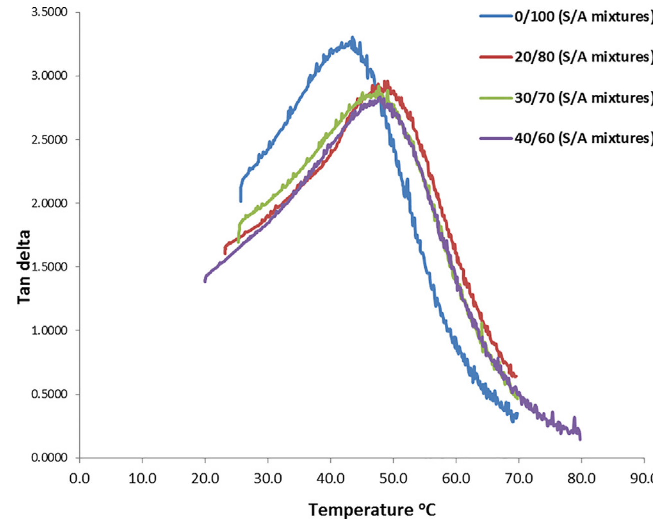

Fig. 5. Tan delta of sulfur asphalt mixture at temperature range of 20–80 °C.

In the Superpave asphalt specification, the visco-elastic parameters (G', G″, and tanδ) have been used to compute G^*/sin δ. It is known that the temperature of the asphalt binders when G^*/sinδ is equal to 1 KPa is defined as a criterion for the high temperature performance of asphalt binder in SHRP (Strategic Highway Research Program) tests [12,27]. Plots of G^*/sinδ versus temperature are displayed in Fig. 6. The results reveal that the maximum anticipated temperature is improved when asphalt binder is substituted by 40% sulfur. The resulting temperatures, from the testing data obtained, of sulfur/asphalt mixtures: 0/100, 20/80, 30/70, and 40/60 are determined as 52 °C, 58 °C, 64 °C, and 70 °C respectively. This indicates that the sulfur asphalt mixtures have a higher performance grade (PG) than base binder. It is also known that higher G^*/sin δ values correlate with higher rutting resistance (permanent deformation). Hence, sulfur/asphalt mixtures would have higher rutting resistance than base binder that agree with results of Jacques Colange [17].

3.3.1.1. Studying the low temperature behavior of sulfur asphalt mixtures. Another critical condition of an asphalt binder is at the lowest pavement temperature, at which the asphalt mixture is the weakest and most susceptible to crack when stressed. The low temperature behavior of

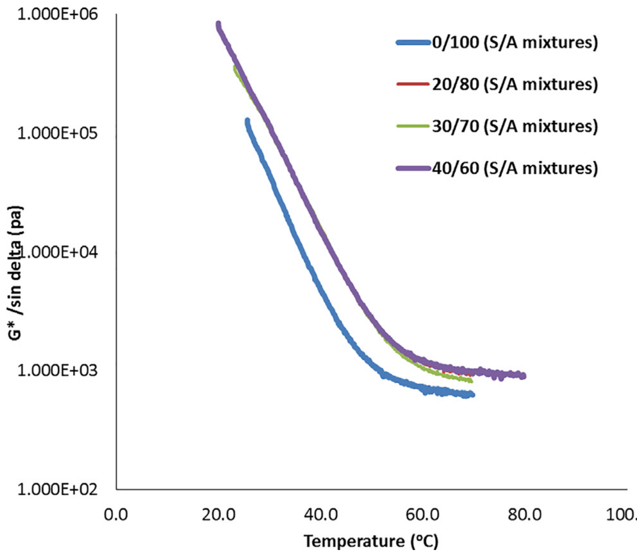

Fig. 6. G^*/sin delta of sulfur asphalt mixtures at temperature range of 20–80 °C.

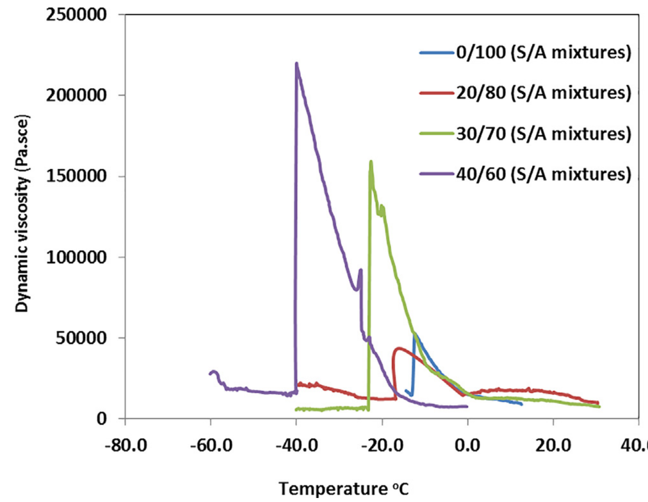

Fig. 8. The dynamic viscosity of sulfur asphalt mixtures at low temperature ranges.

sulfur/ asphalt mixtures has been studied by using DMA test at which the temperature sweep has been applied over the range of – 40 °C to 20 °C. Various viscoelastic parameters, such as G' (storage shear modulus), G'' (Loss shear modulus), and tan δ have been collected automatically from the DMA test, and the complex shear modulus G^* has been calculated from G' and G'' as shown previously in Eq. (6). Moreover, the dynamic viscosity η^* has been computed as shown in Eq. (7) by considering that sulfur/ asphalt mixtures are modeled by a Maxwell model with a complex shear modulus of G^* and a viscosity of η^*.

$$\text{Dynamic Viscosity,}\ \eta^* = G^*/w \qquad (7)$$

From the DMA data shown in Figs. 7 and 8, it is noticed that sulfur asphalt mixtures have higher complex moduli and dynamic viscosities at low temperatures than those of the base asphalt binder. Furthermore, complex modulus and dynamic viscosity values increase with increasing sulfur content. This can be explained by the hardness of polysulfide present in the structure of sulfur asphalt mixtures. The data of dynamic viscosity shown in Fig. 8 confirmed that the viscosity of sulfur asphalt mixtures at the lowest anticipated temperature is kept lower than the limiting value of $2 * 10^{10}$ poises of low temperature cracking Davis [8]. Accordingly, the low temperature cracking would be unlikely to occur in sulfur asphalt mixtures. The effects of the elastic property of asphalt binder on low-temperature cracking can be also understood by analyzing how a viscoelastic material modeled by a Maxwell model with a shear modulus of G and a viscosity of η would release its stress after it is subjected to a forced strain γ_o that could be caused by a sudden drop in pavement temperature. If the material is subjected to a forced strain of γ_o at $t = 0$, the instantaneous induced stress would be equal to $\gamma_o G$, but the stress will decrease with time according to the following expression:

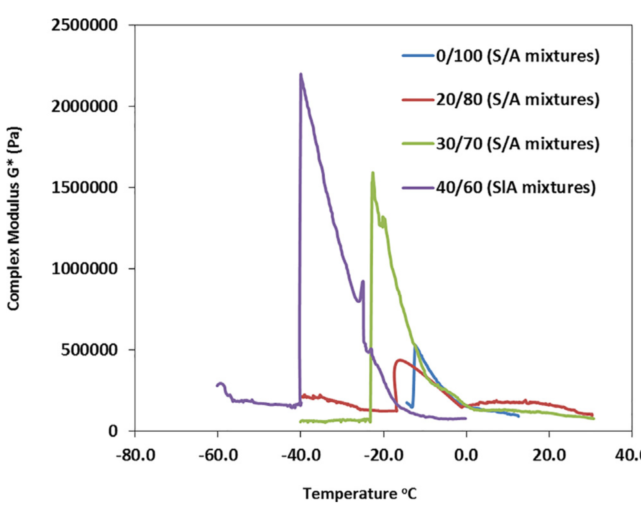

Fig. 7. The complex modulus of sulfur asphalt mixtures at low temperature ranges.

$$\tau = \gamma_0 G e^{-Gt/\eta} \qquad (8)$$

It can be seen that the rate of stress release is proportional to G/η. The reciprocal of this parameter, η/G, is commonly known as the relaxation time. To maximize the rate of relaxation, it is desirable to have a low relaxation time, η/G, or a higher G/η. The parameter tanδ as obtained from the dynamic shear rheometer (DMA) test is directly proportional to G/η. Thus, high tanδ value would be desirable to reduce the potential for low-temperature pavement cracking. As shown in Fig. 9, sulfur asphalt mixtures have higher tan δ than that of base binder which confirms the effect of sulfur on reducing the potential for low temperature pavement cracking.

Moreover, the experimental data showed that tan δ of an asphalt binder decreases with decreasing temperature. Goodrich [15] stated that, the temperature at which tan δ

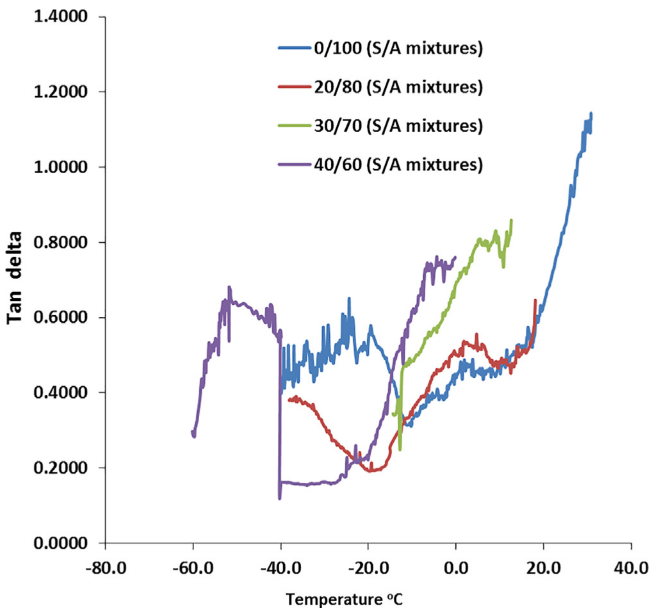

Fig. 9. Tan delta of sulfur asphalt mixtures at low temperature ranges.

of the binder is equal to 0.4 corresponds approximately to the temperature at which the asphalt mixture would reach its limiting stiffness. So, it is concluded that a limiting stiffness of base asphalt binder 0/100 (S/A) will be at temperature −2 °C while the limiting stiffness temperature of sulfur/asphalt mixtures 20/80, 30/70, and 40/60 would decrease to be −6.8 °C, −12.4 °C, and −15.2 °C respectively. From studying the overall rheological parameters of sulfur asphalt mixtures at low temperatures, it is concluded that 40/60 sulfur asphalt mixture gives the lowest anticipated temperature (−15.2 °C), after which the cracking could occur Soleimani [30].

4. Conclusion

The viscoelastic properties of modified sulfur asphalt binder have not been studied before in detail. In the present work we have performed a laboratory study to examine the viscoelastic properties of a modified sulfur asphalt binder using Dynamic Mechanical Analyser (DMA) and Brookfield rotational viscometer test. Asphalt binder was partially substituted by substantial quantities 20%, 30%, and 40%, of modified sulfur to obtain three mixtures 20/80 S/A, 30/70 S/A, and 40/60 S/A respectively to evaluate the rheological behavior of modified sulfur binders to predict the major distresses that could occur in hot mix asphalt (HMA) pavement such as rutting, and thermal cracking. The results of this work led to the following conclusions.

1. According to the conventional tests performed on S/A binder, increasing sulfur content from 20% to 40% causes an increase in softening point, and decrease in penetration value due to reaction of modified sulfur with aromatics in asphalt binder forming polysulfide.
2. The penetration index (PI) values were increased from −0.7 (base asphalt) to 0.9 for (40/60 S/A) binder confirmed that S/A mixture attains the highest resistance against thermal cracking of the pavement at low temperatures, and rutting resistance at high temperatures.
3. The viscosity of (40/60 S/A) mixture at temperatures >120 °C is lowered by a factor of 1.6 than that of base asphalt binder that allow a reduction in power consumption by virtue of decreasing the temperature of the binder heating, and asphalt concrete mix preparation by 25 °C to 30 °C. Since low preparation temperatures correspond to low emission of toxic gases and low processing costs, so we can consider the concretes that will be based on modified sulfur asphalt as green paving materials.
4. Based on the results of DMA test performed on S/A binders at low temperatures from – 40 °C to 20 °C, the low temperature cracking would be unlikely to occur since the viscosities of S/A binders at the lowest anticipated temperature is 2.25×10^6 poise which were kept lower than the limiting stiffness value of 2×10^{10} poises that attributed to the positive effect of modified sulfur in decreasing the viscosity of binder.
5. The performance grade PG of S/A mixtures was determined using DMA test through the relation between the parameter of $G^*/\sin\delta$ and temperature. The results indicated that PG of S/A mixture increases from 52 °C for base asphalt, to 58 °C, 64 °C, and 70 °C for 20/80, 30/70, and 40/60 S/A mixture respectively.
6. Also the DMA results approved that S/A mixtures give high rutting factor $G^*/\sin\delta$ than base asphalt that correlate with higher rutting resistance.

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