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# CHARACTERISATION OF POLYMER-MODIFIED ASPHALT BINDERS USING THE DYNAMIC SHEAR RHEOMETER

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**Abstract**

Bitumen is a viscoelastic substance, and it is widely acknowledged that its mechanical behaviour is influenced by both loading time and temperature. The performance of asphalt mixtures is significantly affected by the behaviour of bitumen, which in turn impacts the service life of flexible pavements. The dynamic shear rheometer is a commonly used tool for assessing the physical and rheological properties of both unmodified and modified asphalt binder (PMB). In this investigation, three binders were chosen: 40/60 penetration unmodified bitumen, a hard styrene butadiene styrene (SBS) polymer-modified binder, and a softer SBS polymer-modified binder. These binders were analysed using penetration, softening point, and dynamic shear rheometer frequency sweep tests. The dynamic shear rheometer data revealed that the complex modulus master curves highlighted the stiffening effect of SBS polymer modifiers, which became more evident at low frequencies and high temperatures. Regarding the phase angle master curves, the results indicated that both PMBs experienced a notable reduction in phase angle at low frequencies and high temperatures, resulting in a significant improvement in the elasticity of the bitumen. Finally, the black diagram results suggested that both PMBs, which contained SBS modifiers, displayed a plateau region where the phase angle remained constant and shifted towards lower values.

*Keywords:* dynamic shear rheometer; rheological characterisation; polymer-modified asphalt; bituminous binders.

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## 1. Introduction

Asphalt, or bituminous mixture, consists of bitumen and mineral aggregate. This type of mixture is frequently used in the construction of highway pavement layers, parking lots, and pedestrian pathways. Once the asphalt mixture is compacted to the desired air void content, the mineral aggregate, with its various size fractions, serves as the structural framework that provides strength. Bitumen, or binder, functions as the adhesive that binds the aggregate particles together, enhancing the performance of the mixture [1–3]. Bituminous binder is a viscoelastic material, and the distinctive behaviour of viscoelastic substances is influenced by their mechanical response to loading and temperature. As such, the physical properties of the binder are crucial for a comprehensive understanding of asphalt mixtures [4–6]. At the same time, an asphalt mixture functions by creating a framework of interlocking aggregate particles. The movement between these particles is constrained by the bitumen or binder that surrounds each particle contact [7]. Therefore, the performance of the mixture relies on the effectiveness of the aggregate skeleton, the properties of the bitumen, and the strength of the bitumen-aggregate adhesion [8, 9].

Bitumen is known as a product manufactured from crude oil. It is generally agreed that crude oil originated from the remains of marine organisms and vegetable matter deposited with mud and fragments of rock on the ocean bed. Although a large number of different crude oils are currently

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available, only a small percentage of these are directly suitable for bitumen manufacture [10]. Because oils are complex and varied organic compounds, it is unsurprising that bitumens are similarly complex. The individual molecules in a bitumen are large and non-uniform and they vary enormously in molecular weight and therefore in resulting physical attributes. It is useful for engineers to discern four broad classes of molecule including asphaltenes, resins, aromatics and saturates [8, 11].

Although bitumens have been used for thousands of years in construction, their properties and behaviour still exhibit interesting gaps to many of the vast number of people involved in their application. Bitumen is complex and in general non-Newtonian in its behaviour. The complexity of bitumen properties raises questions of considerable interest in understanding behaviour of bitumens [7]. It is agreed that a bitumen works as a thermoplastic material that behaves like glass at low temperature, in that it is very elastic and brittle, and like a fluid at very high temperature, in that it is able to flow when subjected to shear loading. At intermediate temperature, it behaves in a viscoelastic manner, possessing both elastic and viscous properties [1]. The study of bitumen's behaviour therefore has become an essential research aspect, and many previous studies have been conducted to investigate bitumen properties.

Rheology is a fundamental interdisciplinary science that is concerned with the study of the internal response of real materials to stresses [12]. There are two extremes of behaviour, elastic and viscous, for any material. If a stress is applied to a perfect elastic material, then the resulting strain appears immediately. When the load is removed, the material recovers its original form instantaneously. In contrast, viscous materials possess different behaviour as the resulting strain accumulates until the stress is removed. In this case, when the load is applied, the material probably shows an initial resistance to the load but then flows. When the load is removed, no recovery of the initial form is recorded [10]. The rheological properties of pure bitumens significantly depend on the chemical composition of the bitumen, especially on the asphaltene content [1]. This is in agreement with a study conducted by Mack [13] who found a considerable influence of asphaltene content on viscosity of pure bitumen. The bitumen viscosity increased as the asphaltene content increased.

Airey [12] used the Dynamic Shear Rheometer (DSR) equipment to examine the effect of polymer modification on the physical and rheological properties of various ethylene vinyl acetate (EVA) and SBS PMB's. The results indicated that for SBS samples the thermoplastic rubber SBS copolymer provides polymeric modification by means of a highly elastic network within the bitumen. There is a similar effect of this elastic network on the stiffness and viscoelastic balance of the PMB, but due to the higher melting temperature of the polystyrene blocks it is maintained to higher temperatures, within the compaction temperature range of asphalt mixtures modified with the PMB. In addition, it has been recommended that in order to accurately evaluate the rheological properties, a DSR should be used together with rheological data presentation methods like isochronal plots, master curves and Black diagrams [12].

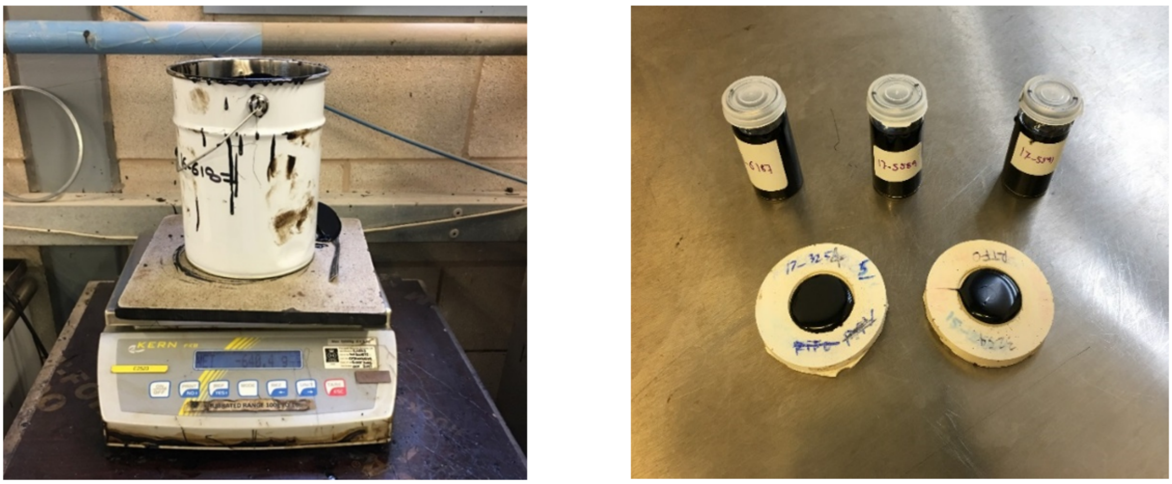
Rheological properties of PMB were examined in investigation carried out by Kumar et al. [14]. They confirmed that the performance of bituminous binders in terms of viscoelastic behaviour can be improved by addition of appropriate polymers. In the investigation, two performance grade (PG) binders, PG58 and PG64, were prepared by blending an elastomeric SBS co-polymer in 60/70 and 80/100 grade neat bitumen respectively. The test results on the rheological behaviours showed that the value of G* decreased with increase in service temperatures and PG binders have higher value of G* than 60/70 and 80/100 base binders at the same test temperature. Similarly, the same trend of phase angle can be observed from test results of 80/100 base binder modified by adding 1.0% SBS. These imply that modified binders provide elastic behaviour at the test temperature in comparison with base bituminous binders. The results are consistent with a previous intensive research conducted by Airey [15]. Airey analysed the rheological characteristics of two different source base bitumens and six PMBs produced by mixing the base bitumens by means of conventional as well as dynamic mechanical analysis using a DSR. The results of the investigation showed that the rheological properties of road bitumens are improved by means of SBS polymer modification as identified by both conventional and more fundamental rheological parameters. It is highlighted that the improved viscoelastic properties of the SBS PMBs can be indicated using dynamic mechanical analysis and the rheological parameters of complex modulus and phase angle. SBS polymer modifier increased the complex modulus and elastic response of the two base bitumens, particularly at low frequency and high temperatures.

In this paper, the binders used included 40/60 penetration unmodified bitumen, commonly used in the pavement design, PMB1 - a hard SBS polymer-modified binder, and PMB2 - a significantly softer SBS-modified binder. These binders will undergo an experimental process comprising penetration, softening point, and DSR frequency sweep tests. The findings will then be analysed, discussed, and summarised in the conclusion.

## 2. Materials and testing methods

### *2.1. Materials*

To prepare experimental binder samples, the test binders were placed into vials or moulds designed for specific tests, including penetration, softening point, and DSR (see Fig. 1). The study utilised three types of binders: 40/60 penetration unmodified bitumen (UB), hard SBS polymer-modified binder (PMB1), and soft SBS polymer-modified binder (PMB2), to assess their physical, mechanical, and rheological properties. This broad selection allows a wide range of bituminous materials through which to investigate the mechanical properties of binders used in asphalt pavements.



(a) Binder bin on the scale

(b) Vials and moulds prepared for the DSR

Figure 1. Binder samples preparation

### *2.2. Testing methods*

a. Penetration and softening point

The penetration test is considered as an indirect measurement of the viscosity of the bitumen at 25 ℃, and is a common test used in the United Kingdom as well as all around the world to specify different grades of bitumen. In this research, the test is carried out according to BS EN 1426 [16].

The ring and ball softening point test is commonly carried out to determine the consistency of bitumens by measuring the equiviscous temperature at the beginning of the fluidity range of bitumens. In this study, the test is conducted according to BS EN 1427 [17].

b. Dynamic Shear Rheometer tests

Dynamic Shear Rheometer (DSR) is an equipment used to measure the rheological characteristics of bitumen, namely the complex shear modulus ($G^*$) and phase angle ($\delta$). The operating principles of the DSR test are illustrated in Fig. 2, where the bitumen sample is sandwiched between two parallel plates, one that is fixed (stator) and the other oscillating (rotor) [12, 18].

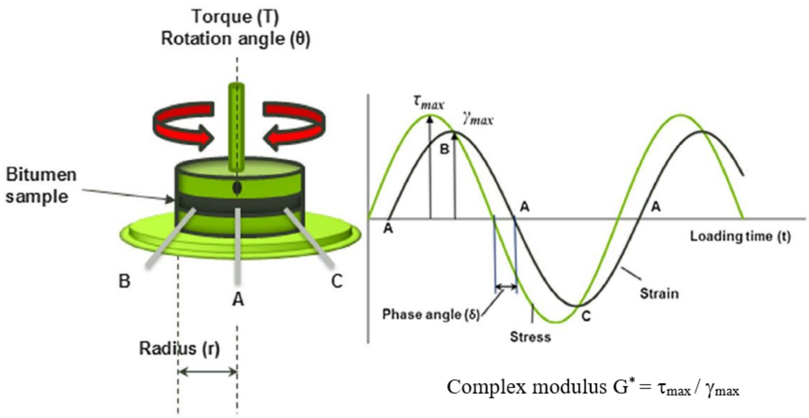



Figure 2. Schematic representation of DSR operation [18]

The bitumen frequency response test is a standard DSR test as described in AASHTO [19]. The frequency sweep method was used for determining the complex modulus $G^*$ and phase angle $\delta$ over a range of temperatures and frequencies. The test was performed in strain control.

In this study, Bohlin Gemini DSR machine provided by Nottingham Transportation Engineering Centre (NTEC), the University of Nottingham, the United Kingdom as displayed in Fig. 3 was chosen to test rheological properties of binders at temperatures of 25, 30, 35, 40, 45, 50, 55, 60, 65 and 70 ºC and frequencies of 0.1, 0.16, 0.25, 0.40, 0.63, 1, 1.59, 2.51, 3.98, 6.31 and 10 Hz.

The sample geometry of the test consists of two parameters: Plate diameter and gap thickness. In fact, there are no standard geometries which have been identified as relevant for specific ranges of binder stiffness. However, Airey [12] recommended that the complex modulus and phase angle can be measured under different temperature and frequency conditions for different plate diameters and gap thickness as shown in Table 1. Therefore, a plate diameter of 25 mm and a gap thickness of 1 mm following the testing configuration two were selected for use in this study.

Table 1. DSR test conditions for sample geometry [12]

| Parameter | Testing Configuration one | Testing Configuration wo |
|---|---|---|
| Temperature | 10 ºC to 35 ºC | 25 ºC to 75 ºC |
| Frequency | 0.01 Hz to 15 Hz | 0.01 Hz to 15 Hz |
| Plate diameter | 8 mm | 25 mm |
| Gap thickness | 2 mm | 1 mm |

The amplitude of stress is defined as the stress at the outer edge of the specimen and calculated from the torque transmitted through the sample in response to the applied strain. Similarly, the strain considered is that at the outer edge of the specimen. Thus, the stress and strain parameters can be calculated as follows:

$$\sigma = \frac{2T}{\pi r^3} \tag{1}$$

where $\sigma$ is the shear stress; $T$ is the torque; $r$ is the radius of parallel plates.

$$\gamma = \frac{\theta r}{h} \tag{2}$$

Figure 3. Bohlin Gemini DSR Equipment

where $\gamma$ is the shear strain; $\theta$ is the deflection angle; $h$ is the gap between parallel plates.

## 3. Results, analysis and discussion

### *3.1. Penetration and softening point test results*

The penetration and softening point test results carried out for the UB, PMB1 and PMB2 are presented in Table 2.

Table 2. Penetration and Softening point test results of binders

| Test | UB | PMB1 | PMB2 |
|---|---|---|---|
| Penetration (0.1 mm) | 41 | 21 | 65 |
| Softening point (ºC) | 51.4 | 93.1 | 78.8 |

Penetration is a commonly used test to assess the hardness of bitumen. In certain cases, it is also employed to evaluate the consistency of bitumen. The penetration value reported represents the average of three measurements. As shown in Table 2, the penetration values for the UB, PMB1, and PMB2 were 41, 21, and 65 (0.1 mm), respectively. As mentioned, the wide range of penetration values is intended to encompass various bituminous materials, enabling an investigation into the mechanical properties of binders used in asphalt pavements.

The softening point test, another empirical method, is frequently used to determine the consistency of a binder by identifying the equiviscous temperature at which the binder transitions between solid and liquid states. Moreover, the results of the softening point test can be correlated with the permanent deformation characteristics of asphalt mixtures. The ring and ball softening point values for the UB, PMB1, and PMB2 were 51.4 ºC, 93.1 ºC, and 78.8 ºC, respectively, as presented in Table 2. The ring and ball softening point properties of the binders tested, as expected, covered a wide range, from approximately 51 ºC to 93 ºC, allowing for a broad range of bituminous materials.

### *3.2. DSR frequency sweep test results*

a. Complex modulus master curves

It is important to evaluate the rheological and viscoelastic properties of the PMBs under different loading times and temperatures. This has been obtained by means of dynamic mechanical analysis to define the stress-strain-time-temperature response of the binders [20]. The rheological properties, complex modulus and phase angle master curves for PMBs can be constructed utilising the time-temperature superposition principle (TTSP) and William, Landel and Ferry (WLF) equation [21]. A master curve is constructed for a selected reference temperature by shifting other curves horizontally without changing the shape to coincide with the reference curve and then forming a single curve [22]. In this study, a reference temperature of 40 ℃ is selected.

The complex modulus master curves for UB, PMB1 and PMB2 at a reference temperature of 40 ℃ are presented in Fig. 4. As can be seen from the figure, all three types of binder show a similar trend of complex modulus master curve. It is that as the frequencies increase from 0.1 Hz to 10 Hz, complex moduli increase. At the same frequency, complex modulus of each testing binder decreases when the testing temperature increases from 25 ℃ to 70 ℃. In the graph, the complex modulus for PMB1 lies above the PMB2 and UB curves over the whole range of frequencies and temperatures, while in comparison with UB the PMB2 complex modulus was slightly higher at low frequencies and high temperatures.

The differences in the complex modulus curves indicate varying stiffness and temperature sensitivity among the binders. The gradually increasing complex modulus for PMB1 infers superiority in resistance towards deformation, especially towards high-frequency conditions/high-temperature ambience. PMB2 presented a higher modulus than UB at lower frequencies and higher temperatures which, combined with the decreased elastic modulus across the temperature range, could potentially be beneficial in high-temperature applications, where loading rates are low. All binders showed a decreasing complex modulus with an increase in temperature which is expected for viscoelastic materials, suggesting that a binder should be chosen according to specific service conditions.

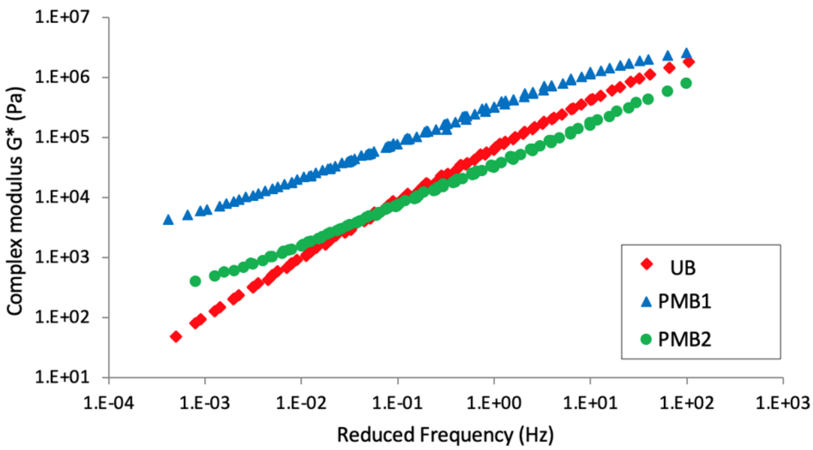



Figure 4. Complex modulus master curves for UB, PMB1 and PMB2 at a reference temperature of 40 ℃

b. Phase angle master curves

Phase angle is one of the key indicators of the viscoelastic properties of bituminous binder. A 0º phase angle represents a purely elastic material, while 90 º is representative of a purely viscous material [23].

Fig. 5 illustrates the phase angle master curves for UB, PMB1 and PMB2 at a reference temperature of 40 ℃. As can be seen from the graph, unlike the complex modulus master curves, the phase angle master curves differ in appearance for the three binders. The phase angle of the UB decreased as the frequency increased for all test temperatures. The PMB1 phase angle master curve shows a peak at intermediate temperatures, followed by a noticeable drop toward higher frequencies and there was a slight decrease in phase angle at lower frequencies (high temperature range). The UB and PMB1 only gave the same pattern of phase angle at higher frequencies although phase angle values of PMB1 were much lower than those of UB for all conditions. The phase angle master curve of PMB2 was quite different. At low and moderate frequencies, it was similar in both shape and amplitude to the PMB1 binder, but at high frequencies the PMB2 phase angle slightly increased before decreasing markedly at very high temperatures. In addition, previous studies suggested that the presence of a plateau in the phase angle curves indicates a higher interaction level between the modifier and bitumen [24]. In this study, the PMB2 phase angle master curve shows a different pattern compared to the UB and PMB1 curves by forming such a plateau zone as shown in Fig. 5. This implies that soft PMBs may be more compatible with bitumen than hard ones.

The addition of polymer modifiers significantly reduced the phase angle, enhancing the elastic properties of the binders, particularly at high temperatures and low frequencies. While the complex modulus master curves for all binders were similar, the phase angle curves varied considerably.

The phase angle master curves show significant differences in the viscoelastic properties of the binders. The UB presents an increase in phase angle according to frequency, thus forming behaviour with a greater viscous component. The PMB1 shows a maximum at intermediate temperatures and a decreased plateau at higher frequencies (the foot of the solid line, indicating a balance between elastic and viscous properties). The curve of PMB2 forms a plateau, especially at high temperatures, which indicates closer association of the modifier and bitumen, i.e., better compatibility. The addition of polymer modifiers significantly reduces the phase angle, enhancing the elastic properties of the binders, especially at high temperatures and low frequencies. These results suggest that PMB1 may perform well across a range of conditions, while PMB2 offers improved stability at high temperatures.

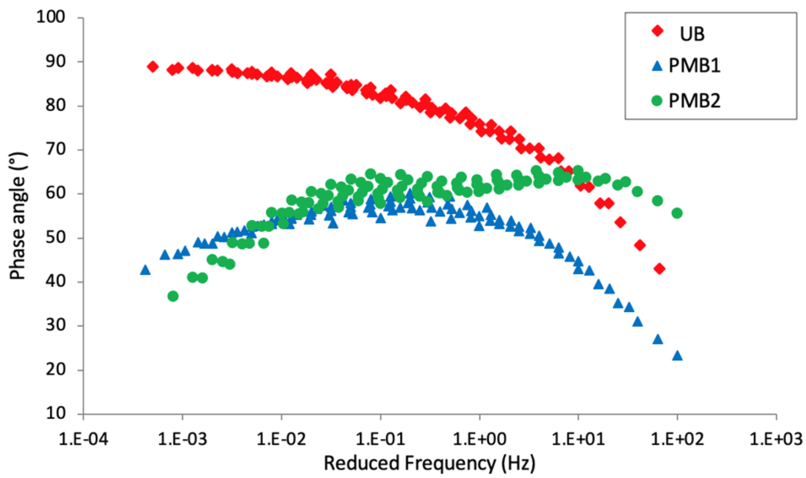



Figure 5. Phase angle master curves for UB, PMB1 and PMB2 at a reference temperature of 40 °C

c. Black diagrams

Black diagrams are often used to present the relation between stiffness and viscoelasticity of materials without the need to apply shift factors to the raw data as required for master curves [12, 22].

The black diagrams for UB, PMB1 and PMB2 are shown in Fig. 6. As with the phase angle master curves, the curves of PMB1 and PMB2 were relatively smooth but differed from those seen for the UB as can be seen in the figure. Both PMB1 and PMB2 with SBS modifiers showed a plateau region of constant phase angle which migrates towards lower phase angles. This is consistent with previous research results [12].

The plateau region shown in the black diagrams for PMB1 and PMB2 implies increased stability and constant viscoelastic characteristic in comparison with different conditions. The shift toward lower phase angles indicates that the SBS modifiers contribute to a more elastic behaviour, enhancing the binder's resistance to deformation. This behaviour contrasts with the UB, which lacks a defined plateau and shows more variability in its phase angle, suggesting less stability and elasticity. These findings highlight the superior performance of the SBS-modified binders in terms of stiffness and temperature sensitivity.

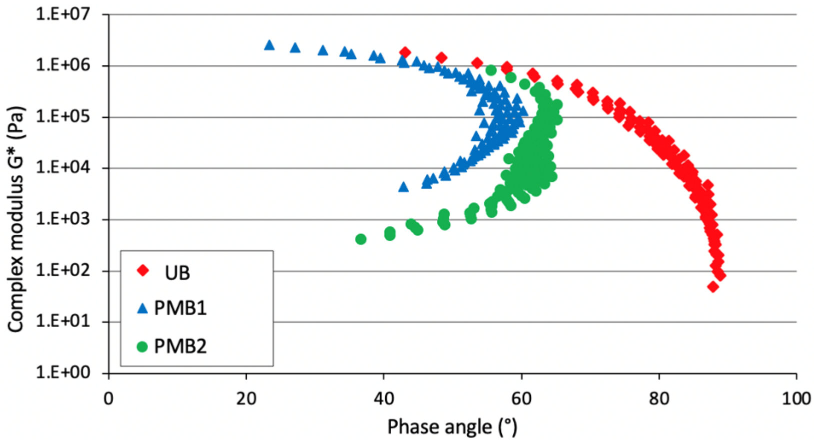



Figure 6. Black diagrams for UB, PMB1 and PMB2

## 4. Conclusions

In this research, the binders selected included 40/60 penetration unmodified bitumen (UB), a hard SBS polymer-modified binder (PMB1), and a softer variant of SBS-modified binder (PMB2). These binders were subjected to a series of experimental tests, including penetration, softening point, and frequency sweep tests using a DSR. Based on the DSR results presented in this paper, the following conclusions are drawn:

The complex modulus master curves derived from the DSR demonstrated that the stiffening effect of SBS polymer modifiers is more pronounced at low frequencies and high temperatures, suggesting enhanced resistance to permanent deformation in asphalt materials containing SBS additives.

The phase angle master curves obtained from the DSR revealed that PMB1 and PMB2 exhibited a notable reduction in phase angle at low frequencies and high temperatures, resulting in a significant improvement in the elasticity of the bitumen. Additionally, the phase angle master curve of PMB2 exhibited a distinct plateau region, suggesting that softer PMBs may have better compatibility with bitumen than harder ones.

Black diagrams produced from the DSR indicated that the curves for PMB1 and PMB2 were relatively smooth but distinctly different from those of the UB. Both PMB1 and PMB2, containing SBS modifiers, displayed a plateau region with a constant phase angle, which shifted towards lower phase angles.

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