Construction and Building Materials 37 (2012) 716–722

Contents lists available at SciVerse ScienceDirect

Construction and Building Materials

journal homepage: www.elsevier.com/locate/conbuildmat

Applicability of the Cox–Merz relationship for asphalt binder

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HIGHLIGHTS

- ▶ The Cox–Merz relationship is followed in the shear-thinning range.
- ▶ The Cox–Merz relationship is not always followed in the zero-shear-rate-limiting viscosity region.
- ▶ The criterion to check whether the Cox–Merz relationship is suitable for asphalt binder or not was established.
- ▶ The Cox–Merz relationship is useful for obtaining the viscosity function of asphalt binder.
- ▶ Both of the Cross model and the Carreau model can be used to analyze the viscosity of asphalt binder.

ARTICLE INFO

Article history:
Received 10 January 2012
Received in revised form 7 June 2012
Accepted 21 July 2012
Available online 13 September 2012

Keywords:
Asphalt binder
The Cox–Merz relationship
Complex viscosity
Steady-state shear viscosity

ABSTRACT

Asphalt binder has a critical effect on the performance of asphalt mixtures. Thus, asphalt binder is of great concern to pavement engineers and researchers. Usually, asphalt binder is studied in dynamic space and steady-state space separately, and the property indices are researched separately. As researchers delved deeper, they found that it is important to combine the dynamic properties and steady-state properties, especially for establishing rheological curves and constitutive equations. The Cox–Merz relationship can connect the properties in dynamic space to those in steady-state space. This relationship is widely used in polymer system and shows good results. This study focused on the applicability of the Cox–Merz relationship as it pertains to asphalt binder. The Cox–Merz relationship was applied to six neat asphalt binders. Then, the applicability of the Cox–Merz relationship for asphalt binder was studied and the criterion to verify whether the Cox–Merz relationship is followed or not was established. Finally the viscosity functions when the Cox–Merz relationship used were studied, and the Cross model and the Carreau model were compared. The results show that the Cox–Merz relationship is followed in the shear-thinning region and not always followed in the zero-shear-rate-limiting viscosity region, and it is useful when obtaining the viscosity function.

1. Introduction

Asphalt binder is one of the two principal constituents of asphalt mixtures, and its mechanical properties are very important to road performance. Thus, asphalt binder is of great concern to researchers and is the focus of many published papers. The mechanical properties of asphalt binder can be divided into dynamic property and steady-state property, both of which can be obtained by measurements taken using the Dynamic Shear Rheometer (DSR). Usually, these two kinds of properties are studied separately. However, in order to formulate the rheological curves and constitutive equations, or obtain the dynamic property from steady-state property or vice versa, it is important to relate the dynamic property to the steady-state property and study the transformational relation between them.

Earlier investigations of rheological properties (at least for polymer melts) have shown that the data under dynamic conditions can be related to those data obtained under steady-state conditions within certain ranges of shear rates and frequencies. There are some methods for correlating dynamic and steady-state data [1–4]. Among the methods, the Cox–Merz relationship is one of the most widely used methods.

The Cox–Merz relationship is an empirical relationship that is of great use in rheology. Cox and Merz are the first researchers to observe that [1], for many polymeric systems, a correspondence occurs between steady-state viscosity, η, at some shear rate, and the magnitude of $|\eta^*|$, at an angular frequency, ω, which is equal to that same shear rate (Eq. (1)).

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

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http://dx.doi.org/10.1016/j.conbuildmat.2012.07.020

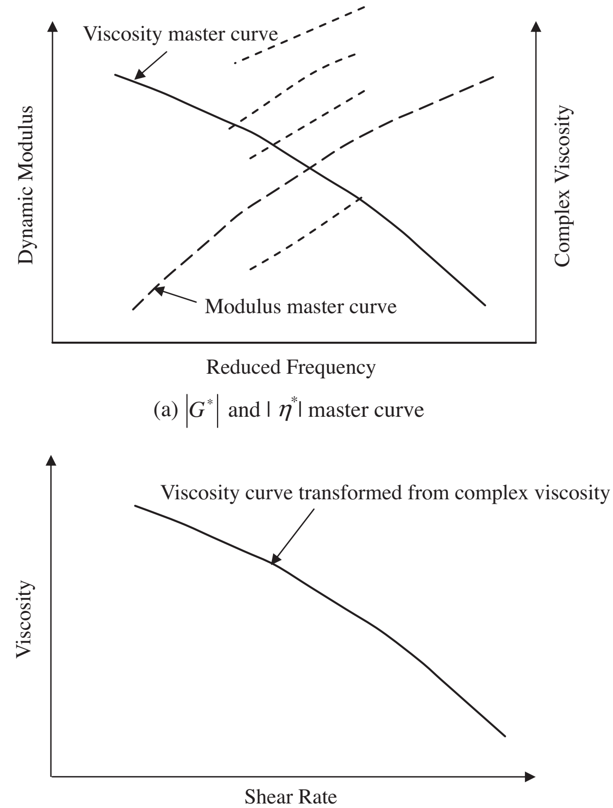
This relationship is widely used in polymeric systems (see Refs. [5,6]). Tanaka and Edwards [7] applied the Cox–Merz relationship to polymers that have different networks and found that the relationship does not hold for an internal flow in reversible networks. Because the Cox–Merz relationship is not suitable for all kinds of materials, some researchers modified or extended the Cox–Merz relationship and obtained good results. Doraiswamy et al. [8] proposed a relationship between the steady-state viscosity and the complex viscosity, which is analogous to the Cox–Merz relationship, by introducing a new parameter, $\gamma_m\omega$, which is termed the effective shear rate in dynamic experiments. Gleissle and Hochstein [9] derived a modified Cox–Merz rule, whereby the shear stress function and complex modulus can be expressed as a function of the solid concentration using the parameters in this modified Cox–Merz relationship.

The Cox–Merz relationship is not as widely used for asphalt binder as it is for polymer systems, but it has researchers' attention. Huh et al. [10] established the shear complex modulus equation for multiphase viscous flow based on the Cross model and the Cox–Merz relationship. Stastna et al. [11] plotted the shear viscosity and the magnitude of the complex viscosity in one graph to check the validity of the Cox–Merz relationship. Polacco et al. [12] found that the Cox–Merz relationship fails to account for the shear-thickening behavior of modified asphalt. Shenoy [13] used the Cox–Merz relationship to establish a theoretical relationship between the fundamental rheological properties obtained from the DSR and the material volumetric flow rate (MVR) obtained from the flow measurement device (FMD), and their results show that the unified curves of the viscoelastic properties of polymer-modified asphalts can be obtained within a determined temperature range. Pérez-Lepe et al. [14,15] applied the Cox–Merz rule to different modified asphalt binder. The results show that some binders hold the Cox–Merz rule and some does not. Partal et al. [16] compared the steady-state viscosity to the complex viscosity for all the studied system and got the results that the studied unmodified bitumens follow the Cox–Merz rule, but the synthetic binders do not. Shan et al. [17] established a flow curve by combining the master curve from dynamic shear test and the data from steady-state flow test using the Cox–Merz relationship, and their results show that the relationship is suitable for the studied asphalt binders. Witczak et al. [18,19] successfully used the Cox–Merz rule in developing the Witczak–Bonaquist model by which the complex modulus can be calculated using viscosity. Then the model was revised by taking into account the loading frequency and temperature.

Analysis of the published researches, it can be seen that most of the researchers use the Cox–Merz relationship directly without proving its validity. The authors have already studied the validity of the Cox–Merz relationship (cf. [17]), but there were only three binders under one temperature. Even Witczak established a model for viscosity and complex modulus, the viscosity was obtained from conventional and Superpave consistency tests not DSR. Besides that, there is no criterion to verify the Cox–Merz relationship for asphalt binder of which both of the dynamic shear viscosity and the steady-state shear viscosity are obtained by DSR. To sum up, more researches should be carried out to study the validity of the Cox–Merz relationship for asphalt binders, and to dig the suitable or unsuitable conditions for the relationship.

2. Proposed methodology

The methodology used to analyze the Cox–Merz relationship can be divided into four steps. The first step is to establish the dynamic shear modulus ($|G^*|$) master curve. Using the frequency sweep test, the dynamic modulus as a function of frequency at different temperatures can be obtained, as shown in Fig. 1a. Then, the



(a) $|G^*|$ and $|\eta^*|$ master curve

(b) After applying the Cox-Merz relationship

Fig. 1. Schematic of transforming the data from dynamic space to steady-state space.

curves at different temperatures can be combined into a single functional relationship by using the time–temperature superposition principle (Fig. 1a).

$$|\eta^*| = |G^*|/\omega \tag{2}$$

In terms of the dynamic shear property of asphalt binder, the dynamic shear modulus is the main material property used to evaluate the material characteristics. However, for the Cox–Merz relationship, dynamic viscosity ($|\eta^*|$) is one of the main parameters. So the second step is to obtain the dynamic viscosity master curve. A simple equation relates $|G^*|$ and $|\eta^*|$, that is, $|\eta^*|$ equals $|G^*|$ divided by the angle frequency, as shown in Eq. (2). By using Eq. (2), each $|G^*|$ data point can be transformed to $|\eta^*|$, so the $|\eta^*|$ master curve can be obtained using the $|G^*|$ master curve, as shown in Fig. 1a.

The third step is to transform the dynamic shear viscosity in dynamic space to the viscosity in the steady-state space. Using the Cox–Merz relationship (Eq. (1)), the steady-state shear curve can be obtained by changing the *x*-axis from reduced frequency to reduced angular frequency then to shear rate and the *y*-axis from dynamic viscosity to steady-state shear viscosity, as shown in Fig. 1b.

The last step is to compare the curve obtained from the third step to the curve obtained in the steady-state flow test. After the above four steps, the Cox–Merz relationship for asphalt binder can be validated, the suitable and unsuitable conditions can be analyzed, and the criterion to verify this relationship can be established.

3. Materials and test methods

Three PG 70-22 asphalt binders (A–C), two PG 64-22 asphalt binders (D and E) and one PG 58-22 asphalt binder (F) were selected for this study. For the remainder of this paper, these materials are labeled as generic binders A–F. All are standard, unmodified asphalt materials. These binders were all aged in a rolling thin film oven (i.e., RTFO-aged) prior to testing to simulate the effects of mixing and compaction. Laboratory testing was performed using a TA Instruments AR-G2 Rheometer. Two types of test were conducted: the frequency sweep test and the steady-state flow test. All tests were conducted with an 8 mm diameter parallel plate geometry and 2 mm gap setting. Two replicates were performed for each test condition. In the case of good repeatability, the average of the two replicates was used for analysis. Good repeatability was assumed when the difference between the two replicates was smaller than 10% of the mean. In the case of larger differences, a third replicate was tested. In order to avoid the adhesion problem between the plate and the binder, the plates ware observed after each test condition. If the plate is not covered by binders which mean adhesion problem occurring, the data should be deleted and the test will be conducted again.

3.1. Frequency sweep test

Frequency sweep tests were conducted for frequencies between 0.1 Hz and 30 Hz and for temperatures of 10, 16, 19, 22, 25, 30 and 40 °C. This combination of temperatures and frequencies ensures sufficient overlap in the material responses so that the data can be shifted horizontally to obtain master curves of the key properties. Prior to performing these frequency sweep tests, a stress sweep was conducted to ensure that all frequency sweep tests were conducted in the linear range.

3.2. Steady-state flow test

In the steady-state flow test, the specimen was sheared under the shear rates that were stepped up gradually from 1×10^{-5} to 2 γ/s. Each rate was maintained until steady-state flow was achieved. In order to analyze the Cox–Merz relationship at different temperatures, four test temperatures were chosen: 15 °C, 20 °C, 25 °C and 30 °C.

4. Results and analysis

4.1. Selection of the valid data in the steady-state flow test

For the oscillation series, the loading applied during the oscillation was small enough to avoid bringing damage into the specimen, so the oscillation series can represent the viscosity changing law when the asphalt binders are intact. But, for the steady-state flow series, no step was taken to avoid damage. In the steady-state flow test, the loading was put on the specimen until it is totally broken, and the data was collected with a fixed frequency during the whole process. For analyzing the viscosity curve or viscosity equation, etc., the viscosity or others values should be obtained when the specimen is intact. So the data which was collected after damage appeared in the specimen should be deleted.

The curve of shear stress versus shear rate is used to delete the invalid data. As shown in Fig. 2, at the beginning of the test, shear stress increases with an increase in shear rate. After the shear stress attains to some value, it drops, and the shear stress starts to decrease with an increase in shear rate. This occurrence indicates that damage appears at this point, and the ability of the specimen to bear loading reduces. Note that, although the results for only a single binder are shown, the results for the other binders are similar. So only the data before the turning point was used for this study.

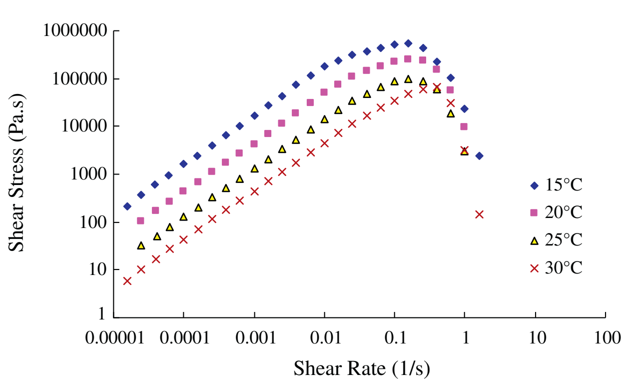


Fig. 2. The curve of shear stress versus shear rate of binder E.

4.2. Verifying the Cox–Merz relationship for asphalt binders

Figs. 3–6 show the results of applying the Cox–Merz relationship to some of the studied asphalt binders at four different temperatures (15 °C, 20 °C, 25 °C and 30 °C). For the similarity of the results between the binders, not all the curves of the binders are shown. All the figures are in log–log scale. In order to see the details of the curves clearly, the *x*-axis starts from 1×10^{-5} (1/s), and the *y*-axis starts from 1×10^{3} (Pa.s).

For the steady-state flow series, Figs. 3–6 show that all the studied binders, in the studied temperature range (except binder F at 30 °C), exhibit a Newtonian behavior over a relatively wide range of shear rate. Nevertheless, as the shear rate is raised, a decrease in viscosity, which corresponds to a shear-thinning behavior, can also be observed. The zero-shear-rate-limiting viscosity they exhibit decreases with increase in temperature. For the oscillation series, the binders do not exhibit Newtonian phase at 15 °C and 20 °C. With increase in temperature, some binders show obvious Newtonian behavior, such as binder A at 25 °C and 30 °C.

Figs. 3–6 show that the Cox–Merz relationship is followed at shear rates outside the zero-shear-rate-limiting viscosity region for almost any of the binders and temperatures studied. Although the superposition conditions for binder A are not so good as other binders, the trend of the steady-state flow series and that of the oscillation series are similar and the two curves superpose to a certain extent. So it is believed that the Cox–Merz relationship is suitable for binder A outside the zero-shear-rate-limiting viscosity region. In other words, it is believed that the Cox–Merz relationship is followed for the studied asphalt binders in the shear-thinning region. In the zero-shear-rate-limiting viscosity region, some binders hold the Cox–Merz relationship and some do not. That depends on whether the oscillation series show Newtonian behavior or not. For example, for binder A, C and E at 30 °C, the oscillation series show Newtonian behavior just like the steady-state flow series do. The steady-state flow series and the oscillation series superpose in the studied range of shear rate. So it can be concluded that the Cox–Merz relationship is suitable at this condition.

To sum up, the Cox–Merz relationship does apply in the shear-thinning region. And whether it is followed in the zero-shear-rate-limiting viscosity region depends on binder type and studied temperature.

4.3. Establishment of the criterion to decide whether the Cox–Merz relationship is suitable or not

From the above analysis, it is found that the Cox–Merz relationship is followed in the shear-thinning region. So the only work to decide whether it is suitable for asphalt binder is to checking whether the relationship is followed in the zero-shear-rate-limiting viscosity region.

In the oscillation domain, the dynamic modulus master curve of asphalt binder can be expressed by CAM model [20] as shown in Eq. (3). Because the complex modulus equals to complex viscosity multiply by angular frequency, as shown in Eq. (2), complex viscosity can be expressed using Eq. (4) by combining Eqs. (2) and (3).

$$G^{*} = \frac{G_g^{*}}{[1 + (\omega_c/\omega')^{k}]^{m_e/k}} \tag{3}$$

where G_g^{*} is the glassy modulus (Pa), ω_c is the cross over frequency (rad/s), ω' is the angular frequency (rad/s), and k and m_e are the shape factor.

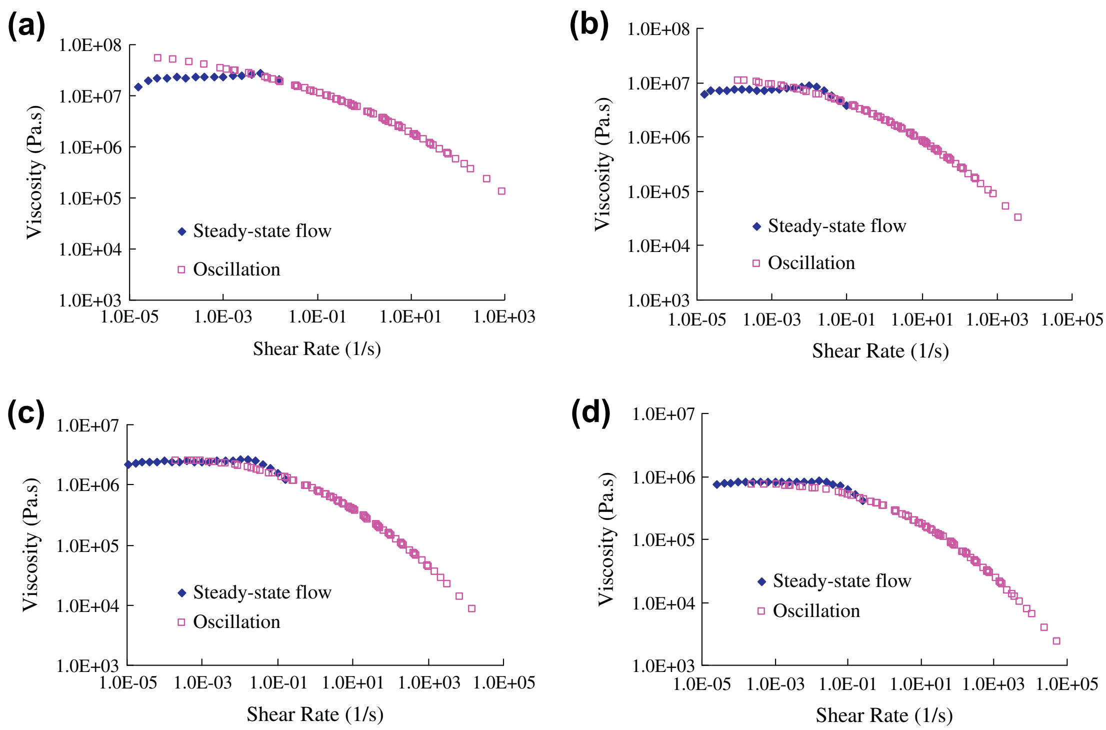


Fig. 3. Applicability of Cox–Merz relationship of binder A: (a) 15 °C, (b) 20 °C, (c) 25 °C and (d) 30 °C.

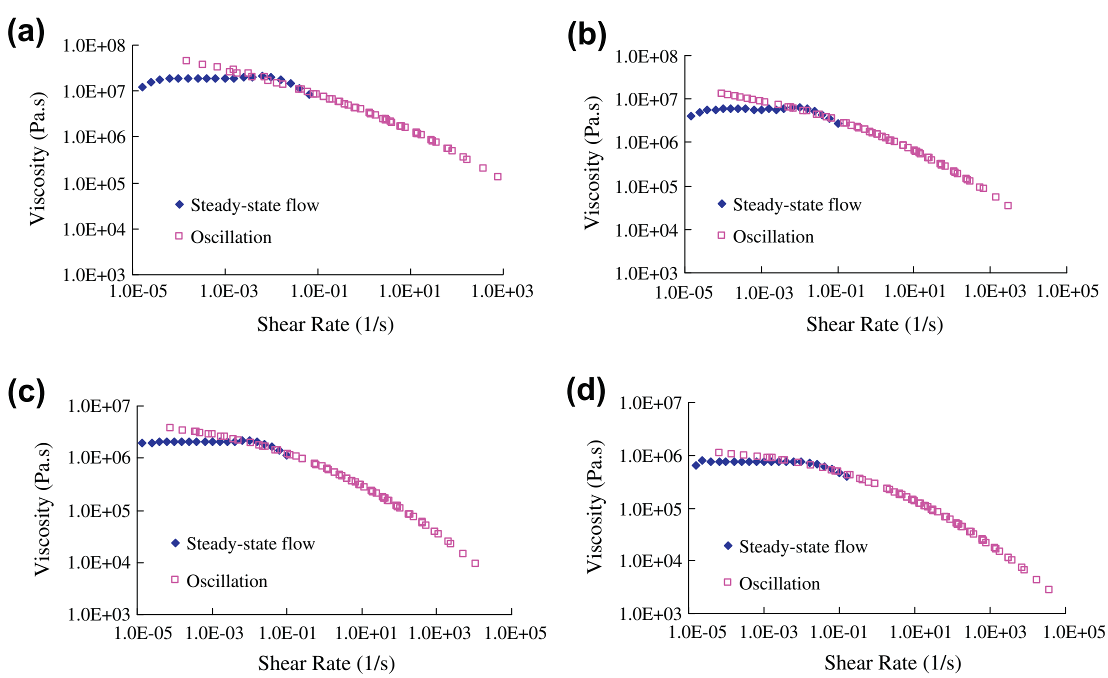


Fig. 4. Applicability of Cox–Merz relationship of binder C: (a) 15 °C, (b) 20 °C, (c) 25 °C and (d) 30 °C.

$$|\eta^*| = \frac{|G_g^*|}{\omega'[1+(\omega_c/\omega')^k]^{m_e/k}} \tag{4}$$

For the steady-state flow test, the viscosity is almost the same in log–log scale in the zero-shear-rate-limiting viscosity region. It is assumed that in that region the shear rate is from $\dot{\gamma}_1$ to $\dot{\gamma}_2$.Thus if the Cox–Merz relationship is followed in that region, the complex viscosity need to be constant in the region where the angular frequency is from ω'_1 (equals to $\dot{\gamma}_1$) to ω'_2 (equals to $\dot{\gamma}_2$). That is $|\eta^*(\omega'_1)|$ equals to $|\eta^*(\omega'_2)|$, and Eq. (5) can be established. After simplifying Eq. (5), Eq. (6) can be obtained. And the whole part in the left side of the equal mark is expressed as C.

Eq. (6) is the criterion to check the Cox–Merz relationship for asphalt binder. When the complex modulus master curve is

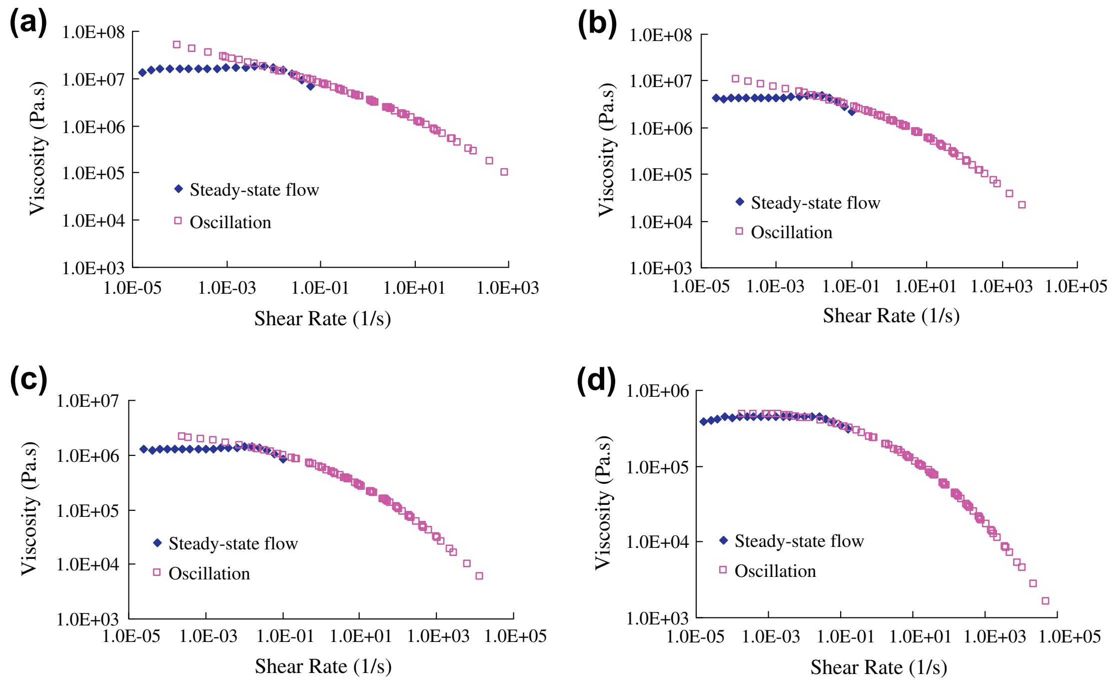


Fig. 5. Applicability of Cox–Merz relationship of binder E: (a) 15 °C, (b) 20 °C, (c) 25 °C and (d) 30 °C.

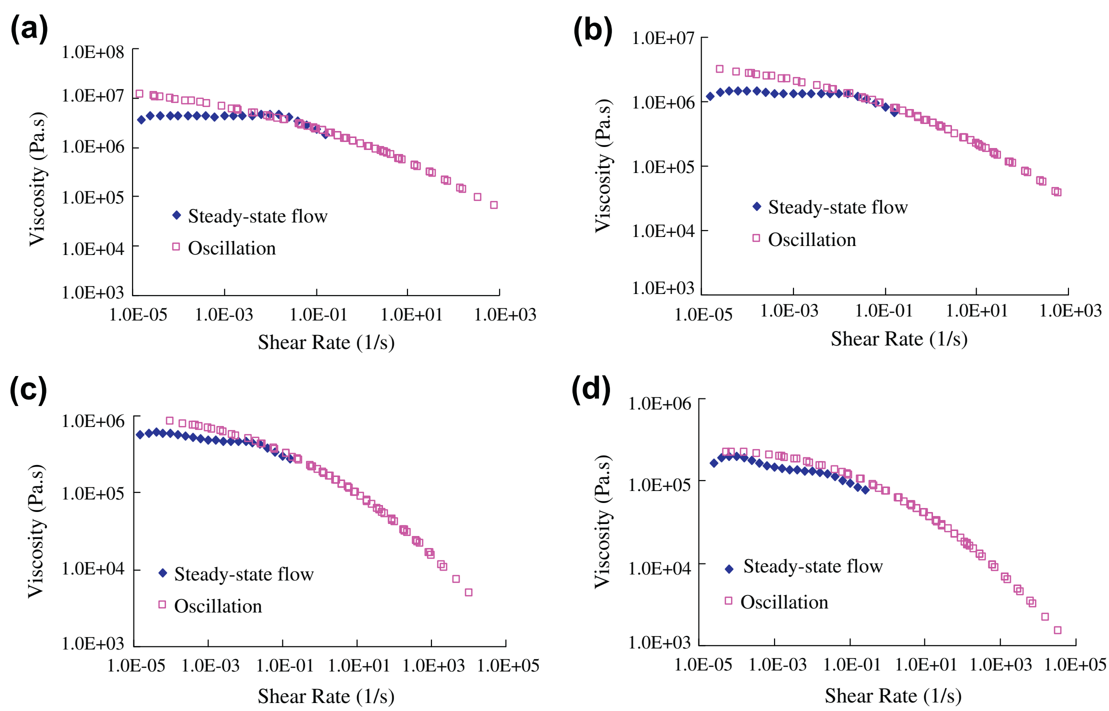


Fig. 6. Applicability of Cox–Merz relationship of binder F: (a) 15 °C, (b) 20 °C, (c) 25 °C and (d) 30 °C.

obtained, three parameters which are ω_c, m_e and k in Eq. (6) can be obtained. After ω'_1 and ω'_2 were chosen, the criterion can be used to check whether the relationship is suitable or not.

$$\log\frac{|G^*_g|}{\omega'_1[1+(\omega_c/\omega'_1)^k]^{m_e/k}} = \log\frac{|G^*_g|}{\omega'_2[1+(\omega_c/\omega'_2)^k]^{m_e/k}} \quad (5)$$

$$C = \frac{\log\left\{\omega'_1\left[1+(\omega_c/\omega'_1)^k\right]^{m_e/k}\right\}}{\log\left\{\omega'_2\left[1+(\omega_c/\omega'_2)^k\right]^{m_e/k}\right\}} = 1 \quad (6)$$

To validate this criterion, the C values of all the studied binders at different temperatures were calculated, and the results are summarized in Table 1. Analyzing the steady-state flow test of all the binders, ω'_1 equals to 10^{-4} and ω'_2 equals to 10^{-2} except binder F at 25 °C and 30 °C. For that condition, ω'_2 equals to 10^{-3}.

It can be seen from Table 1 that C value gets closer to 1 with increase in temperature. It means that the possibility that the Cox–Merz relationship is followed increases with increase in temperature. Comparing Table 1 to the figures (Figs. 3–6), it can be found that for the same binder, the more larger C value is, the more closer between the steady-state flow series to the oscillation series in the

Table 1
C values of all the studied binders.

Binder	*C*			
	15 °C	20 °C	25 °C	30 °C
A	0.748	0.907	0.971	0.989
B	0.708	0.859	0.919	0.954
C	0.784	0.870	0.922	0.954
D	0.716	0.874	0.967	0.993
E	0.668	0.806	0.927	0.985
F	0.893	0.931	0.956	0.977

zero-shear-rate-limiting region. It also shows that the above criterion is suitable to check the applicability of the Cox–Merz relationship for asphalt binder.

4.4. Analyzing the viscosity function of asphalt binders when using the Cox–Merz relationship

The viscosity function is important to study the rheological properties of asphalt binder. The most often used viscosity functions of asphalt binder are the Cross model and the Carreau model, and they are shown in Eqs. (7) and (8), respectively. Note that the formats of these two models are different in different references; the formats of the models used in this paper are taken from the TA instrument software.

$$\frac{\eta - \eta_\infty}{\eta_0 - \eta_\infty} = \frac{1}{\left[1 + (c\dot{\gamma})^d\right]} \tag{7}$$

$$\frac{\eta - \eta_\infty}{\eta_0 - \eta_\infty} = \frac{1}{\left[1 + (c\dot{\gamma})^2\right]^{d/2}} \tag{8}$$

where η_∞ is the infinite viscosity (Pa·s), η_0 is the zero shear viscosity (Pa·s), $\dot{\gamma}$ is the shear rate, and *c* and *d* are the regression coefficients.

An easy way to obtain the viscosity function is to use the model to fit the test data; then the coefficients can be obtained, and the function can be obtained. It can be seen from Figs. 3–6 that for the steady-state flow series the range of the shear rate is about from 1×10^{-5} to 1×10^{-1}. But the oscillation series can reach the shear rate of 1×10^3. In other words, if the Cox–Merz relationship is applied, the data used to get the viscosity function can cover a relatively large shear rate range, and the results could be more reliable.

From the above analysis, it is concluded that the Cox–Merz relationship is followed in the shear-thinning region and not always followed in the zero-shear-rate-limiting viscosity region. So all the valid data in the steady-state flow series and only the data in the shear-thinning region of the oscillation series were used to obtain the viscosity function.

The *R*-square values of all the results are summarized in Table 2. It can be seen from Table 2 that most of the *R*-square values are larger than 0.9, except binder D at 15 °C and Carreau model of binder F at 30 °C. But the *R*-square values of them are still larger than 0.87. It means that both of the Cross model and the Carreau model is suitable to the studied asphalt binder. And which model is much better is dependent on the test condition and the studied binders. It can also be illustrated that the Cox–Merz relationship is useful when analyzing the viscosity function of asphalt binder.

Table 2
R-square values of the regression results.

Binders	Temperature (°C)	Cross	Carreau
A	15	0.948	0.947
	20	0.961	0.962
	25	0.976	0.944
	30	0.976	0.948
B	15	0.926	0.958
	20	0.956	0.975
	25	0.966	0.966
	30	0.973	0.974
C	15	0.938	0.957
	20	0.950	0.974
	25	0.985	0.977
	30	0.990	0.964
D	15	0.886	0.871
	20	0.955	0.943
	25	0.986	0.967
	30	0.989	0.964
E	15	0.908	0.909
	20	0.969	0.949
	25	0.988	0.961
	30	0.989	0.955
F	15	0.944	0.964
	20	0.979	0.975
	15	0.994	0.956
	30	0.965	0.898

5. Conclusions and recommendation

This paper studies the Cox–Merz relationship as it applies to asphalt binder. The applicability of the Cox–Merz relationship for asphalt binder is verified. Next, the criterion to decide whether the Cox–Merz relationship is suitable for asphalt binder is established. Finally, the viscosity functions when using the Cox–Merz relationship are studied, and the Cross model and the Carreau model are compared. Based on the results presented, several conclusions can be drawn:

1. The Cox–Merz relationship is followed in the shear-thinning region and not always followed in the zero-shear-rate-limiting viscosity region, as found from the research method implemented in this paper.
2. The criterion established in this research is suitable to verify whether the Cox–Merz relationship is followed.
3. The Cox–Merz relationship is useful for obtaining the viscosity function. Both of the Cross model and the Carreau model can be used to analyze the viscosity of asphalt binder.

Although based on six kinds of neat asphalt binder, it is recommended to use the Cox–Merz relationship when using DSR data to obtain viscosity function.

Acknowledgement

This study was supported by National Natural Science Foundation of China (51108138).

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