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Rheological master curves for modified asphalt binders

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Abstract. Modified bitumen enhances the performance properties of asphalt mixtures. A number of studies in the last decade have evaluated the performance properties of modified binders at different service temperatures. It is however possible to evaluate some modified binder time-temperature characteristics and performance properties through their rheological master curves. In this research, styrene butadiene styrene, ethylene vinyl acetate, polyphosphoric acid and crumb rubber were used to modify a PG58-22 base binder. Rheological master curves for $|G^*|$ and phase angles of these binders were generated using data from the dynamic shear rheometer and bending beam rheometer at temperatures ranging from -30°C to about 88°C. The rheological interpretation of the results indicated improved high and low temperature properties and reduced temperature susceptibility for most of the modified binders. A comparison of the modified binders was done based on the visual inspection of master curves for different temperature ranges.

1. Introduction

Modifying bitumen can improve the performance of binders in asphalt mixtures to withstand higher applied stresses, particularly under harsh environmental conditions. The performance of unmodified asphalt binders at different temperatures can be assessed using rheological parameters measured by a Dynamic Shear Rheometer (DSR), Bending Beam Rheometer (BBR) or rotational viscometer [1]. However, none of these test methods are capable of representing the benefits of polymer modification of binders. New testing methods to determine performance characteristics of Polymer Modified Binders (PMBs) are being investigated [2-4]. These tests include the Multi-Stress Creep and Recovery (MSCR) test for high-temperature binder specification [3], stress sweep or strain sweep as an accelerated binder fatigue test to consider damage and nonlinear characteristics of a binder at intermediate temperatures [2], and the new low temperature specification method using an Asphalt Binder Cracking Device (ABCD) developed to simulate thermal cracking of asphalt pavement in the laboratory [4].

The performance properties of binders are directly influenced by their rheological properties. The rheological characteristics of materials in the linear viscoelastic range are traditionally exhibited in master curves. Rheological master curves provide a fundamental understanding of the materials since they allow an estimation of mechanical properties over temperatures and loading times (or frequencies) that are experienced in the field but not tested or simulated in the laboratory. Master curves can also provide qualitative criteria for assessing binder performance at different temperatures. Information obtained from the master curves of binders and asphalt mixtures are being used to obtain input parameters for the new Mechanistic-Empirical Pavement Design Guide (MEPDG) software [5].

The present research used master curves to eval-

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Table 1. Bitumen and modifier specifications and mixing conditions.

Polymer specifications				Mixing conditions				Modified binder	
Modifier	Company	Product	Spec.	Mixer type	Mixer speed (rpm)	Mix dur. (min.)	Temp. (°C)	Dosage (%)	Grade
EVA	Hanwha	EVA 1316	19% VA content	High shear	5000	120	180	6	PG 76-22
								10	PG 82-22
SBS	Dynasol	Calprene 501	31% styrene-linear structure	High shear	5000	120	180	4	PG 70-22
								7	PG 76-28
Crumb rubber	Yazd Tire	–	12% on #30; %80 on # 50; (ambient ginding)	High shear	5000	60	180	9	PG 70-28
								18	PG 76-34
PPA	Merck	807471 PPA	85% of P2O5	Low shear	350	30	165	0.5	PG 64-22
								2.5	PG 82-22

uate and compare the efficacy of different modifiers used with a neat binder, and the performance of the resulting binders.

2. Materials and methods

Materials used in this research comprised a PG58-22 binder modified with four modifiers. Relatively low and high concentrations of the modifiers were used as shown in Table 1. The SARA fractions (ASTM D4124-01) of the base binder were 10.6% asphaltene, 11.2% saturates, 44.6% naphtene aromatics and 33.6% polar aromatics.

Two tests were conducted to generate the master curves of complex modulus G^*; the frequency sweep test using a dynamic shear rheometer and the Bending Beam Rheometer (BBR) test. The frequency sweep test was performed under a constant strain mode at frequencies between 0.01 and 40 Hz and between 5 and 88°C using a Bohlin rheometer DSR10 at its maximum temperature and frequency capacity. For temperatures of 5 to 40°C, 8-mm plate geometry with a 2 mm gap setting was used and, for 40 to 88°C, 25-mm plate geometry with a 1 mm gap setting was used according to ASTM D7175. A reference temperature of 40°C was arbitrarily chosen to generate the master curves. It was not necessary to shift the overlapping data available at this temperature. The G^* data at other temperatures was shifted based on the Time-Temperature Superposition Principle (TTSP) [6]. Different shifting techniques can be applied to master curve isotherms [7]. The absolute value of $|G^*|$ was selected as the key parameter for shifting.

BBR tests were conducted at -12, -18, -24, and -30°C to obtain a range of low temperature creep stiffness modulus data in addition to DSR data. The DSR and BBR tests consisted of two replicate measurements. The absolute value of the complex shear modulus was determined from creep stiffness data for 8 to 240 sec of loading time using the equation [6]:

$$|G^*(\omega)| = S(t)/3. \tag{1}$$

Shift factors determined for the G^* master curves were also used for the phase angle. The Kramers-Kronig equation as presented by Booij and Thoone [8] was used to estimate the phase angles from the BBR test results:

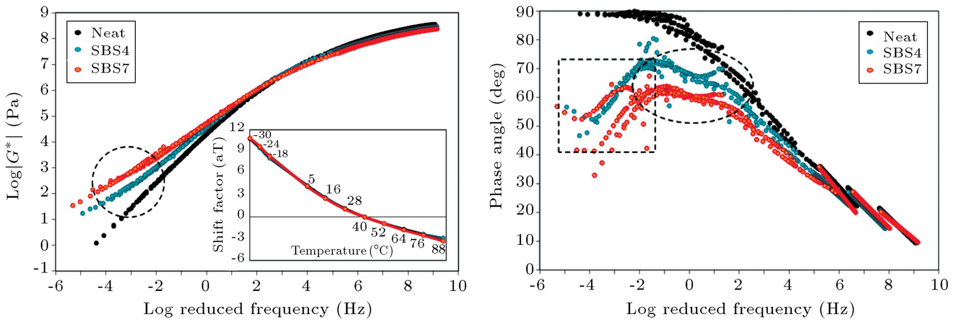
$$\delta(\omega) \cong 90 \times \frac{d\log\left(G^*(w)\right)}{d\log w}. \tag{2}$$

A second order polynomial fit was made of $|G^*(\omega)|$ and the slope of the curve was used to calculate the phase angle values. The Kramers-Kronig method is not a precise approach, thus, the curves obtained using this method were only used to give an initial estimate of the phase angle master curves in the same frequency range of the $|G^*|$ master curves and not to interpret the phase angle master curves.

3. Results and discussion

3.1. SBS modification

The $|G^*|$ master curves for both PMBs is shown in Figure 1(a). They both show considerable increase in low frequency (high temperature) range and some decrease in the frequency (low temperature) range.



(a)

(b)

Figure 1. Rheological results for neat and SBS modified binders: (a) $|G^*|$ master curves; and (b) phase angle master curves.

This indicates that SBS modification has made the binder less temperature susceptible. An increase in $|G^*|$ in low frequency range is common in polymer modification of binders. slight decrease in the slope of the PMBs in the low frequency region (dashed circles) 1(a) can be attributed to the molecular interaction, dispersion, swelling and compatibility of the neat binder and SBS [9]. Where compatibility is good, the elastomeric part of SBS absorbs the aromatic oils or maltene fractions from the binder and swells [10]. This is a binder dependent behavior that is not found for all SBS PMBs in other research [9]. The binder used in this research had high aromaticity (44.6% wt. of naphtene aromatics and 33.6% wt. of polar aromatics) and enjoyed good polymer compatibility, which resulted in a strong polymer-rich network in the low frequency and high temperature ranges of the master curve. The formation of an elastic polymer-rich network is also proportional to the polymer content [10,11] as shown in this research.

The phase angle master curve in Figure 1(b) shows a reduction in the entire frequency region, meaning that the SBS modified binders exhibit more elastic behavior than the neat binder. This reduction led to the formation of a plateau in the intermediate frequency range of the phase angle master curves (dashed ellipse), especially for the high level of modification, and a sudden decrease in the phase angle accompanied by a reverse slope in the low frequency range (dashed square). The plateau in the phase angle master curve (dashed ellipse) is an indication of the physical cross-linking of polystyrene blocks or entanglements in the modified binder [9]. The considerable reduction of the phase angle (dashed square) is also a result of polymer swelling and the polymer-rich elastic network [9]. Thus, it can be concluded that the neat binder used in this research is compatible with the SBS copolymer used and can result in better field performance.

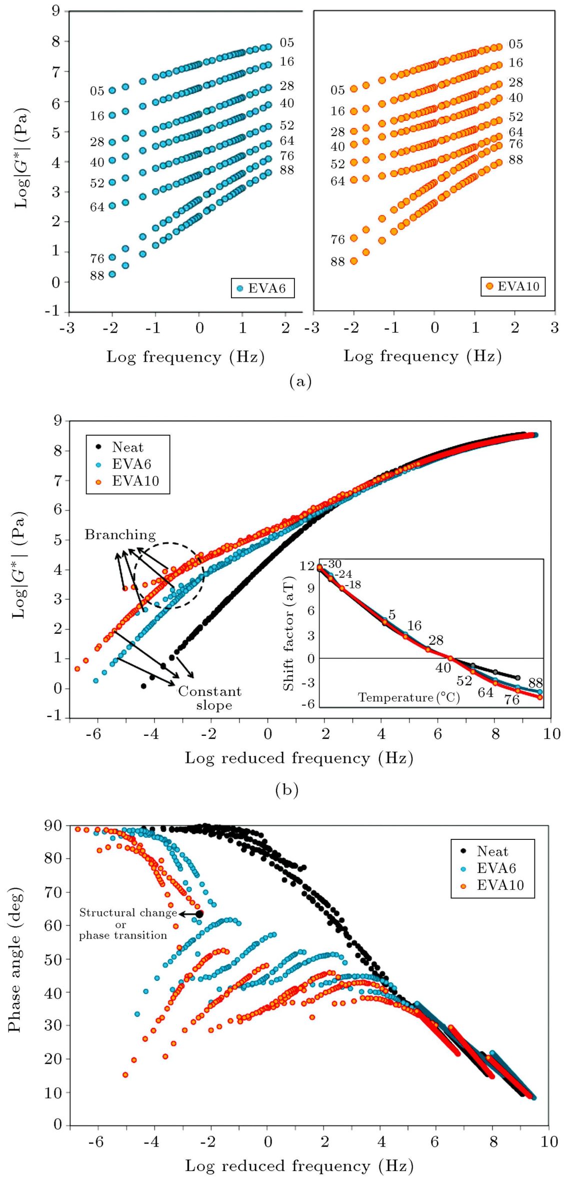
3.2. EVA modification

EVA is a plastomer that improves the physical properties of a binder through formation of a tough, rigid three-dimensional network that is plastomer-specific [12]. $|G^*|$ isotherms, $|G^*|$ master curves and phase angle master curves of the neat binder and 6% and 10% EVA modified binders are shown in Figure 2.

The $|G^*|$ isotherms diagram in Figure 2(a) show that the isotherms at and below 64°C have different slopes from isotherms at 76 and 88°C. As a result, the shape of the master curves for the EVA modified binders is different from that of the neat asphalt in Figure 2(b). It is clear that a break in the slope exists in the low frequency range (circled in Figure 2(b)). For temperatures greater than the breaking point (lower frequencies), a constant slope of isotherms can be seen. At temperatures lower than the breaking point (higher frequencies), branching of the curve is more pronounced. It has been postulated that the main reason behind this phenomenon is the melting of semi-crystalline EVA copolymers [12,13].

The isotherms in the phase angle diagram (Figure 2(c)) exhibit a completely separate two-phase data set as seen in the figure. This unique feature is attributed to a structural change or a bitumen phase transition in the binder. Furthermore a broken up plateau region can be seen at the phase angles for 30° to 50° for both modified binders (dashed rectangle). This indicates the reduced temperature susceptibility of the binders and may be indicative of a more rubber-like behavior of the PMBs. The presence of a wide plateau has been contributed to better compatibility between the neat binder and the polymer [14].

The discontinuous shape of the isotherms resembles a series of waves. Researchers have attributed this to the presence of crystalline structures at different temperatures within the EVA PMBs [12]. Differential Scanning Calorimetric (DSC) measurements on the EVA copolymer and EVA PMBs were carried out to investigate this behavior (Figure 3). The DSC on the EVA copolymer was performed using a Netzsch 200 F3 on an 11.59 mg EVA sample sealed in an aluminum pan and purged with nitrogen according to ASTM D3418. DSC measurements on the neat and EVA modified binders were performed using a DSC8000



(a)

(b)

(c)

Figure 2. Rheological results for neat and EVA modified binders: (a) $|G^*|$ isotherms; (b) $|G^*|$ master curves; and (c) phase angle master curves.

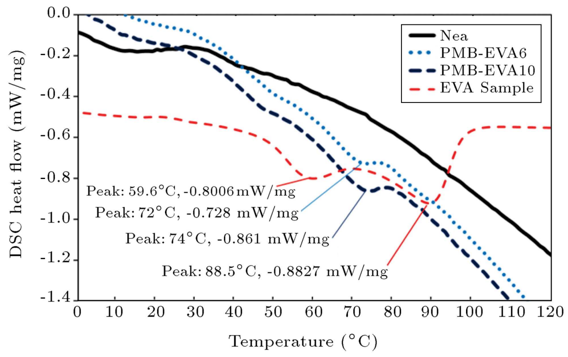


Figure 3. DSC results for EVA copolymer, neat binder, 6% EVA and 10% EVA modified binders.

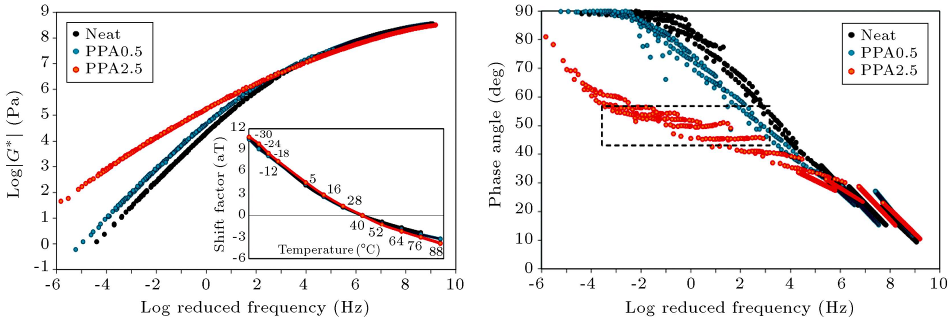
PerkinElmer device. The EVA results revealed small and large peaks at 59 and 89°C, respectively, which is a common EVA signature [15,16]. These are considered the melting points for smaller imperfect crystallite structures and larger more regularly formed structures, respectively [15,16]. The results from PMB samples of 6% and 10% EVA showed peaks at close temperatures of about 72 and 74°C; the melting temperatures of PMB. Apparently, polymer content does not alter the melting temperature significantly [17]. The melting temperature for the PMBs (about 74°C) is less than that of pure EVA polymer (89°C); this is the result of EVA swelling in the binder. During swelling, bitumen disrupts the crystalline component of the polymer and yields crystallites with a distribution of smaller sizes [17], leading to a decrease in melting temperature. The PMB melting temperature agrees with the temperature for the change of slope in the $|G^*|$ isotherms (between 64 and 76°C).

From the master curves of the EVA modified binders, it can be expected that, at temperatures below the melting point of EVA, the behavior is similar to a filler-type modification rather than a polymer-type modification [12]. This phenomenon depends on the asphalt binder [12] and the type of EVA [13,18], and can only be expected from a compatible binder and polymer like those used in this research.

3.3. PPA modification

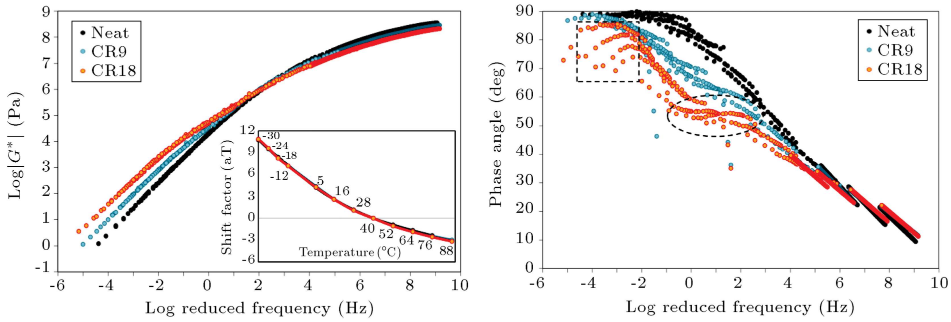
PPA catalyzes the air blowing process and is used as an independent modifier without the need for air blowing to achieve desired binder grades [19]. Figure 4 shows that the $|G^*|$ master curve for 0.5% PPA differs slightly from that of the neat binder, whereas the 2.5% PPA modification shows a distinctly different behavior. This stems from the formation of different colloidal structures within the binders [20]. Considerably lower temperature susceptibility of 2.5% PPA modified binder is also clear from its $|G^*|$ master curve.

The phase angle master curve for the 2.5% PPA in Figure 4(b) shows flat regions for a wide range of intermediate frequencies. It appears that each isotherm was independent of frequency changes and small vertical shifts occurred from one isotherm to the other in the intermediate frequency range (dashed rectangle). This type of behavior has been attributed to the transformation of binder properties from sol-like at high temperatures to gel-like at intermediate temperatures [20]. The conversion of aromatics to resins and resins to asphaltenes takes place during PPA modification, leading to an increased asphaltene content [21,22] or asphaltene swelling of the PMB [23] and a corresponding decrease in the resin fraction [24]. This effect is comparable to a mild oxidation, or blowing process that increases the peptized material



(a) (b)

Figure 4. Rheological results for neat and PPA modified binders: (a) |G^*| master curves; and (b) phase angle master curves.



(a) (b)

Figure 5. Rheological results for neat and CR modified binders: (a) |G^*| master curves; and (b) phase angle master curves.

and decreases peptizing material with a shift toward a gel structure [22].

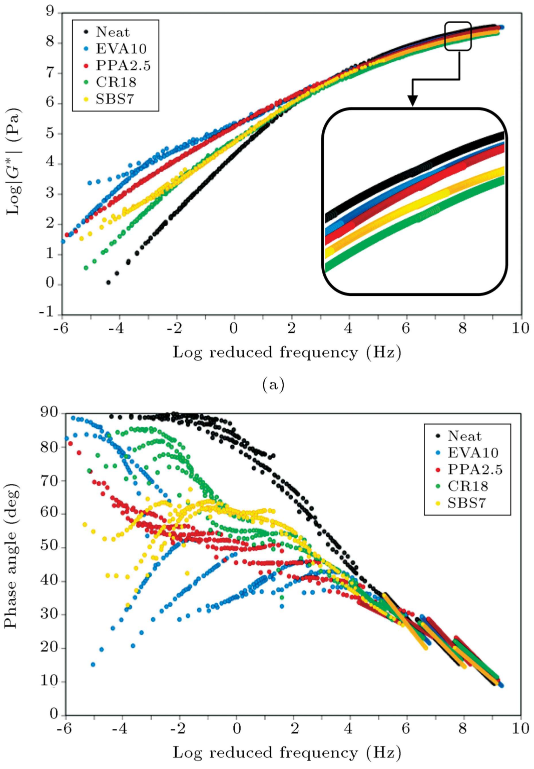
3.4. CR modification

|G^*| and phase angle master curves for crumb rubber modified benders are shown in Figure 5. Figure 5(a) shows uniform stiffness rise and fall at high and low temperatures, respectively, for both PMBs. The viscoelastic response of CR modified binders depends on the asphalt source, CR production process, particle size and content as well as the mixing conditions [25,26].

The plateau in the intermediate frequency region of Figure 5(b) for 18% CR (dashed ellipse) is in accordance with previous research [25,26]. This plateau, at phase angle values of about 55° (dashed rectangle), corresponds to the partly elastomeric behavior of PMB. It was caused by entanglement in the network of the 18% CR. The end reduction of the phase angle (dashed square) is also as a result of the swelling of CR particles at very high temperatures, after absorbing components of the bitumen, as occurred for the SBS PMB. The last phenomenon was not observed for 9% CR binder, but the plateau formation was about to take place.

3.5. Performance interpretation

Figure 6 compares modified binders with high levels of modification. All exhibit stiffening at high



(a)

(b)

Figure 6. Rheological results for neat binder and high-dosage modified binders: (a) |G^*| master curves; and (b) phase angle master curves.

temperatures and slight or considerable softening at low temperatures, both being favorable. Stiffening of PMBs at high temperatures together with lower phase angles guarantees more resistance to permanent deformation.

At temperatures below melting (about $74°C$), EVA10 showed considerable stiffening and higher elasticity (lower phase angles). Since the tough, three-dimensional network can withstand permanent deformation at high temperatures, similar behavior was exhibited by EVA6, as seen in Figure 2.

SBS7, with highly elastic behavior, and PPA2.5, with a unique stiffening response, also exhibit good high temperature performance. SBS master curves in Figure 1 indicate that SBS4 binder, with a medial master curve, performed better than the neat binder, but not as good as SBS7 over the range of temperatures. The master curve in Figure 4 for PPA0.5 shows marginal improvement for high temperature performance, as expected. The improved high temperature properties of CR18 are the result of increased $|G^*|$ and the presence of an elastic network in the phase angle master curve and has also been found in past research [27,28]. High temperature properties of the same modified binders and mixtures have been studied elsewhere and similar rankings for PMBs were reported [29].

The literature shows that the improvement of the low temperature properties of CR modified binders is more dependent on binder type than the crumb rubber [25]. The reduction in creep stiffness of CR modified binders may be caused by softening of rubber particles, which are softer than the base bitumen at low temperatures [30]. CR18 decreased the stiffness of the modified binders more than the others, followed by SBS7 (Figure 6(a)). A similar effect for high rubber content has also been reported by Tabatabaee et al. [28]. Some researchers have reported improved low temperature binder performance for PPA modified binders [21] and others have reported a negative effect [31]. A comparison of stiffness values at high frequencies in Figure 6(b) shows minimal low temperature improvement for PPA2.5 modified binders over the neat binder. Except for CR18 and SBS7, the low temperature improvement of other binders was marginal to none. For the same reason, no low temperature improvement was expected at high temperatures for PPA0.5. The EVA10 and EVA6 modified binder curves closely resemble the neat binder curve at low temperatures.

In the intermediate temperature range, stiffening was less for CR18 and SBS7 than for the other modifiers. The relatively low stiffness of CR18 and SBS7 modified binders and the intermediate temperature plateau shown in Figure 6(b) are indicative of decreased fatigue cracking in the intermediate temperature range. CR9 and SBS4 show a lower effect for the same reasons.

Both positive [23] and negative [19,31] effects of PPA modification on fatigue performance have been reported. In this research, lower phase angle values were observed for PPA2.5 and EVA10 (and EVA6) modified binders in the intermediate temperature range of the master curves. Because they were not accompanied by lower stiffness values, the reduction in the phase angles is not necessarily an improvement in fatigue behavior as it was for SBS7 and CR18. These performance estimates based on the master curves of $|G^*|$ and phase angles are in good agreement with the performance grades of these binders according to AASHTO M320, as shown in Table 1. Table 2 summarizes the rheological interpretations of these binders.

4. Closing remarks

This research focused on the evaluation and interpretation of PMB rheological master curves based on the

Table 2. Rheological features and expected performance for PMBs

Binder	Rheological interpretation	High . temp.	Inter. temp.	Low temp.
SBS4	Polymer swelling, physical cross-linking of polystyrene blocks,	+	+	+
SBS7	entanglements	++	++	++
EVA6	Different crystalline structures at different temperatures,	++	*	-
EVA10	melting of EVA copolymers	++	*	-
PPA0.5	No major rheological specialty	-	-	-
PPA2.5	Transition from sol-like to gel-like behavior	++	*	-
CR9	No major rheological specialty	+	+	+
CR18	Polymer swelling, physical cross-linking, entanglements	++	++	++

(-)no major changes; (+) slight improvement; (++) considerable improvement; and (*) no comments.

shape of their master curves with the aim of predicting binder performance at different temperature ranges.

Good agreement was observed between the interpreted performance and the measured PG. However, peculiarities observed in the master curves of each binder have implications for the engineering of modified binders and their detailed performance that are not offered by PG alone.

- Good compatibility between the neat binder and all tested modifiers exists.
- Higher levels of SBS and CR modification exhibit better compatibility and performance properties than the lower levels of concentration at all temperature ranges.
- The presence of EVA crystalline structures with melting points at different temperatures results in distinctly different high temperature performance properties for EVA modified binders.
- SBS physical cross-linking, crumb rubber swelling, and the transition from sol-like to gel-like behaviour in the PPA binder have significant effect in rheological behavior and performance properties of modified binders.
- The modified binders exhibit lower temperature susceptibility than the neat binder.

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Biographies

Seyed Mohammad Asgharzadeh received the BS degree in Civil Engineering and MSc degree in Transportation Engineering from Sharif University of Technology (SUT), Tehran, Iran, in 2004 and 2006, respectively. He is currently a PhD candidate in Transportation Engineering at the same university. His research interests lie in the general areas of asphalt modification, pavement materials viscoelastic modeling and laboratory testing of bituminous materials. He has the experience of teaching in different transportation fields including pavement design, railway engineering, airport design, highway and traffic engineering, road geometric design and engineering economics in Sharif University of Technology, Kish international campus, and Babol University of Technology. He has recently published a paper in Materials and Structures, presenting a new model for asphalt binder master curves.

Nader Tabatabaee has been a member of the Civil Engineering Faculty at Sharif University of Technology (SUT) since 1991. He teaches and conducts research on pavement materials and design, particularly on modified asphalts and asphalt mixtures. He received his BS from the University of California at Berkeley and his MS and PhD from The Pennsylvania State University. He has published more than 60 peer reviewed publications and conference papers in his field as well as many technical reports for various public and private agencies. From 1997-2001, he served as the chair of the Department of Civil Engineering at SUT and, from 2001 to 2006, as the Dean of Graduate Studies. Dr. Tabatabaee is a founding member of the Civil Engineering Congress in Iran and the Middle East Society of Asphalt Technologists (MESAT) and is a member of TRB, AAPT, ISAP and RILEM. His areas of research interest include pavement instrumentation, pavement design and maintenance, and asphalt technology.