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Study on viscosity of conventional and polymer modified asphalt binders in steady and dynamic shear domain

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Abstract This study focuses on evaluating the flow behavior of conventional and polymer modified asphalt binders in steady- and dynamic-shear domain, for a temperature range of 20–70 °C, using a Dynamic Shear Rheometer (DSR). Steady-shear viscosity and frequency sweep tests were carried out on two conventional (VG 10 and VG 30) and two polymer (SBS and EVA) modified asphalt binders. Applicability of the Cox–Merz principle was evaluated and complex viscosity master curves were analyzed at five different reference temperatures. Cross model was used to simulate the complex viscosity master curves at different temperatures.

It was found that asphalt binders exhibited shear-thinning behavior at all the test temperatures. The critical shear rate increased with increase in temperature and was found to be lowest for plastomeric modified asphalt binder. The Cox–Merz principle was found to be valid in the zero-shear viscosity (ZSV) domain and deviated at higher frequency/shear rate for all the binders. Results from the study indicated that the ratio of ZSV can be successfully used as shift factors for construction of master curves at different reference temperatures. Cross model was found to be suitable in simulating the complex viscosity master curves at all the test temperatures. Analysis of model parameters indicated that a strong relationship exists between ZSV and the critical shear rate. ZSV and critical shear rate varied exponentially with temperature. This relationship was used to propose a simple equation for assessing the shift factors for construction of master curves.

Keywords Bitumen · Polymer · Cox–Merz · Master curves · Zero-shear viscosity · The Cross model

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1 Introduction

Use of different modification techniques for asphalt binders has become very popular in the recent years for enhancing the rheological and viscoelastic response of the original binders (Alataş and Yilmaz 2013; Nuñez et al. 2014; Yang and You 2015). Amongst these techniques, polymer modification has gained a lot of attention (Alataş and Yilmaz 2013; Kumar and Veeraragavan 2012; Yildirim 2007). Two of the most commonly used polymers for modification of asphalt binders are elastomeric styrene butadiene styrene (SBS) and plastomeric ethylene vinyl acetate (EVA) (Airey 2002, 2003; Ameri et al. 2013; Luksha et al. 2006; Panda and Mazumdar 1999; Rek et al. 2013; Sengoz and Isikyakar 2008a; Zhu et al. 2014).

Quantification of mechanical/rheological properties of any material can be done using predictive models and equations. It is time saving, less laborious and does not require any skilled operators. From the past six decades much research have been conducted for predicting the linear viscoelastic characteristics of bitumen using nomographs, empirical equations and use of mechanical elements (spring and dashpot) (Saboo and Kumar 2016). Most of these techniques have been applied to predict the variation of complex modulus and phase angle master curves, at any desired reference temperature. Yusoff et al. (2011) presented a brief overview of all the models developed over the past years. Most of the algebraic models consist of large number of model parameters which are empirical and does not have any physical significance. A more simple model is hence desired which can be directly related to the flow properties of the binder.

Viscosity of viscoelastic materials can be determined using steady-shear and dynamic-shear response. In the dynamic domain the viscosity is termed the complex viscosity. The empirical Cox–Merz rule is used to examine the viscosity correlation between dynamic oscillatory and steady-shear measurements (Shan et al. 2012). The change in the viscosity with variation in loading time at a particular temperature has raised concerns about interpreting the results of viscosity tests. Zero-shear viscosity (ZSV) of an asphalt binder is considered as an alternative performance indicator of resistance to permanent deformation of asphalt pavements (Hajikarimi et al. 2015; Rowe et al. 2002). The viscosity at zero shear or very low shear rates remains constant and is a replica of the dissipated motions in negligibly perturbed, equilibrium “no-flow” structures. Various mathematical models have been proposed to calculate the ZSV from the viscosity response of asphalt binders. Out of the various models, the Cross model had been very promising for simulating the shear-thinning behavior of polymers (Liao and Chen 2011). However, not many studies have been done to obtain and analyze the Cross model parameters for asphalt binders.

This study focusses on studying the viscosity of asphalt binders in steady- and dynamic-shear domain at different temperatures. The suitability of the Cross model for simulating the complex viscosity master curves will be explored and the use of model parameters in defining the flow behavior of the binders will be analyzed.

1.1 Cox–Merz principle

The study of asphalt binder in dynamic and steady state are usually done separately. Researchers found that the association between these two states stands important for arriving at the rheological curves and mathematical interrelationships (Shan et al. 2012). Cox–Merz relationship is one such tool which connects the dynamic- and steady-state properties. Cox–Merz relationship is an empirical relationship relating dynamic- and steady-state viscosities applicable mainly to polymer melts. Cox and Merz (1958) were the first researchers who observed that the steady-state viscosity at some shear rate ($\dot{\gamma}$) can be successfully related to the dynamic complex viscosity as follows:

$$\left|\eta^*(\omega)\right| = \eta(\dot{\gamma}). \tag{1}$$

$|\eta^*(\omega)|$ is the absolute value of complex viscosity at a frequency ω equal to the shear rate $\dot{\gamma}$. As an empirical relationship, its applicability as a general rule stands debatable (Doraiswamy 1991; Shan et al. 2012; Winter 2008). Modifications and changes to the principle have also be done to relate the dynamic and steady-state relationships.

1.2 Construction of master curves

One of the most useful extrapolation techniques for rheological parameters in dynamic mechanical analysis is based on the time-temperature superposition principle (TTSP) (Di Benedetto et al. 2011). The technique can be used to predict the rheological behavior of time and temperature dependent by shifting the curves from tests at different temperatures horizontally along a logarithmic time axis to generate a single master curve at a reference temperature (Airey et al. 2016). The shift factors of thermorheologically simple materials can be related to temperature using either the Williams–Landel–Ferry (WLF) or the Arrhenius equation (Williams et al. 1955). The WLF equation which is based on the free volume concept of Doolittle and has been widely used to describe relation between horizontal shift factor (a_T) and temperature dependency of bitumens can be written as

$$\log a_T = \frac{-C_1(T - T_{ref})}{C_2 + (T - T_{ref})}. \tag{2}$$

Here, C_1 and C_2 are empirical constants. T_{ref} is the reference temperature (K), and T is the test temperature (K). The implication of this model lies in the determination of these constants. Moreover, this equation has been mainly found suitable when reference temperature is close to T_g. Many universal constants have been proposed based on the reference temperature. The most famous and frequently used values are 8.86 and 101.6 for C_1 and C_2 as proposed by Williams et al. (1955). These values have shown a good fit when $T - T_{ref} > -20$ °C. Other values proposed by Anderson et al. (1991) were 19 and 92, which were based on some defining temperature T_d. This defining temperature is not very clear and is bitumen specific.

Observation of the dependence of viscosity of liquids on temperature led to the development of the Arrhenius relationship that can be expressed as

$$a_T(T) = \exp\left[\frac{E_a}{2.303R}(1/T - 1/T_{ref})\right]. \tag{3}$$

Here E_a is called the activation energy, typically 250 kJ/mol for bituminous binders. It is the minimum energy required for any intermolecular movement. R is the universal gas constant (8.314 J/°K-mol). The Arrhenius equation has been found to fit data at the terminal and plateau zones for linear polymers as long as the temperature is well above the glass transition temperature T_g. Also a meaningful activation energy has not been defined so far. Variation in E_a with frequency and modulus has also been reported.

Studies have also shown that zero-shear viscosity (ZSV) can also be used as the shift factors for obtaining the master curve. It can be written as

$$a_T(T) = \frac{\eta_0(T)}{\eta_0(T_{ref})}. \tag{4}$$

The viscosity is a fundamental property to define the flow behavior of any material. In this study the ZSV obtained from steady-shear viscosity test was used as the shift factor for obtaining complex modulus master curves.

1.3 Cross model

The shear-thinning behavior of bitumen can be expressed using generalized Newtonian model. The nonlinear characteristics of the shear viscosity can be quantified using this simple model, which can be used for practical applications. Amongst the several models, the Cross model has been commonly used to characterize the polymer melt flow. Mathematically, it can be written as follows:

$$\eta = \eta_\infty + \frac{\eta_0 - \eta_\infty}{1 + (\lambda \cdot \dot{\gamma})^m}. \tag{5}$$

Here η is the viscosity and $\dot{\gamma}$ is the shear rate. η_∞ and η_0 represent the infinite and zero-shear viscosities. The zero-shear viscosity (ZSV) can be defined as a viscosity in which the rate of shear approaches zero and it is a function of the temperature and molecular weight of the material under consideration. λ is the time constant pertaining to the relaxation times of the polymer in solution and m is the dimensionless exponent. λ signifies an average time required for the entangled molecular chains to line up in the direction of flow when a certain shear stress is applied. This is a function of the zero-shear viscosity (or molecular weight) and temperature (Rao 2014). Usually the value of η_∞ is very low in magnitude (for bituminous materials) and is neglected. For small values of η_∞, the Cross exponent m tends to a value $(1 - n)$,, where n is a shear susceptibility index. n is a parameter which quantifies the resistance of the molecular chain against the shear flow. The value of n ranges from zero to one. Smaller the values, lower is the resistance. For Newtonian materials the value of n is 1. The final equation for asphalt binders can now be written as

$$\eta = \frac{\eta_0}{1 + (\lambda \cdot \dot{\gamma})^m} \tag{6}$$

For the shear rate, $\dot{\gamma}^c$, where $\eta = (\eta_0 + \eta_\infty)/2$, the Cross time constant is $\lambda = 1/\dot{\gamma}^c$. Generally, $\dot{\gamma}^c$ gives an order of magnitude of the critical shear rate marking the end of the zero-shear rate Newtonian plateau or the onset of the shear-thinning region. It is therefore important to recognize the shear rate dependence of the rheological behavior of bitumen and the difficulty involved in obtaining experimental data over the applicable shear rate range. In this study the Cross model was used to simulate the complex viscosity master curves at different temperatures. The model parameters were obtained using a nonlinear regression technique by minimizing the sum of least squares between the measured and modeled data.

2 Materials

This study was done on four different asphalt binders. VG10 and VG 30 were the viscosity graded (VG) binder. VG 10 was modified using Styrene Butadiene Styrene (SBS) and Ethylene Vinyl Acetate (EVA) at 3% and 5% modification level. The optimum dosage were directly taken from another done by the authors (Saboo and Kumar 2015). EVA copolymer (Evatane®) was supplied in pellet form of 2–3 mm in size. Evatane® 2805 which contains vinyl acetate content of 27–29% is a highly flexible plastomer designed for bitumen modification and especially for road paving. The SBS polymer used was Kraton D-1101 which

Table 1 Properties of Evatane® and Kraton D-1101

Composition	Specification	Evatane®	Kraton D-1101
Molecular structure	–	Linear	Linear
Specific gravity	ASTM D792	0.92	0.94
Shore hardness (A)	ASTM D2240	82	71
Melt index	ASTM D1238	5–8	< 1
Elongation at break (%)	ASTM D412	700–1000	875
Tensile strength at break (MPa)	ASTM D412	33	31.8

Table 2 Properties and processing variables of binders used in the study

Properties/processing variables	VG 10	VG 30	PMB (E)	PMB (P)
Penetration, dmm	75	62	56	49
Softening point, °C	47	49	60	65
Penetration index	-1.01	-0.95	1.31	1.92
Viscosity @ 60 °C, Pa s	258	375	2120	6120
Storage stability, ΔSoft. point, °C	–	–	1.5	1.3
High temperature PG grade	PG 58-XX	PG 64-XX	PG 70-XX	PG 76-XX
True grade, intermediate temperature, °C	25.3	20.1	15.7	12.2
Mixing temperature, °C	–	–	180	190
Blending time, minutes	–	–	60	30
Shear rate, rpm	–	–	1500	600

is a linear SBS polymer in powder form that consists of different combinations made from blocks of polystyrene (31%) and polybutadiene of a very precise molecular weight (Sengoz and Isikyakar 2008b). These blocks are either sequentially polymerized from styrene and butadiene and/or coupled to produce a mixture of these chained blocks. Table 1 presents the properties of the polymers adopted in the study. The conventional properties along with the high temperature performance grade (PG) specification and true intermediate grade temperature for these binders are presented in Table 2. In this paper the polymer modified bitumen will be represented as PMB (E) and PMB (P) indicating modification with SBS and EVA.

3 Experimental

To characterize the flow behavior of bitumen rheologically, the following experiments were conducted.

1. Steady-shear viscosity test using dynamic-shear rheometer (DSR)

   Shear rate: 0–100 S^{-1}
   Test temperature: 20, 30, 40, 50, 60 and 70 °C.
   Spindle specification: 2mm dia. and 1mm gap for 20–40 °C.
   8mm dia. and 2mm gap for 50–70 °C.

2. Frequency sweep test using DSR
   Frequency: 0.1–20 Hz
   Test temperature: 20, 30, 40, 50, 60 and 70 °C.
   Spindle specification: 2mm dia. and 1mm gap for 20–40 °C.
   8mm dia. and 2mm gap for 50–70 °C.

4 Results and Analysis

4.1 Steady-shear viscosity and applicability Cox–Merz principle

Steady-shear viscosity test was conducted on the unmodified and modified asphalt binders, at six different temperatures. The complex viscosity at the corresponding temperatures were also evaluated for the given range of angular frequency. The suitability of the Cox–Merz principle can be evaluated using Fig. 1(a)–(d). The data for all the binders at temperatures of 50, 60 and 70 °C are shown. Similar results were obtained at other temperatures. In the figure SSV denotes the steady-shear viscosity while CV stands for the values of complex viscosity. Usually it is assumed that conventional binders such as VG 10 and VG 30 exhibits

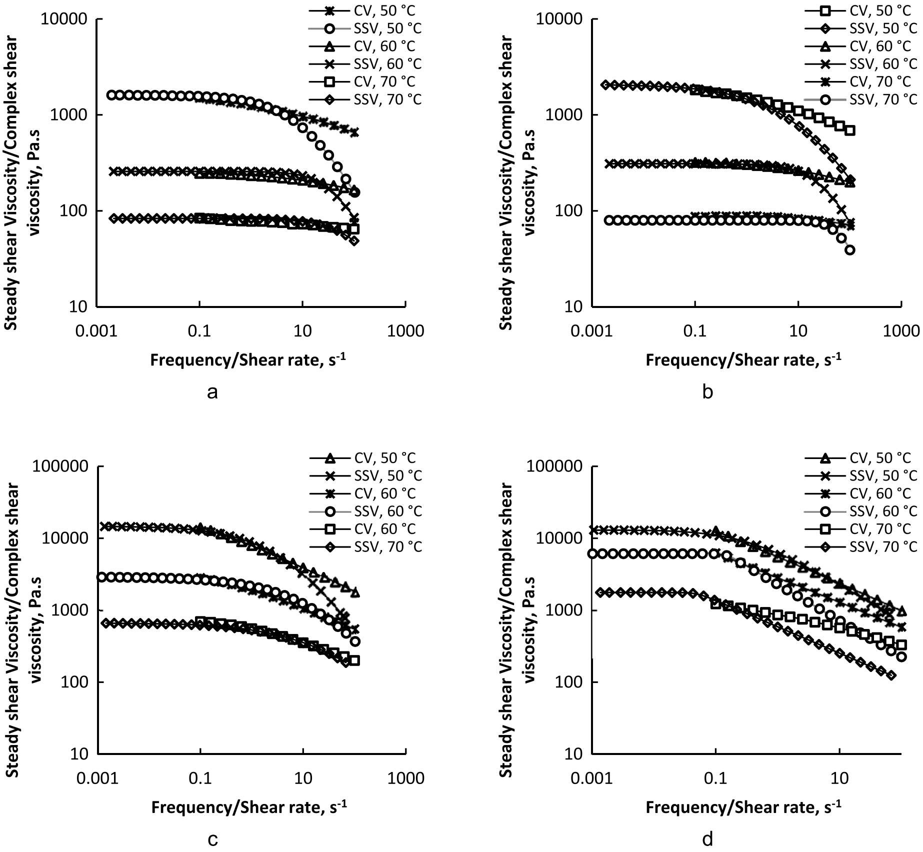

Fig. 1 Steady-shear and dynamic viscosity for (**a**) VG 10, (**b**) VG 30, (**c**) PMB (E) and (**d**) PMB (P)

Newtonian behavior above the softening point. However, in this study it was observed that VG 10 and VG 30 displayed some evidence of shear-thinning behavior, even at higher temperatures. As can be seen in Fig. 1(a), (b), at 50 °C there is a sharp reduction in the values of viscosity with increase in shear for the conventional binders. However, the critical shear rate, which can be defined as the point from where the reduction in viscosity starts, was different for the two binders. The reason behind this sharp decrease in viscosity can be the rapid loosening of the molecular networks in the binders with change in shear rate. Due to the higher stiffness of the binder at this temperature, it behaves as a shear-thinning fluid. Beyond 50 °C, the non-Newtonian behavior gradually diminishes with smooth transitions. This may be attributed to the dominance of Newtonian behavior.

The dominance of shear-thinning behavior was observed for modified binders at all the test temperatures. A sharp decrease in viscosity was observed for PMB (P) after 50 °C, beyond the critical shear rate. At higher temperatures, due to the melt of EVA crystallites, bitumen phase becomes more dominant as compared to the polymer phase. This results in the sudden reduction in viscosity, as was observed in the study. This argument is based on the work done by Airey (1997) on polymer modified binders. A study on the change of molecular level of different binders at different temperatures will throw more light on approving such behaviors. This paper, however, deals only with the physical characteristics of binder. The critical shear rates for modified binders were considerably lower as compared to the conventional binders at all the test temperatures. Shenoy et al. (1982) in his study has mentioned that the asphaltenes present in the asphalt binders are mainly responsible for the shear-thinning behavior, whereas the maltene fraction, which comprises resins, aromatics and saturates, governs the Newtonian flow (Shenoy et al. 1982). The rise of non-Newtonian behavior of PMB may be attributed to the decrease in effective maltene fraction, used by polymers for dispersion.

It can be seen that SSV is almost same as the CV at lower frequency/shear rate and deviates at the end giving lower values. This deviation was more prominent at higher temperatures mostly for modified binders. This indicates that the Cox–Merz principle is valid in the zero-shear viscosity domain.

4.2 Evaluation of complex viscosity master curves

The complex viscosity master curves for all he binders at six different reference temperatures have been presented in Fig. 2(a)–(d). It was found that smooth master curves can be obtained at all the temperatures and for different binders. This indicates that ZSV can be suitably used for construction of master curves. In general complex viscosity decreases with increase in frequency and temperature. The difference in the values at consecutive temperatures is more prominent at lower frequency as compared to higher frequency levels. It can also be seen that the individual curves at different temperatures are close to each other for modified binders as compared to conventional binders. This indicates that polymer modifications makes the binder less temperature susceptible.

4.3 Modeling the complex viscosity master curve

The complex modulus master curves for all the binders were simulated using the cross model. The curve fit for VG 10 and PMB (E) are shown in Fig. 3(a)–(b) for 30 and 50 °C. A similar fit was obtained at other temperatures and for all the asphalt binders. Those curves are not shown here for brevity. As can be seen, the measured and modeled data are in good agreement with each other. This indicates that the Cross model can be successfully used

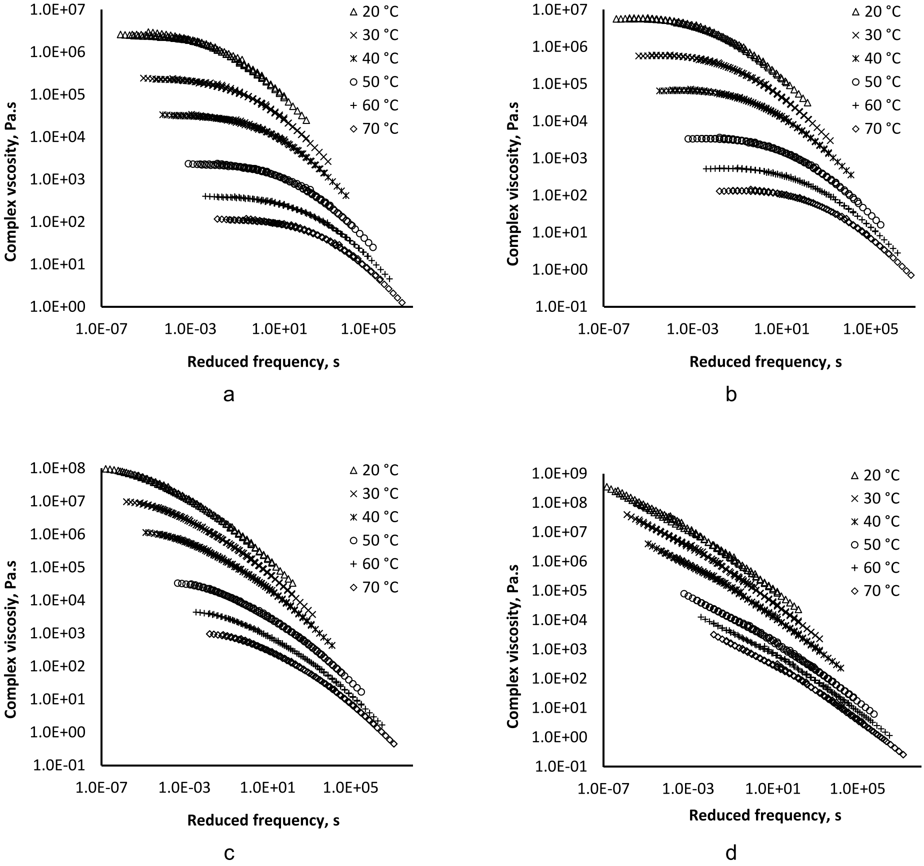

Fig. 2 Complex viscosity master curves for (**a**) VG 10, (**b**) VG 30, (**c**) PMB (E) and (**d**) PMB (P)

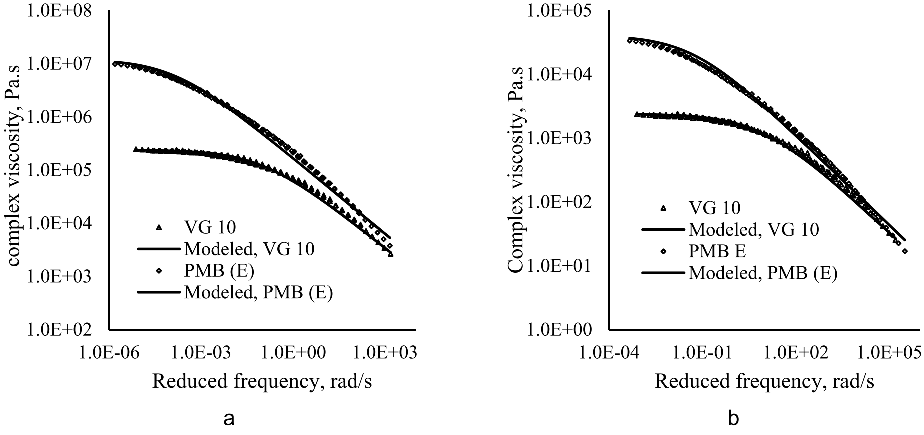

Fig. 3 Cross model fit at (**a**) 30 °C and (**b**) 50 °C

Table 3 Cross model parameters at different temperatures

Temperature (°C)	Binder	η_0 (Pa s)	λ (s)	m	Critical shear rate (s^{-1})
20	VG 10	2.66E+06	1.05E+02	4.39E-01	9.50E-03
	VG 30	6.23E+06	3.25E+02	4.43E-01	3.07E-03
	PMB (E)	1.21E+08	1.78E+05	4.06E-01	5.62E-06
	PMB (P)	9.18E+08	2.46E+07	4.30E-01	4.07E-08
30	VG 10	2.40E+05	9.68E+00	4.11E-01	1.03E-01
	VG 30	6.28E+05	3.84E+01	4.32E-01	2.60E-02
	PMB (E)	1.21E+07	1.52E+04	4.07E-01	6.59E-05
	PMB (P)	1.48E+08	9.64E+06	4.15E-01	1.04E-07
40	VG 10	3.35E+04	1.11E+00	4.10E-01	9.03E-01
	VG 30	7.21E+04	3.60E+00	4.37E-01	2.77E-01
	PMB (E)	1.42E+06	1.91E+03	4.07E-01	5.24E-04
	PMB (P)	2.19E+07	3.71E+06	4.02E-01	2.69E-07
50	VG 10	2.40E+03	1.19E-01	4.13E-01	8.37E+00
	VG 30	3.54E+03	1.79E-01	4.41E-01	5.58E+00
	PMB (E)	4.10E+04	4.87E+01	4.07E-01	2.05E-02
	PMB (P)	4.96E+05	1.06E+05	4.01E-01	9.45E-06
60	VG 10	4.09E+02	1.87E-02	4.03E-01	5.35E+01
	VG 30	5.63E+02	2.72E-02	4.36E-01	3.67E+01
	PMB (E)	5.45E+03	7.00E+00	4.06E-01	1.43E-01
	PMB (P)	7.05E+04	1.44E+04	3.88E-01	6.94E-05
70	VG 10	1.16E+02	4.48E-03	4.16E-01	2.23E+02
	VG 30	1.41E+02	6.66E-03	4.39E-01	1.50E+02
	PMB (E)	1.19E+03	1.40E+00	4.01E-01	7.16E-01
	PMB (P)	1.57E+04	2.37E+03	3.95E-01	4.22E-04

to simulate the complex viscosity master curves for asphalt binders at any given reference temperatures.

Table 3 presents the model parameters obtained at different reference temperatures for each binder. The critical shear rate, which indicates the onset of the shear-thinning behavior is also presented. It is the inverse value of λ obtained using the Cross model. In general the value of η_0 and λ decreases with increase in temperature. The change in the value of m is not very significant with change in temperature. The value ranges from 0.38–0.44 for all the asphalt binders at different temperatures. The critical shear rate increased with increase in temperature for all the binders. This may be attributed to the flow of binder at higher temperatures which shifts the behavior towards a Newtonian like fluid. The critical shear rate was lower for the modified binders as compared to viscosity graded binders indicating higher shear-thinning behavior induced by the addition of polymers.

The variation of ZSV and the critical shear rate ($\dot{\gamma}_c$) with temperatures was also assessed in this study. This variation can be seen in Fig. 4(a)–(b). Both ZSV and $\dot{\gamma}_c$ followed an exponential law with change in temperature (with a R^2 value greater than 0.93) which can be mathematically written as follows:

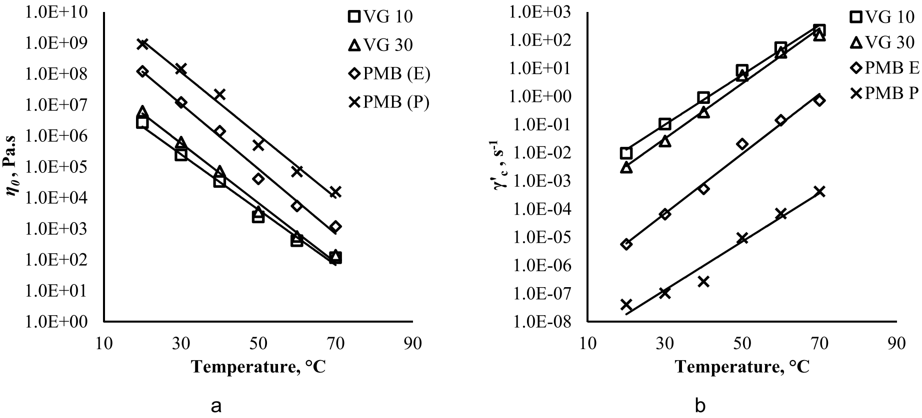

Fig. 4 Variation of ZSV (**a**) and critical shear rate (**b**) with temperature

Table 4 Values of A, B, P and Q

Binder	A	B	P	Q
VG 10	0.0002	0.204	1.00E+08	0.206
VG 30	4.00E-05	0.223	4.00E+08	0.222
PMB (E)	5.00E-08	0.244	1.00E+10	0.242
PMB (P)	4.00E-10	0.198	1.00E+11	0.233

$$\eta_{0,T} = A \cdot e^{-B.T}, \tag{7}$$

$$\dot{\gamma}_{c,T} = P \cdot e^{Q.T}. \tag{8}$$

The values of A, B, P and Q are binder and temperature dependent and are presented in Table 4.

As was seen earlier, the ratio of ZSV at temperature T and temperature T_{ref} is taken as the shift factor for the construction of master curves. So, from Eq. (7) we obtain

$$\frac{\eta_{0,T}}{\eta_{0,T_r}} = e^{-B\cdot(T-T_{ref})}, \tag{9}$$

$$a_T = e^{-B\cdot(T-T_{ref})}. \tag{10}$$

As can be seen from Table 4 the values of the parameters B and Q are almost the same (equal till two significant digits) for all the binders except for PMB (P). This may be attributed to the nonlinearity associated with the plastomeric modified binder. For other binders, if $B \approx Q$, we obtain

$$\eta_{0,T} \cdot \dot{\gamma}_c = A \cdot P \cdot e^{T\cdot(Q-B)}, \tag{11}$$

$$\dot{\gamma}_c = \frac{A \cdot P}{\eta_{0,T}}. \tag{12}$$

This shows that the critical shear rate can be linked with ZSV using a simple linear relationship as above. Higher will be the value of ZSV; lower will be the critical shear rate. This seems to be justified, as higher ZSV indicates higher stiffness and thus a lower value of $\dot{\gamma}_c$, as was seen for PMB (P).

5 Conclusions

From the study conducted, the following conclusions can be derived:

(a) Both conventional and polymer modified asphalt binders exhibited shear-thinning behavior between the temperature range of 20–70 °C. The dynamic- and steady-shear viscosity values were almost equal at low-shear/zero-shear rates. At higher shear rates the values in the dynamic-shear domain were found to be higher than the steady-shear domain. The applicability of the Cox–Merz principle can be appreciated in the ZSV regime.

(b) Smooth complex viscosity master curves for all the binders indicated that ZSV can be successfully used as appropriate shift factors. In general complex viscosity decreases with increase in frequency and temperature. The difference in the values at consecutive temperatures is more prominent at lower frequency as compared to higher frequency levels.

(c) Cross model was found to be appropriate in simulating the complex viscosity master curves at different temperatures for all the binders. The value of η_0 and λ decreased with the increase in temperature. The critical shear rate increased with increase in temperature for all the binders. The critical shear rate was lower for the modified binders as compared to viscosity graded binders indicating higher shear-thinning behavior induced by the addition of polymers. Both ZSV and $\dot{\gamma}_c$ followed an exponential law with change in temperature (with a R^2 value greater than 0.93). A simple equation has been proposed for calculating the shift factor, which is a function of temperature and type of binder. The critical shear rate can be inversely related to the ZSV using a simple linear equation.

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