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The "Journal of Transport" publishes the most significant results of scientific and applied research carried out in universities of transport profile, as well as other higher educational institutions, research institutes, and centers of the Republic of Uzbekistan and foreign countries.

The journal is published 4 times a year and contains publications in the following main areas:

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- Water Supply, Sewerage, Construction Systems for Water Protection;
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- Materials Science and Technology of New Materials;
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- Car Service;
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Tashkent State Transport University had the opportunity to publish the scientific-technical and scientific innovation publication "Journal of Transport" based on the Certificate No. 1150 of the Information and Mass Communications Agency under the Administration of the President of the Republic of Uzbekistan. Articles in the journal are published in Uzbek, Russian and English languages.

CONTEXT / MUNDARIJA

Relationship of rheological properties bitumen with empirical Ring and Ball softening point test

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Abstract: It was reported that for most unmodified bitumens, the R&B softening point equated to a needle penetration of about 800×0,1 mm^2 (Heukelom, 1969). R&B softening point is used as the elevated temperature performance of binders. However, the R&B softening point relevance is questionable for modified bitumens so that Dynamic viscosity is used as the elevated service temperature performance. A good correlation is reported with G*/sinδ at a frequency of 10rad/s and 60°C before and after rolling thin-rolling oven (RTFO)-aging. The correlation the dynamic viscosity with R&B softening point was good after RFTO-aging, but not before (Alexander, 2000). No relationships have been demonstrated for modified bitumen using dynamic viscosity with R&B softening point. (Balaidi, 2001). There are some relationships, but no correlation has been found in the papers between R&B softening point and other bitumen tests.

Keywords: Ring and Ball Test, Softening Point of Bitumen, Rheological properties.

1. Introduction

Bitumen is one of the oldest known engineering materials. It is known that 6000BC there was a thriving shipbuilding industry in Sumeria that produced and used natural bitumen. Also, bitumen was used in the mummification process as long ago as 1200 BC during the Ptolemaic period. In the 1900s, refined bitumen was first produced by refining cruel oil in the USA. Bitumen consumption has increased rapidly, especially in road construction. (Jiqing Zu 2014) Bitumen is a dark viscous mixture made of hydrocarbons. Bitumen is manufactured from crude oil. Nearly all bitumens present a more or less pronounced visco-elastic behavior, their resistance to deformation depends on the temperature and time during which a force is applied. Under normal temperature conditions, viscous and elastic behavior play their part. Tests are necessary for characterizing different bitumen grades. Examples of such methods of characterization and their appropriate consistency are:

Fraass breaking point – brittleness

Penetration- semi solid range Softening point- beginning of fluidity Viscosity – fluidity range Penetration Index- temperature susceptibility. The two tests used most frequently to characterize bitumens are penetration and softening point. From these two tests can be obtained important engineering properties namely, high temperature viscosity and low temperature stiffness. As the penetration and softening point tests are empirically derived it is essential that they are always carried out under exactly the same conditions. The softening point of a bitumen is used as an indication of the temperature at which the bitumens start showing fluidity. The European standard for R&B softening test is EN1427. Until 1999, most countries had their own test version which were similar to the harmonized test. In (EN1427) this test a steel ball 3.5 gram is placed on a sample of bitumen contained in a brass ring which is suspended in a water or glycerine bath. Water is used for bitumen with a softening point of 80 C or below, glycerine is used for softening points greater than 80 C. The bath temperature is raised at 5C per minute, the bitumen softens and eventually deforms slowly as the ball falls through the ring. When the steel ball touch a base plate (25±0.4) mm below the ring, the temperature of the water is recorded. After the test the mean of the two measured temperatures is reported. If the difference between the two results more than 1 C , the test must be repeated. The reported temperature is designated the softening point of the bitumen, and represents an equi-viscous temperature. (EN1427)

There are no universally accepted specifications for bitumen, cutback bitumen or bitumen emulsions. British Standards are used in the UK, in Europe, the national specifications for bitumen are in the process which is being consolidated in a unified CEN specification, while in the world the ASTM specifications are commonly used.. the principal difference was that including, the ASTM D36-95 version of the softening point test bath is not stirred, whereas in the EN1427 version the water is stirred, therefore the softening point determined by using these two methods differ. As a consequence the ASTM results are usually 1,5 C higher than for the EN1427 method. The procedure for performing the softening point test must be followed precisely to obtain accurate results. Sample preparation, rate of heating, and accuracy of temperature measurement are critical.(bitval,2006.).

2. Methodology

2.1. G*/sinδ parameter relationship with R&B softening point

A systematic approach to the development of new binder specifications was facilitated by the bitumen industries' efforts to define 'performance-related' requirements for paving binder formulations. In spite of the continuous development of new test methods, binder specifications are often based on and evolved from historic, empirical test methods that have been proven effective. R&B softening point is often used to obtain information about elevated

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temperature behavior of binders. However, its relevance to polymer-modified bitumen is contested. In many countries, especially those involved with PMB, a slow and gradual shift from the penetration-based bitumen specification to a performance related specification utilizing dynamic shear rheometer (DSR) is expected in the near future, especially, for PMBs. A good correlation is reported G*/sinδ at a frequency of 10 rad/s and 60 ºC before and after rolling thin-film oven (RFTO)-aging. The correlation of dynamic viscosity with the R&B softening point was good after RFTO-aging, but no relationships have been indicated for PMBs. (Khattak et al. 2001). G*/sinδ parameter was the elevated service temperature property in the Superpave (Superior performing asphalt pavements) binder specification of this rheological parameter and was developed by strategic highway research program (SHRP). This value calculated from the complex shear modulus G* divided by the sine of the phase lag(phase angle). Higher values of this parameter lead to a decreased tendency to permanent deformation. G*/sinδ correlated well with the tendency for permanent deformation. However, this parameter is less useful for binders that contain polymers or other modified binders. (Claxton et al. 1996; Carswell at al. 2000).

2.2. Equi-viscous and equivalent modulus temperatures" based on a DSR relationship with R&B softening point of bitumen

A reliable quantitative relationship can be used to estimate the old empirical parameters from fundamental DSR results, which is more efficient and time-saving than running additional tests. Furthermore, it can lead to a better understanding of why the old empirical parameters were inadequate. It is important to know how DSR results relate to the R&B softening point of modified and unmodified bituminous binders.(J.Ziqing et al. 2020).

Some studies investigated the R&B softening point of bitumen to determine its equivalent softening point based on DSR test. It was reported the concept " equiviscous temperature" based on low shear viscosity (LSV) using a DSR in low frequency oscillation mode. This is temperature at which the complex shear modulus is 2kPa·s and it was demonstrated that it is a good approximation for unmodified bitumen but not for PMBs. (Zoorob et al. 2011)

Fan Liang (F. Liang et al. 2014) tested more than 30 different asphalt binders using a DSR test.

2.3. Binder-Fast-Characterisation-Test (BTSV Bitumen-Typisierungs-Schnell-Verfahren) relationship with R&B softening point test

Whenever modified binders were investigated, there was no reliable correlation between rheological parameters and empirical softening point. Seven plain binders and polymer modified binders with different penetration grades were tested in the DSR at a frequency of 10 rad/s. Figure 2 shows the complex shear modulus G* at the R&B softening point temperature with a different binders. (Alisov at al. 2020). According to Figure 2, some of the plain binders (70/100, 50/70, 30/45) have an approximate complex shear modulus of 15 kPa on average, while others show either much smaller values or much higher ones. Thus, the R&B softening point reflects equivalent rheological properties only for plain binders. G* varies significantly for PMBs. This indicates that when polymer modified is used to enhance the material property the R&B softening point does not behave consistently. (Alisov et al. 2020).

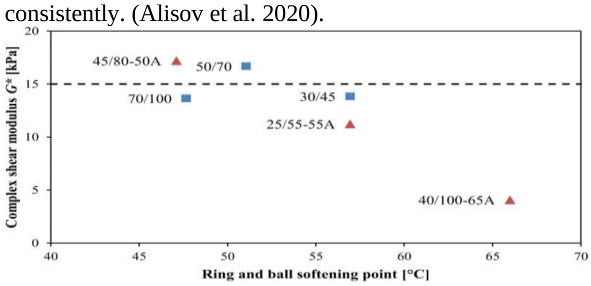

Figure 1. the complex shear modulus G*, measured at the R&B softening point temperature with a frequency of 10rad/s for different asphalt binders. (Alisov et al. 2020)

In a logarithmic scale was used to describe binders tested at their R&B softening point temperature in the DSR (Figure 3). Figure 3 clearly shows how the temperature of R&B softening point for PMBs can lead to much lower values of the complex shear modulus (G*=2563Pa), whereas the highest values were measured for plain binder (G*=15682Pa). According to the exponential relationship between binder and material stiffness, a range of approximately 13 kPa corresponds to a difference of 12,5 ºC. Softening point is relatively inaccurate at representing equivalent rheological material states.(Alisov et al. 2017)

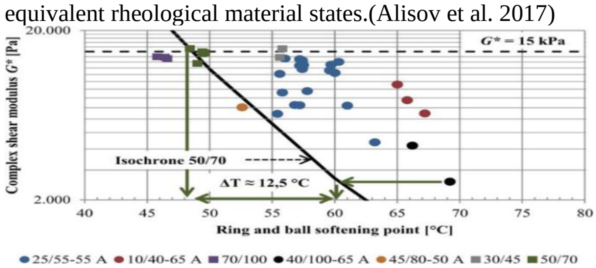

Figure 2. Complex shear modulus G*, measured at the R&B softening point temperature, with a frequency of 10rad/s for different asphalt binders and an isochrones for 50/70. (Alisov et al. 2017)

It is therefore possible to characterize the material behavior of binder at high temperatures by determining the isomodulus temperature corresponding to a complex shear modulus of 15 kPa. After the complex shear modulus reaches the threshold value of G*=15 kPa, the result of the test is obtained. At this point, the temperature T_{BTSV} which is related to the R&B softening point test and δ_{BTSV} are determined as the two key parameters that describe the material. 164 different binders were selected for testing and some of the binders were aged with RFTO at the temperature of 163 ºC (Alisov et al. 2018). BTSV is a test method that mesures complex shear modulus and corresponding phase angle as a function of temperature.

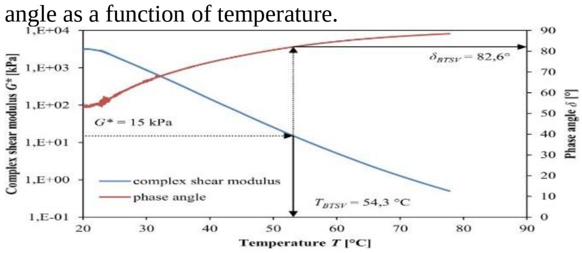

Figure 3. Example for determining T_{BTSV} and corresponding phase angle δ_{BTSV} for a 50/70 pen-grade bitumen from BTSV results. (Alisov et al. 2018)

The master curves of the complex shear modulus and phase angle acquired from frequency and temperature sweep tests. The BTSV is obtained by first determining the reduced frequency that corresponds to a complex shear modulus of 15 kPa and then determination of phase angle that corresponds to that reduced frequency. From the reduced frequency, the temperature T_{BTSV} corresponding to a frequency of 1.59 Hz can be back-calculated using the William Landel Ferry equation.

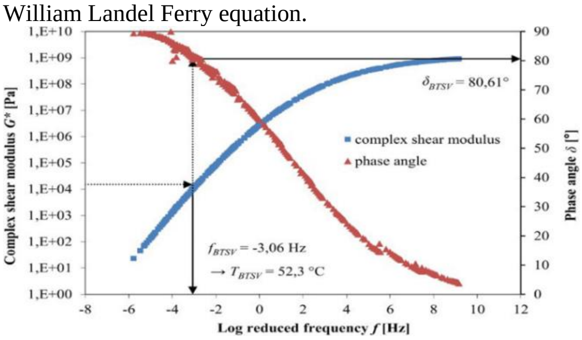

Figure 3. . Determination of phase angle from master curve of a 50/70 pen-grade binder. (Alexander Alisov et al. 2018)

Theoretical equation is used to obtain BTSV results. (1). (NCHRP, 2001)

$$G^*=G_e^*+\frac{G_g^*-G_e^*}{(1-(\frac{f_c}{f'})^k)^{\frac{m_e}{k}}} \qquad (1).$$

Where, G_e^*- G^*(f→0), equilibrium complex modulus, G_e^*=0 for binders. $G_g^*=G^*$ (f→∞),glass complex modulus; f_c- location parmeter with dimensions of frequency f'- reducued frequency, function of both temperature and strain; m_e, k – shape parameters, dimensionless.

Using William-Landeln-Ferry equation (2) T_{BTSV} temperature can be calculated. (William and Landeln, 1955)

$$\log a_T(T_{BTSV})=-\frac{c_1(T_{BTSV}-T_0)}{c_2(T_{BTSV}-T_0)} \qquad (2)$$

Last but least, using the reduced frequency f' equals to 1,59 Hz multiplied for the temperature shift factor, determined in the previous step.

From (3) the value of δ_{BTSV}

$$\delta_{BTSV}=90\cdot I-(90\cdot I-\ \delta_m)\cdot(1+(\frac{(\frac{f_d}{f'})}{R_d})^2)^{-\frac{m_d}{2}} \qquad (3).$$

Where, δ_m- is the phase at the inflexion point, f_d-is the frequency at which δ_m occurs. R_d and m_d – shape parameters. I = 0 if f>f_d and 1 if f≤f_d T_{BTSV} which indicate hardness of the binder (R&B softening point) and δ_{BTSV} shows the elastic ratio of viscoelastic behavior of asphalt binders. These parameters can be calculated empirical and theoretical way. After testing more than 164 different binders T_{BTSV} is a good estimation of R&B softening point for unmodified bitumen, but not for PMBs. In figure 6 shows how T_{BTSV} and R&B softening point related to each other. (Jiqing Zhu at al. 2021)

3. Results and discussion

3.1. G*/sinδ parameter relationship with R&B softening point

Higher values of this parameter lead to a decreased tendency to permanent deformation. G*/sinδ correlated well with the tendency for permanent deformation. However, this parameter is less useful for binders that contain polymers or other modified binders. (Claxton et al. 1996; Carswell at al. 2000).

3.2. Equi-viscous and equivalent modulus temperatures" based on a DSR relationship with R&B softening point of bitumen

The equivalent modulus rule can be used to calculate the R&B softening point of neat asphalt binder. And between the calculated and the measured softening points a good relationship was found for unmodified binders. As for the complex shear modulus of bitumen was 13.034kPa.

3.3. Binder-Fast-Characterisation-Test (BTSV Bitumen-Typisierungs-Schnell-Verfahren) relationship with R&B softening point test

The BTSV method utilizes an iso-modulus approach (G∗ = 15 kPa at 10 rad/s) to evaluate bitumen, focusing on equivalent temperature (TBTSV) and phase angle (δBTSV) plots. For unmodified binders, lower penetration grades (harder bitumen) correspond to higher TBTSV values, with δBTSV values around 82°. After RTFOT ageing, TBTSV increases and δBTSV decreases to about 80°, a trend expected to continue with further ageing (Alisov et al., 2020). Modified bitumen with 3% SBS exhibits similar ageing trends, with lower δBTSV values compared to unmodified binders. With 5% SBS, δBTSV further decreases and remains stable post-RTFOT, while TBTSV increases. The BTSV method offers an iso-modulus evaluation of bitumen, typically at a higher modulus than PMB at the Ring and Ball softening point.(Jiqing Zhu et al.)

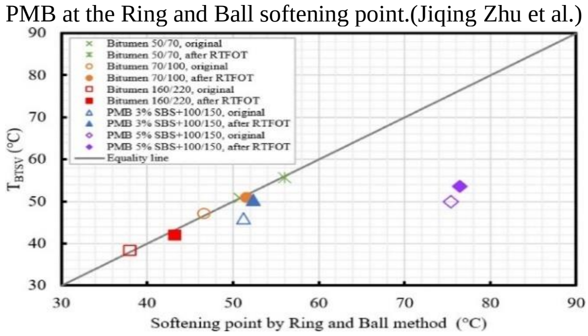

Figure 4. T_{BTSV} results of bitumen versus the R&B softening point. (Jiqing at al. 2021)

4. Conclusion

After conducting a comprehensive review of the state-of-the-art literature on the relationship between the rheological properties of bitumen and the Softening Point Ring and Ball test, several key insights can be drawn.

The development of new binder specifications is moving towards performance-related requirements, particularly for polymer-modified bitumen (PMB). Despite advancements in test methods, traditional empirical tests like the R&B softening point remain common. There is a gradual shift towards using the dynamic shear rheometer (DSR) for performance specifications. The parameter G*/sinδ, used in the Superpave binder specification, shows good correlation with permanent deformation tendencies, although it is less effective for polymer-modified binders

A quantitative relationship derived from dynamic shear rheometer (DSR) results can estimate old empirical parameters efficiently, saving time and providing insight into their limitations. Understanding how DSR results relate to the R&B softening point is crucial, particularly for modified and unmodified bituminous binders. Studies have shown that while the concept of "equiviscous temperature" works well for unmodified bitumen, it is less effective for polymer-modified bitumen (PMB). Testing has also demonstrated a strong correlation between calculated and measured softening points for unmodified binders using the equivalent modulus rule. When investigating modified binders, no reliable correlation was found between rheological parameters and the empirical R&B softening point. Testing showed that while the R&B softening point is consistent for plain binders, it does not behave consistently for polymer-modified binders (PMBs). The complex shear modulus (G*) at the R&B softening point temperature varies significantly for PMBs, indicating the inadequacy of the softening point for representing their rheological properties. To better characterize binder behavior at high temperatures, the isomodulus temperature corresponding to a G* of 15 kPa can be used. This approach, combined with the BTSV test method, provides more accurate parameters for describing binder materials.

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