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TEMPERATURE SUSCEPTIBILITY AND RHEOLOGICAL AGING CHARACTERISTICS OF THE BITUMEN HAVING DIFFERENT PENETRATION GRADES

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Abstract: The performance of the asphalt pavement highly depends on the properties of the bitumen. Therefore, it is important to classify the bitumen's characteristics in order to ensure the selected binder will provide the desired properties to the asphalt pavement. The Penetration grade, which categorizes the asphalt binder depending on the penetration value of the sample, is one of the most popular system for classification. Since the system considers the penetration value, the bituminous materials having different penetration grade is expected to have different physical, chemical and rheological properties. Within the scope of this study, four bitumen having different penetration grades as 50/70, 70/100, 150/200 and 160/220 were investigated by conventional and rheological analyses. The Penetration Index (PI) and the Rheological Aging Index (RAI) of the samples were calculated to evaluate the effects of penetration grade system on temperature susceptibility and rheological aging characteristics of the samples. As a result, the binders having lower penetration grades are found to be better in terms of rheological aging performance and temperature susceptibility. Furthermore, a very high correlation was found between RAI and PI values of the samples by statistical analysis.

Keywords: Penetration grade, Temperature susceptibility, Penetration index, Rheological aging index

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1. Introduction

Bitumen has the properties of a thermoplastic material, which makes it to soften while temperature rises and hardens when the temperature decreases. Bitumen acts like Newtonian liquid at high temperatures (over 70°C), which can be explained by its decreased viscosity as temperature rises and its viscoelastic behaviour at ambient temperature (Redelius, 2006).

Bitumen is mostly used as an asphalt binder, while it can be used inside the roofing materials, water tightening etc. (Redelius and Soenen, 2015). Bitumen is a complex combination of different organic molecules, mostly hydrocarbons, but it also contains sulphur, nitrogen, and oxygen, as well as metals such as vanadium, nickel, and iron (Traxler, 1936; Traxler and Coombs, 1936; Romberg et al., 1959). Asphalt binders mostly classified as their penetration grade or performance grade. Performance grade (PG) system considers the properties of the bitumen at various temperatures. The primary goal of using the PG system to choose asphalt binders, is to ensure that, the binder has the right characteristics for the field's environmental conditions. Although PG system has more advantages, the penetration grade system is more common and many countries including The Republic of Turkey, still uses the penetration grade system in the road construction specifications.

In penetration grade system, the penetration test is used to describe the bitumen by considering its hardness. The classification was done depending on the penetration value of the sample at 25°C. However, the results of the softening point tests and Frass Breaking point tests are also controlled to be in agreement with the specification limits. For road construction, the most common penetration grade range varies from 25 to 200. Lower bitumen penetration grade is more convenient for hot regions; while the higher the penetration grade is preferable for cold regions for the construction of asphalt pavement (Kasak et al., 2004).

In the literature, researchers studied the impacts of penetration grade of the bitumen samples considering different properties. Kuloglu et al. investigated the workability and rheological performance of the samples having different penetration grades. According to their result, it was determined that once the penetration grade of binder increases, the viscosities and rutting resistance of the samples decreases (Kuloğlu et al., 2008). Yang et al., studied the effects of aging on the chemical and rheological properties of bituminous samples having different penetration grades and they concluded that micro-mechanics of the samples, which depends on the penetration grade, is directly related with the rheological characteristics (Yang et al, 2018). Rahman et al., assessed the different penetration grade influences on conventional properties of the binders as: penetration, ductility, softening point and specific gravity etc. As a result, they recommended selecting the suitable grade of bitumen for road construction depending on the climates and the conditions of the area (Rahman et al., 2019). Similarly, in the study of Sağlık and Öztürk (2014) the rheological characteristics of different type of binders were investigated and the results were altering by the penetration grade of the samples.

Bitumen ages at several phases of its service life, including processing, mixing, shipping, laying, and the rest of its usage and service life (Mallick and Tahar, 2013). The first stage of ageing is related with the loss of volatalization in asphalt during the construction and mixing phase, also known as short-term aging. The second and final stage of aging is linked with the progressive oxidation in the field of service, also known as long-term ageing (Bell, 1989). Oxidation occurs when bitumen interacts with the oxygen.

The aging resistance of the bituminous materials are directly depends on the hardness/softness of the samples, which is related with the amount of oily fractures (saturates and aromatics) within the structure. Aging results in hardening of the bitumen by causing the evaporation of the oily fractions of its colloidal structure. The more the amount of these fractions the more the hardness, thus the higher the penetration grade for bitumen. Therefore, it is possible to say that, the aging can affect more to the bitumen having higher penetration grade, since there are more compounds to be evaporated. The effects of aging can be evaluated by considering the changes on the structural, morphological, thermal or rheological properties of the bituminous samples. Researchers, were able to examine the bitumen ageing resistance using several techniques and calculation of rheological ageing index (RAI) is among them (Salomon and Zhai, 2004; Zhang et al., 2018; Gao et al., 2020; Celauro et al., 2020).

Within the scope this study, the effects of penetration grades on the temperature susceptibility and rheological aging characteristics of the samples were examined. The Penetration Index (PI) and Rheological Aging Index (RAI) of the binders having 50/70, 70/100, 100/150 and 160/220 penetration grade, were calculated by penetration and softening tests together with the generation of complex modulus master curves obtained by Dynamic Shear Rheometer (DSR) analyses. Additionally, the correlation between the PI and the RAI results were investigated to evaluate the relationship between the temperature susceptibility and rheological aging characteristics of the samples.

2. Material and Methods

2.1. Testing Methods and Equipment

2.1.1. Aging of the samples

There are different test methods that simulate the effects of short and long-term aging in the laboratory environment. The current and most popular test method for simulating short-term aging is the Rolling Thin-Film Oven Test (RTFOT). The experiment was carried out in cylinder RTFO containers, poured 35-40 g of bitumen at 163°C at 4000ml/min. It is performed by applying air in the air stream for 85 minutes (ASTM D2872). In long-term aging simulation, the Pressure Aging Vessel (PAV) is used and its conditions are as follows: temperature of 100°C, air pressure of 2.1Mpa and aging time of 20 hours (ASTM D6521). The total aging effects of the asphalt pavement at the end of its service life can be obtained more accurately with the combination of these aging method (short term+long term aging) (Jiang et al., 2020). Therefore, in this study, long-term aged samples were obtained by applying the two aging in combined mode.

2.1.2. Rheological analysis

Complex modulus (G*) is defined as the ratio of maximum shear stress to maximum strain, and provides a measure of the total resistance to deformation, when the bitumen is subjected to shear loading. The complex modulus (G*) consists of the loss module (G'') and the storage module (G'). The storage module, which represents the elastic component, shows the amount of energy stored in the sample during each loading cycle. The lost module, which represents the viscous component, shows the amount of energy lost in the sample during each loading cycle. It is possible to examine the changes in the viscoelastic behavior of bitumen with the complex shear modulus value. The complex shear modulus (G*) can be obtained by using the shear stresses applied with Dynamic Shear Rheometer (DSR). Within the scope of the study, the changes in the complex shear modulus (G*) values before and after aging of bitumen having different penetration grades were examined using Bohlin (Gemini II) model DSR under the following test conditions:

- Mode of loading: Controlled-strain;
- Temperatures: 10 – 80°C (10 °C intervals);
- Frequencies: 0.1–10 Hz
- Plate geometries: 8-mm diameter with a 2-mm gap (10– 40°C) and 25-mm diameter with a 1-mm gap (40– 80°C).

2.1.3. Penetration index (PI)

Within the scope of the study, the temperature sensitivity of the binders with different penetration grades were evaluated by comparing the Penetration Index (PI) values. Temperature susceptibility is defined as the change in the consistency parameter as a function of temperature. The most common approach to PI calculation is given in Equation 1 (Read and Whiteoak, 2003):

$$PI=\frac{1952-500\log(Pen25)-20\times SP}{50\log(Pen25)-SP-120} \quad (1)$$

Where Pen25 is the penetration value at 25°C and SP is the softening point of the sample.

2.1.4. Rheological aging index (RAI)

The Time Temperature Superposition Principle (TTSP) is used to generate master curves for materials with linear viscoelastic behaviour, such as bitumen (Partal et al., 1999; Polacco et al., 2003; Polacco et al., 2004; Ferry, 2016). The complex modulus values of different temperatures are shifted by the selected reference temperature to create master curves. This allows researchers to quickly acquire rheological characteristics of bitumen at extremely low frequencies that are difficult to study in the lab conditions (Liang et al., 2015). The shifting factors (αT) for the generation of master curves can be determined using a variety of methods. The reference temperature in this investigation was set to 20°C, and the horizontal shifting factors (αT) were calculated using Arrhenius-like formulae. The RAI values of the samples were computed using a technique established by Cavalli et al., after the master curves for each unaged and aged sample were generated. Within a specific range of frequencies, the area under the master curves for unaged and aged samples was computed. To provide an accurate comparison, the frequencies utilised for integral limits should be specifically chosen, ensuring that the same frequencies are attained when the master curves are generated for each sample. Equation 2 is used to determine the RAI.

$$RAI = \int_{-4}^{4} \log G^* (\xi_{aged}) - (\xi_{unaged}) d\xi \quad (2)$$

Where G* is the complex modulus and ξ is the logarithm of the frequency.

3. Results and Discussion

3.1. Penetration Index Results

In order to obtain the Penetration Index of the binders having different penetration grade, the penetration and softening point tests were conducted on the samples. Table 1 presents the results of these tests for each binders.

Table 1. Penetration and Softening point values of the samples

Bitumen	Penetration (25 °C; 0.1 mm) ASTM D5-97	Specification limits of Penetration	Softening point (°C) ASTM D36-95	Specification limits of Softening Point
50/70	68	50-70	54	46-54
70/100	91	70-100	49.8	43-51
100/150	130	100-150	45.5	39-47
160/220	190	160-220	41	35-43

As can be seen in Table 1, both softening point and penetration values of each binders were within the specification limits. The Penetration Index (PI) for each binders were calculated with the values in Table 1 according to the Equation 1 and the results are given in Figure 1.

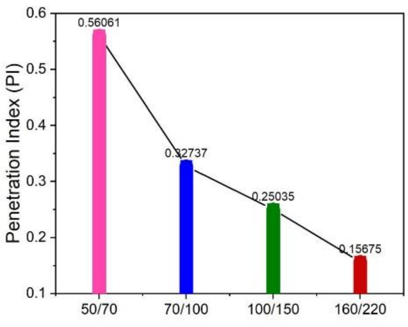

Figure 1. PI values of the samples.

The Penetration Index (PI) is used to evaluate the temperature susceptibility of the bituminous samples. PI value of less than -2 indicates that the bitumen is very sensitive to temperature, while a value higher than +2 indicates less sensitivity (Keyf, 2010). Fig 1 clarifies that, the bitumen having higher penetration grade has less penetration index, which is reasonable because, as the binder hardness increases, the PI value increases, so the temperature susceptibility decreases (Kuloğlu et al., 2008).

3.2. Rheological Aging Index Results

Master curves of each sample were constructed by choosing the reference temperature as 20°C and presented in Figure 2.

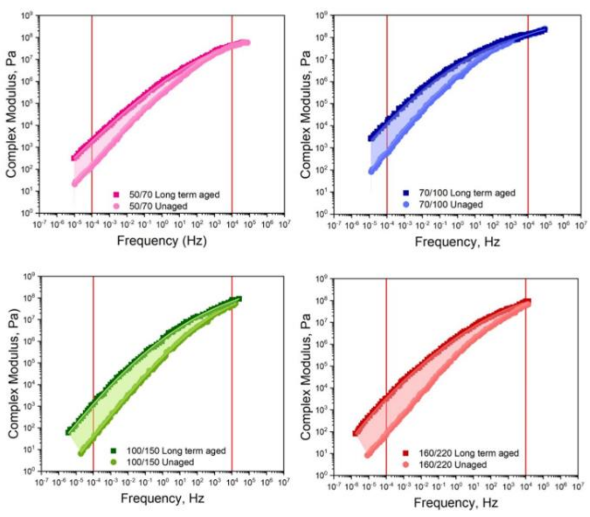

Figure 2. Master curves of aged and unaged samples.

The frequency sweep tests were conducted at frequencies between 0.1 Hz and 10Hz, however as can be seen in Figure 2, the master curves made it possible to evaluate the viscoelastic properties of the bitumen at frequencies between 0.0001 and 10000 Hz. Fig 2 reveals that, the complex modulus at high frequency is considerably higher than at low frequencies. The reason for this is that bitumen behaves more flexible under high frequencies. Additionally, the complex modulus value corresponding to the same frequency were slightly lower for the samples having higher penetration grades. This can be explained by the decreased elastic behaviour of the bitumen when becomes to be softer.

The change in the complex modulus master curves of the samples after subjecting to the aging is a sign of aging resistance. The more resistant samples to the aging, the less the change in the complex modulus master curves. It is clear in Figure 2 that, the complex modulus of the samples having higher penetration grades are affected more from the aging and in order to clarify that, the RAI values of each samples were calculated according to the Equation 2 and presented in Figure 3.

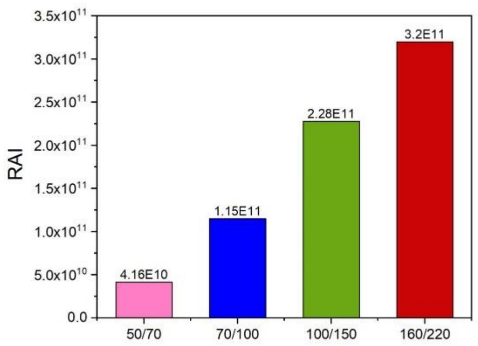

Figure 3. RAI values of the samples.

As can be seen in Figure 3, the higher penetration grades resulted in higher RAI for the samples. The higher the RAI value means the decreased aging resistance (Poulikakos et al., 2019). Therefore, it is possible to say that, binders having higher penetration grade, or in other word, the softer binders, are more influenced by aging. This might be clarified by the aging phenomenon, since aging results in the evaporation of oily fractions of the bitumen, the softer bitumen involving more of these fractions, will have more to lose.

3.3. Correlation between the PI and RAI

The relationship between conventional properties and the rheological characteristics of the samples can be evaluated together (Sarışın et al., 2021; Oner, 2019). As a final approach the correlations between the RAI and PI values of the samples were investigated to evaluate the relationship between the temperature susceptibility and rheological aging characteristics of the samples. The determination coefficient (R^2) and the equation of the correlation are given in the graph (Figure 4).

An exponential relationship was established between RAI and PI with a very high correlation coefficient (R^2=0.98). This result can be interpreted as the rheological characteristics of the bitumen, is related with the conventional properties of the samples as softening point and penetration value. It was also observed that as the PI value of the binders increased, the RAI value decreased. This is because the temperature is the main reason of aging, and better temperature susceptibility results in increased aging resistance.

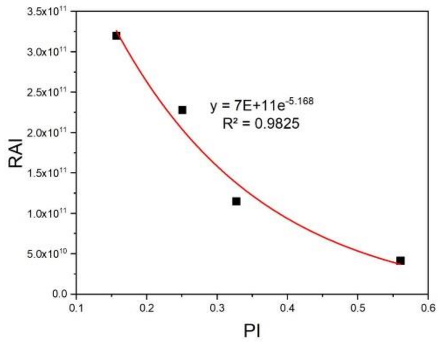

Figure 4. Correlation between the RAI and PI.

4. Conclusion

In this study, the effects of penetration grade of the bitumen on the temperature susceptibility and the rheological aging characteristics were investigated by calculating the PI and the RAI values of the samples. Additionally, the correlation between abovementioned properties of the bitumen were obtained and discussed. The findings of the study are as shown below:

- Lower penetration grades results in higher PI values, which results in higher temperature susceptibility.
- The complex modulus at high frequencies are considerably higher than at low frequencies due to the fact that, bitumen behaves more flexible under high frequencies. Also, the complex modulus value corresponding to the same frequency are slightly lower for the samples having higher penetration grades depending on the viscoelastic behaviour of the bitumen.
- Lower RAI value is obtained for the binders with lower penetration grades, which is a result of better aging resistance. This can be explained by including less oily fractions that can evaporate easily after aging.
- A good correlation is found between RAI and PI values of the bitumen. Therefore, it is possible to say that temperature susceptibility can be evaluated with rheological aging characteristics of the samples or vice versa.

As a result, the binders having lower penetration grades are found to be better in terms of rheological aging performance and temperature sensitivity. However, further studies by considering the low temperature or traffic characteristics should be investigated to evaluate the binders' penetration grade effects on the performance of the asphalt roads.

Author Contributions
All tasks were done by the single author. The author reviewed and approved the manuscript.

Conflict of Interest
The author declared that there is no conflict of interest.

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