

# Investigation of Rutting and Low Temperature Cracking Behavior of Reactive Ethylene Terpolymer and Waste Cooking Oil Modified Bitumen

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## ABSTRACT

With sustainability being the most crucial issue of recent years, the use of waste materials in bitumen modification is increasing and becoming widespread. In this experimental study, it was aimed to investigate the high- and low-temperature behavior of bitumen samples modified with waste cooking oil (WCO), reactive ethylene terpolymer (RET), and polyphosphoric acid (PPA). Accordingly, the multiple stress creep and recovery (MSCR) test and the bending beam rheometer (BBR) test were conducted. Depending on the increasing WCO ratio, the  $J_{nr}$ ,  $R\%$ ,  $\Delta T_c$ , and  $\lambda$  parameters of modified bitumens were examined in detail. It was observed that with increasing WCO ratio, the  $J_{nr}$  value increased, and elastic recovery and stiffness decreased. In addition, it has been determined that this composite-modified bitumen is resistant to heavy traffic loads and has sufficient flexibility at low temperatures.

**Keywords:** Reactive ethylene terpolymer, waste cooking oil, bitumen modification, MSCR, BBR.

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## **Nomenclature**

AASHTO	: American Association of State Highway and Transportation Officials
ASTM	: American Society of Testing and Materials
BBR	: Bending beam rheometer
DSR	: Dynamic shear rheometer
Jnr	: Creep compliance
MSCR	: Multiple stress creep and recovery
m-value	: Creep rate
PAV	: Pressure aging vessel
PG	: Performance grade
PMB	: Polymer-modified bitumen
PPA	: Polyphosphoric acid
R%	: Percent recovery
RET	: Reactive ethylene terpolymer
RTFO	: Rolling thin-film oven
WCO	: Waste cooking oil
$G'$ , $G''$ , $G^*$	: Storage modulus, Loss modulus, Complex modulus
$G^* / \sin \delta$	: Rutting parameter
$G^* \cdot \sin \delta$	: Fatigue parameter
$\Delta T_c$	: Difference between critical temperatures by $S(t)$ and m-value, delta $T_c$
$S(t)$	: Creep stiffness
$\delta$	: Phase angle
$\lambda$	: $S(t)$ divided by m-value, lambda

## **1. INTRODUCTION**

Bituminous pavements have been used in flexible pavements for many years. Although bituminous pavements performed adequately in the early years of their use, they were subject to stress over time due to increasing traffic volumes [1, 2]. In other words, population growth and the development of living standards have led to an increase in traffic loads, resulting in pavement deterioration. This has created a need to improve the performance of asphalt pavements. One of the methods used to increase the performance of asphalt pavements is bitumen modification [3]. Polymers are additives frequently used as bitumen modifiers and are generally divided into elastomers and plastomers [4-7].

In some studies, reactive polymers have been included in the polymer additive classification as a third group [8-10]. These polymers contain functional groups that are assumed to bind with bitumen molecules. Maleic anhydride and thermoplastic elastomers functionalized with ethylene-based copolymers containing epoxy rings are examples of this group [10]. The second is commercially available as random terpolymers of ethylene, glycidyl methacrylate, and an ester group. Based on their composition, they are often called reactive ethylene terpolymers (RETs) [10].

A terpolymer is a chemical substance that is produced when a polymer has a molecular structure that is primarily or entirely made up of several bonded or comparable units (such as a complex resin). Terpolymers are created when three distinct monomers are

copolymerized. It has been stated in many studies that RET has good compatibility with bitumen [8-11].

Recently, some researchers have drawn attention to an environmentally friendly approach by using waste materials in bitumen modifications. Accordingly, rubbers [12], plastics [13], oils [14], and various waste materials [15, 16] are used in bitumen modification. Waste cooking oils (WCOs) are used as rejuvenators or softeners of bitumen. Additionally, the low-temperature performance of bitumen increases with the addition of the WCO [17].

As known, the performance of base and modified bitumen in pavement is simulated by experimental methods in the laboratory environment. In this context, dynamic shear rheometer (DSR) and bending beam rheometer (BBR) tests are widely used to determine the high-temperature and low temperature performance of bitumen, respectively. Additionally, with the data obtained from these tests, the Performance Grade (PG) of bitumen can be determined, which indicates the maximum and minimum temperatures the pavement will serve in the field [18].

Over time, just as there was a need to improve the properties of bitumen, there was also a need to enhance experimental methods used to characterize bitumen. In particular, in studies conducted with accelerated road tests, it has been stated that the rutting parameter obtained from the DSR test has a very low correlation with reality [19]. It is thought that this is because bitumen behaves in the linear elastic region when tested with DSR. On the other hand, rutting is associated with nonlinear behavior. Nevertheless, this parameter was well correlated with the unmodified bitumen. However, with the widespread use of bitumen modification, it began to be questioned. Therefore, the AASHTO M 332 [20] standard has been developed to overcome the disadvantages. Accordingly, the multiple stress creep and recovery (MSCR) test has been added to the PG system to evaluate the rutting behavior of bitumen independently of the modification type.

Similarly, it is also a matter of debate in the literature which of the two different parameters (creep stiffness,  $S(t)$  and creep rate,  $m$ -value) obtained from the BBR test should be used when determining the low-temperature grade of bitumen in the PG system [21]. In this regard, new parameters such as  $\Delta T_c$  [22] and  $\lambda$  [23], where both parameters of the BBR test are used together, have been developed. It is thought that evaluating the low temperature behavior of bitumen over these parameters will give more realistic results from the low temperature performance of the PG system.

In a previous study by Kumandaş et al. [24], PG of RET + polyphosphoric acid (PPA) + WCO-modified bitumen was determined according to the Superpave specification. As a result, they stated that both high and low-temperature performance of base bitumen could be improved by adding RET+PPA+WCO additive combination. Furthermore, they observed that the low-temperature performance also increased with increasing WCO ratio in the additive combination. Considering the benefits of this additive combination, it has also been used in this study. On the other hand, it was considered that both low- and high-temperature performance should be examined in detail with different methodologies, considering that the investigation based on the PG system would be insufficient, as stated before. To the best of the authors' knowledge, it is believed that there is no study in which the rutting behavior of RET and WCO-modified bitumen is examined in detail with the MSCR test. Thus, this study was undertaken to fill this gap in the literature. Furthermore, this study gives pavement

engineers an understanding of the impact of the WCO utilization in bitumen modification on pavement performance.

The objective of this research was to examine the impact of varying ratios of WCO addition to 1.5% RET and 0.2% PPA modified bitumen on the rutting behavior at high temperatures and cracking behavior at low temperatures. The study utilized MSCR and BBR tests to evaluate the resistance against rutting and low-temperature cracking, respectively.

## **2. MATERIALS AND METHODS**

### **2.1. Preparation of Modified Bitumen**

50/70 penetration-graded bitumen obtained from the Kırıkkale oil refinery was used as the base bitumen. This penetration grade was chosen based on other studies in the literature [25, 26]. Elvaloy® RET 5160 was used as the polymer and supplied by Komsa company in Türkiye. 1.5% RET was used in polymer-modified bitumen (PMB), and this ratio was also chosen based on other studies in the literature [27, 28]. The physical properties of base bitumen and RET are given in Tables 1 and 2, respectively. PPA has been suggested for a quicker reaction between bitumen and RET [9, 29]. Accordingly, PPA was used as a catalyst in PMB, and the added PPA ratio was determined as 0.2% based on the studies in the literature [30, 31]. On the other hand, the WCO used in this study was only filtered and obtained from Degam company in Türkiye. In order to investigate the effect of WCO addition on the PMB, different ratios (2-8%, 2% increments) of WCO were used.

*Table 1 - Physical properties of base bitumen.*

<b>Physical Properties</b>	<b>Value</b>	<b>Standard</b>
Penetration	52 dmm	ASTM D5
Softening Point	48 °C	ASTM D36
Ductility	150+ cm	ASTM D113
Elastic Recovery	%10.82	ASTM D6084
Flash Point	332 °C	ASTM D92
Fire Point	370 °C	ASTM D92
Specific Gravity	1.042 g/cm <sup>3</sup>	ASTM D70

*Table 2 - Physical characteristics of Elvaloy® RET 5160.*

<b>Characteristics</b>	<b>Value</b>	<b>Standard</b>
Tensile strength	3653 psi	ASTM D638
Elongation at break	%718	ASTM D638
Density	0.95 g/cm <sup>3</sup>	ASTM D792
Melting point (DSC)	80 °C	ASTM D3418
Freezing point (DSC)	55 °C	ASTM D3418
Highest processing temperature	220 °C	-

During the preparation of the modified bitumen, the base bitumen was first heated in an oven at 180 °C until it became fluid and then placed on the heater plate of the mechanical mixer. To ensure a uniform distribution of heat and asphaltene-maltene components in the base bitumen, a thorough mixing process was carried out at 180 °C for 1-2 minutes at a speed of 1000 rpm prior to the addition of any additives. Then, RET was added and mixed for 2 hours at 1000 rpm at 180 °C. After this period, PPA was added and mixed for 30 minutes under the same conditions. Prepared samples were left in an oven at 180°C for a 90-minute curing process. Thus, the preparation process of the PMB was completed. These PMB preparation conditions were implemented based on other studies in the literature [24, 32].

Once the PMB samples were prepared, composite-modified bitumen samples were prepared by adding WCO to the PMB. Accordingly, to begin with, PMB samples were heated up to 150°C. Subsequently, WCO was added and mixed at 1000 rpm for 30 minutes. Thus, the preparation process of the modified bitumen samples used in the study was completed, and the coding of the related samples is given in Table 3.

Table 3 - Representation of samples.

Sample Content	Code
AC 50/70 bitumen	Base
AC 50/70 bitumen + 1.5% RET + 0.2% PPA	PMB
AC 50/70 bitumen + 1.5% RET + 0.2% PPA + 2% WCO	2W
AC 50/70 bitumen + 1.5% RET + 0.2% PPA + 4% WCO	4W
AC 50/70 bitumen + 1.5% RET + 0.2% PPA + 6% WCO	6W
AC 50/70 bitumen + 1.5% RET + 0.2% PPA + 8% WCO	8W

## 2.2. Aging of Bitumen Samples

In the construction of asphalt pavements, bitumen is exposed to high temperatures during mixing with aggregates, transportation, paving, and compaction. These high temperatures cause a change in the structure of the bitumen, which is known as short-term aging. On the other hand, during its service life, bitumen is subjected to repeated vehicle loads and climatic conditions. This effect also causes changes in the internal structure of the bitumen and is called long-term aging [18]. Generally, the aging phenomenon has a vital impact on the performance of bitumen. Therefore, short- and long-term aging of bitumen has also been considered in the PG system.

In the PG system, two methods are used to simulate the aging of bitumen: rolling thin-film oven (RTFO) and pressure aging vessel (PAV) tests. In the RTFO test, bitumen weighing  $35\pm 0.5$  g is placed in tubes specified in the ASTM D2872 [33] standard and aged for 85 minutes at 163 °C. Besides, following the ASTM D6521 [34] standard in the PAV test, RTFO-aged bitumen weighing  $50\pm 0.5$  g placed in test containers is aged for 20 hours at 100 °C under 2.10 MPa pressure.

### **2.3. Dynamic Shear Rheometer (DSR) Test**

Bitumen shows viscous behavior at high temperatures and long loading periods, whereas it shows elastic behavior at low temperatures and short loading periods. It shows viscoelastic behavior between low and high temperatures, a combination of these two behaviors. The DSR test, conducted following the AASHTO T 315 [35] standard, is widely used to investigate the viscoelastic behavior of bitumen. This test determines the elastic (storage modulus,  $G'$ ) and viscous (loss modulus,  $G''$ ) components of bitumen as well as the phase angle ( $\delta$ ), which is a measure of the lag in the response of the material to the applied shear stress ( $\tau$ ). In the PG system, the complex shear modulus ( $G^*$ ), which is the resultant of elastic and viscous components, and  $\delta$  were used as the fundamental parameters to evaluate the performance of the bitumen. In fact, the limit values of the specification were determined based on these parameters.

In the PG system, the  $G^*/\sin \delta$  value is associated with the rutting of the pavement at high temperatures, and it is specified to be at least 1.0 kPa for unaged bitumen and at least 2.2 kPa for RTFO-aged bitumen. Additionally,  $G^* \cdot \sin \delta$  value was associated with the fatigue cracking formed at intermediate temperatures, and it is specified to be at most 5000 kPa for PAV-aged bitumen [36].

### **2.4. Multiple Stress Creep and Recovery (MSCR) Test**

The AASHTO M 332 [20] standard, which can be considered as a technically advanced version of the PG system, is believed to better characterize the performance-related properties of bitumen at elevated temperatures than the PG standard [37]. Through the MSCR test, which is the fundamental test of the relevant standard, the behavior of bitumen can be examined both at high strains and under repeated loading. This eliminates the problem of lack of complete activation of the polymer networks of PMBs, which was investigated at low strains in the DSR test [19]. Therefore, more realistic investigations of the behavior of PMB can be performed.

MSCR test is carried out, following the AASHTO T 350 [38] standard, at the high-temperature level that the pavement will be exposed to; in other words, the temperature remains constant. If the high-temperature performance requirement of bitumen is PG 58 according to the Superpave method, tests are carried out at 58 °C. After determining the test temperature, shear stresses are applied to the bitumen, and performance evaluation is made with the stress and strain relations. Stress is applied to the bitumen for 1 second, and strains are allowed to recover for 9 seconds. A total of 10 loading periods, one of which is 10 seconds, are applied (Figure 1). Besides, the effect of stress differences is evaluated by performing the test at two different stress levels: 0.1 and 3.2 kPa. The first ten periods applied at a stress of 0.1 kPa are called the conditioning phase. The evaluation is made by taking the average values obtained from the following ten periods.

In this test, two main parameter is calculated by using Equation (1), (2), (3), and (4). These parameters are nonrecoverable creep compliance ( $J_{nr}$ ) and percent recovery (R%). Studies conducted in accelerated loading facilities showed that the  $J_{nr}$  parameter is highly correlated with rutting in pavements [19]. On the other hand, the R% parameter is an important

parameter related to the elastic response of bitumen and indicates how much of the strain that occurs after loading can be recovered.

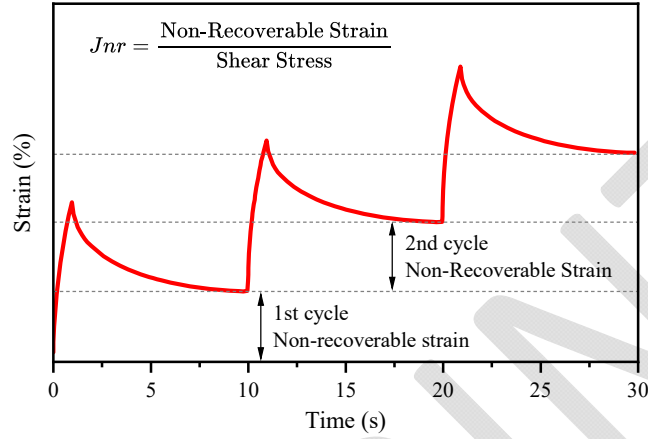


Figure 1 - Strain-time relationship in MSCR test (redrawn based on [39]).

$$Jnr_{0.1} = \frac{\sum_{n=11}^{20} Jnr(0.1, N)}{10} \quad (1)$$

$$Jnr_{3.2} = \frac{\sum_{n=21}^{30} Jnr(3.2, N)}{10} \quad (2)$$

$$R\%_{0.1} = \frac{\sum_{n=11}^{20} R\%(0.1, N)}{10} \quad (3)$$

$$R\%_{3.2} = \frac{\sum_{n=21}^{30} R\%(3.2, N)}{10} \quad (4)$$

where,  $Jnr_{0.1}$  is the nonrecoverable creep compliance at 0.1 kPa stress level,  $Jnr_{3.2}$  is the nonrecoverable creep compliance at 3.2 kPa stress level,  $R\%_{0.1}$  is the percent recovery at 0.1 kPa, and  $R\%_{3.2}$  is the percent recovery at 3.2 kPa.

Table 4 - Traffic-related grading in the MSCR system [20].

Grading	Traffic Conditions		Jnr <sub>3.2</sub> (kPa <sup>-1</sup> )
	Volume (ESALs)	Speed (km/h)	
Standard traffic, S	< 3x10 <sup>6</sup>	> 70	< 4.5
Heavy traffic, H	> 3x10 <sup>6</sup>	< 70	< 2.0
Very heavy traffic, V	> 10x10 <sup>6</sup>	< 20	< 1.0
Extremely heavy traffic, E	> 30x10 <sup>6</sup>	< 20	< 0.5

In the AASHTO M 332 [20] standard, a traffic grade is defined in addition to the temperature grades in the PG system by establishing a relationship between bitumen performance and the traffic conditions to which the pavement is exposed. Four different grades are specified as standard (S), heavy (H), very heavy (V), and extremely heavy (E). Between traffic conditions of these grades and nonrecoverable creep compliance at a stress level of 3.2 kPa ( $Jnr_{3.2}$ ), the relationship given in Table 2 was established. It is clear from Table 2 that the specification limits are set for the  $Jnr_{3.2}$  value of the bitumen, and as the  $Jnr_{3.2}$  value decreases, the traffic conditions that the pavement can withstand become more severe.

It is possible to evaluate the stress sensitivity of bitumen through MSCR tests conducted at different stress levels. Accordingly, the AASHTO M 332 [20] specification defines the parameter  $Jnr_{diff}$  in this regard. Additionally, besides the  $Jnr_{3.2}$  limits for traffic classification, an upper value for the  $Jnr_{diff}$  parameter has also been set. Therefore, it is stated that the  $Jnr_{diff}$  value calculated through Equation (5) can be at most 75%. In this way, the stress sensitivity of the bitumen is intended to be kept below a certain level.

$$Jnr_{diff} = \frac{(Jnr_{3.2} - Jnr_{0.1})}{Jnr_{0.1}} \cdot 100 \quad (5)$$

Another characteristic of bitumen that can be examined by the MSCR test is the presence of a polymer network. Based on this aspect, in the AASHTO M 332 [20] standard, a relationship has been established between R% and Jnr values. Accordingly, a curve is defined in the R%-Jnr graph, and the position of the bitumen value with respect to this curve is examined. It was stated that a bitumen value above this curve can indicate the presence of an elastomeric polymer in the bitumen sample [20]. Subsequently, Anderson [40] suggested that R% should be taken as 55% for the part of the curve in the standard where Jnr is less than 0.1. In another study, Salim et. al. [41] proposed that the relation between R%-Jnr should be represented by another relation according to the sample set in their study. The curves in the standard and the literature are given together in Figure 2. The presence of a polymer network in the bitumen samples in this study was analyzed using this figure.

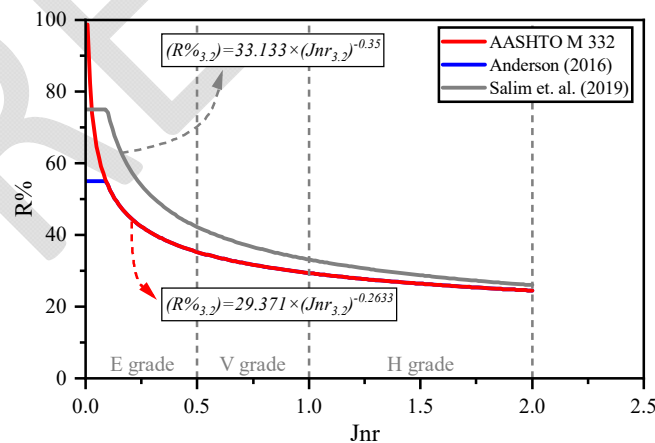


Figure 2 -  $R\%_{3.2}$  -  $Jnr_{3.2}$  relationship.



## 2.5. Bending Beam Rheometer (BBR) Test

BBR is a test applied to PAV-aged bitumen samples according to ASTM D6648 [42] standard and is associated with the low temperature performance of asphalt pavements. Two basic bitumen-related parameters are obtained from the test: creep stiffness ( $S(t)$ ) and creep rate (m-value). The deflection of a bitumen beam of a given size at a given time ( $t$ ) is measured, and the  $S(t)$  of the bitumen is calculated using Equation (6) from classic beam theory. On the other hand, the creep rate is related to the change of  $S(t)$  with the time ( $t$ ). For practical reasons, the software of the BBR device establishes a logarithmic relationship between  $S(t)$  and  $t$  in the form of Equation (7). The m-value, which is the slope of the  $\log S(t)$  -  $\log t$  graph at a given time  $t$ , can be calculated using Equation (8), which is the derivative of Equation (7) with respect to  $\log t$ .

$$S(t) = \frac{PL^3}{4bh^3\delta(t)} \quad (6)$$

$$\log S(t) = A + B \log t + C(\log t)^2 \quad (7)$$

$$m(t) = \left| \frac{d[\log S(t)]}{d(\log t)} \right| = B + 2C \log t \quad (8)$$

where:  $S(t)$  is the flexural creep stiffness at time  $t$  in MPa,  $m(t)$  is the creep rate at time  $t$ ,  $P$  is the measured test load in mN,  $L$  is the span length in mm,  $h$  is the depth of the specimen in mm,  $\delta(t)$  is the deflection of test specimen at time  $t$ , and  $A$ ,  $B$ ,  $C$  are regression coefficients.

At low temperatures where the BBR test is conducted, the bitumen stiffens and loses its elasticity. Thus, bitumen with high stiffness at low temperatures is more prone to crack formation [18]. Therefore, in the Superpave bitumen specification, an upper limit of 300 MPa is imposed on the  $S(t)$  to keep the bitumen stiffness below a certain level [36]. However, it is not entirely appropriate to evaluate the susceptibility of bitumen to crack formation at low temperatures solely based on its creep stiffness. Because bitumen is a viscoelastic material, it can relax the applied stress [21]. This stress relaxation ability is associated with the m-value in the BBR test. A relatively high m-value means that the stiffness of bitumen changes rather quickly and hence indicates better stress relaxation ability [21]. Considering this fact, a lower limit of 0.300 for the m-value was imposed in the Superpave bitumen specification to ensure that the bitumen has a sufficient stress relaxation feature [36]. Thus, bitumen with an m-value above the limit is assumed to be capable of quickly dissipating the stresses caused by the thermal changes.

When determining the low-temperature PG of the bitumen, the limit values given in the specification for the two parameters obtained from the BBR tests are considered. However, since the PG specification was introduced in the 1990s, it has been a matter of debate whether the low-temperature grade is governed by the  $S(t)$  or the m-value [21]. This is related to whether the behavior of the bitumen is  $S(t)$ -controlled or m-controlled.  $S(t)$ -controlled bitumen reaches the 300 MPa limit at temperatures higher than the m-value limit. Conversely, m-controlled bitumen reaches the 0.300 limit at a higher temperature than the  $S(t)$  limit [21]. Based on this information, various studies have been carried out, and different parameters

have been produced in which both  $S(t)$  and m-value are considered simultaneously. Among these parameters,  $\Delta T_c$  and  $\lambda$ , which can be calculated quite practically, have been included in this study. Thus, the behavior of the prepared samples at low temperatures was examined in detail through different parameters.

The  $\lambda$  parameter was first used by Liu et al. in the performance evaluation of crumb rubber-modified bitumen [23]. By simultaneously using the two parameters obtained from the BBR test as  $\lambda = S(t)/m$ -value, they performed a low-temperature performance assessment. Accordingly, they stated that as the  $\lambda$  value decreases, the low temperature performance of bitumen increases. Subsequently, Liu et al., in another study [43] on establishing a relationship between low-temperature performance parameters, determined the existence of a physical equation between creep stiffness and m-value in the form of  $m$ -value/ $S(t)$ . Based on this established relation, they stated that the parameter  $m$ -value/ $S(t)$  could be used as a promising material property to evaluate the low temperature performance of bitumen. Furthermore, they noted that this parameter is particularly suitable for bitumen where it is challenging to select a low-temperature performance grade based on the  $m$ -value or  $S(t)$ .

In the two different studies mentioned above, the researchers established two inverse equations, one of which is  $S(t)/m$ -value and the other is  $m$ -value/ $S(t)$ , between the low-temperature parameters. This situation, which only changes the result numerically, has caused a dilemma among researchers. Therefore, in some studies [12, 44, 45],  $S(t)/m$ -value value was used to evaluate low-temperature performance, while in others [46-48]  $m$ -value/ $S(t)$  value was used. Although this preference does not bring any technical differences, the use of the  $\lambda = S(t)/m$ -value parameter is preferred in this study as it expresses the numerically obtained data in a more easily understandable way. Accordingly, a decrease in  $\lambda$  value indicates an increase in the low temperature performance of bitumen.

The  $\Delta T_c$  concept was introduced in a project conducted by the Airfield Asphalt Pavement Technology Program (AATP) [49]. This project aimed to develop new methods to determine whether asphalt pavements at airports require maintenance. One of the outputs of the project is the  $\Delta T_c$  parameter. Subsequently, Anderson et al. [22] presented a summary of the project and described how  $\Delta T_c$  should be calculated. The  $\Delta T_c$  value can be defined as the difference between the temperature at which  $S(t)$  reaches the limit value and the temperature at which the  $m$ -value reaches the limit value. Accordingly, Anderson et al. [22] proposed the use of Equation (9) and Equation (10), obtained by interpolating between two different test temperatures, to calculate the critical temperature values. Therefore, the  $\Delta T_c$  value can be calculated as in Equation (11).

$$T_{c,S(t)} = T_1 + \frac{(T_1 - T_2) \times (\log 300 - \log S_1)}{\log S_1 - \log S_2} \quad (9)$$

$$T_{c,m} = T_1 + \frac{(T_1 - T_2) \times (0.300 - m_1)}{m_1 - m_2} - 10 \quad (10)$$

$$\Delta T_c = T_{c,S(t)} - T_{c,m} \quad (11)$$

where:  $S_1$  is the creep stiffness at  $T_1$  in MPa,  $S_2$  is the creep stiffness at  $T_2$  in MPa,  $m_1$  is the creep rate at  $T_1$ ,  $m_2$  is the creep rate at  $T_2$ ,  $T_1$  is the temperature in °C at which  $S(t)$  and m-value passes, and  $T_2$  is the temperature at which  $S(t)$  and m-value fails in °C.

While m-controlled bitumens have lower  $\Delta T_c$  values, their stress relaxation ability is limited. Therefore, a relatively low  $\Delta T_c$  means a high cracking potential [50]. Anderson et al. [22] proposed two different critical  $\Delta T_c$  values for the regional conditions in their study: a warning limit of -2.5 °C and a cracking limit of -5.0 °C. Furthermore, the  $\Delta T_c$  value has also been used to evaluate the hardening of bitumen due to reclaimed asphalt pavement (RAP), reclaimed asphalt shingle (RAS), re-refined engine oil bottoms (REOB) and various additives [50]. Such that, the  $\Delta T_c$  value has also found its place in standards such as AASHTO PP 78 [51]. Understandably, the concept of  $\Delta T_c$  is a growing concept that continues to be studied.

### 3. RESULTS AND DISCUSSION

#### 3.1. Rutting Behavior of Bitumen Samples

Within the framework of this study, MSCR tests were carried out at different temperatures, and the  $Jnr$  and  $R\%$  data of the modified bitumen were collected. The variation of the  $Jnr$  values is given in Figure 3. When the results at 0.1 kPa stress level are examined, it is seen that the addition of WCO to PMB causes an increase in  $Jnr_{0.1}$  values at all temperatures. This increase is more distinct at high temperatures. Consequently, the rise in WCO reduces the permanent deformation resistance of the bitumen at low stress levels. PMB gives the lowest  $Jnr_{0.1}$  values at all temperatures and has the highest rutting resistance. Also, 2W and 4W outperform the Base at all temperatures. Similarly, at 3.2 kPa stress level, the addition of WCO to PMB causes an increase in  $Jnr_{3.2}$  values at all temperatures. At 58 °C, this increase in  $Jnr_{3.2}$  values becomes more pronounced after 6% WCO addition, whereas at 64 °C and 70 °C, it becomes more pronounced after 4% WCO addition. As a result, it was found that more than 4% WCO addition makes PMB more susceptible to permanent deformations than Base at high temperatures. Besides, even the addition of 2% WCO increases the  $Jnr_{3.2}$  values of PMB by approximately two times at all temperatures.

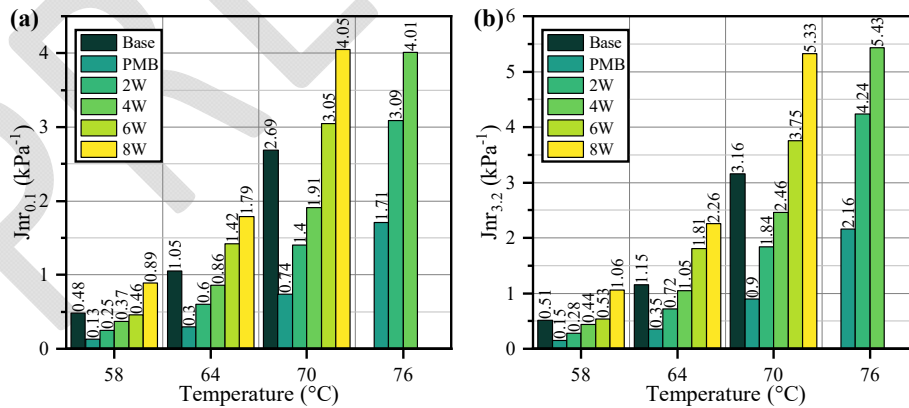


Figure 3 - Change of  $Jnr$  values with the temperature at different stress levels: (a) 0.1 and (b) 3.2 kPa.

To the authors' knowledge, since MSCR results for RET+WCO modified bitumen cannot be found in the literature, it is impossible to directly compare the results for RET+WCO modified bitumen with any other study in the literature. However, to confirm the compatibility of the findings of this study with the literature, studies with RET-modified bitumen or WCO-modified bitumen were examined separately in the literature, and comparisons were made. Accordingly, it has been shown in the literature that the addition of RET reduces the Jnr value of base bitumen [26, 52]. In addition, it has been observed in the literature that the addition of WCO increases the Jnr values of both base bitumen and pre-modified bitumen with other additives [53-55].

If the effect of temperature on Jnr is examined in Figure 3, it is seen that Jnr values increase with increasing temperature, and as expected, the rutting resistance of bitumen decreases. This change in Jnr values with the temperature can also be observed in other studies in the literature for RET or WCO-modified binders [26, 52, 56]. Additionally, PMB is least affected by temperature increase. On the other hand, the 2W and 4W samples outperform the Base at all temperatures. Increasing the temperature from 58 °C to 70 °C increases the Jnr values of the samples by 5-6 times.

The variation of the R% values is given in Figure 4. When the results at 0.1 kPa stress level are examined, it is seen that the addition of WCO to PMB causes a decrease in R%<sub>0.1</sub> values at all temperatures. At 58 °C, this decrease in R%<sub>0.1</sub> values becomes more pronounced after 6% WCO addition, whereas at temperatures higher than 64 °C, it becomes more pronounced after 2% WCO addition. Additionally, the Base gives the lowest R%<sub>0.1</sub> values at all temperatures. Besides, the 8W has 2.6, 3.0, and 3.4 times more R%<sub>0.1</sub> values than Base at 58 °C, 64 °C, and 70 °C, respectively. Similarly, at 3.2 kPa stress level, the addition of WCO to PMB causes a decrease in R%<sub>3.2</sub> values at all temperatures. The R%<sub>3.2</sub> of the Base decreases significantly with stress and temperature increase. Also, 8W has approximately four times more R%<sub>3.2</sub> value than Base. When the results of the R% values are compared with the results in the literature, it is seen that the addition of RET increases the R% [26, 52, 56] of the base bitumen, and the addition of WCO decreases [55]. Therefore, the results obtained from this study are in line with the studies in the literature.

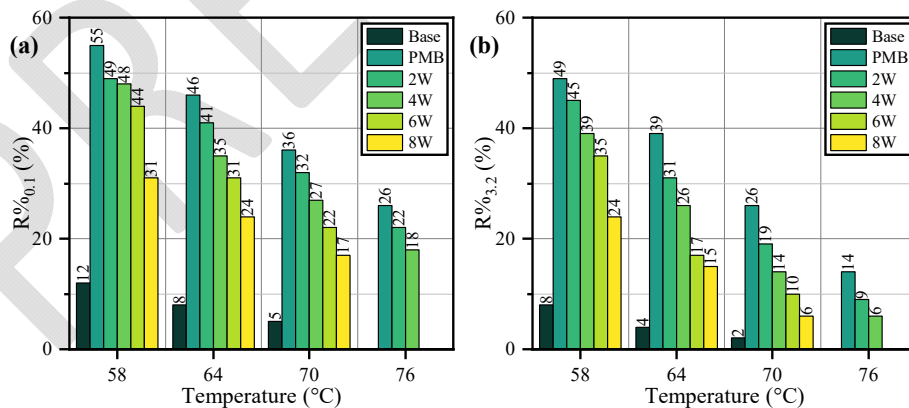


Figure 4 - Change of R% values with the temperature at different stress levels: (a) 0.1 and (b) 3.2 kPa.

When the effect of temperature on R% is examined in Figure 4, it is seen that the elastic properties of the Base significantly decrease with increasing temperature, similar to the studies in the literature [55]. For bitumen containing WCO, this decrease is not as much as in the Base. The R% values of all binders decrease linearly with increasing temperature. The increase in temperature from 58°C to 76°C causes the R% values of PMB to reduce by 34% and 46% at 0.1 kPa and 3.2 kPa stresses, respectively. These values are 34% and 56% for the 2W. It can be said that the decrease in the elastic properties of bitumen containing WCO with increasing temperature is similar to that of PMB.

With the parameters obtained from the MSCR test, traffic grading of bitumen for different temperatures was done, and the results are given in Table 5. The  $G^*/\sin \delta$  values obtained from DSR tests and the corresponding PG are also given in this table. The Base is suitable for very heavy traffic conditions at 58 °C and heavy traffic conditions at 64 °C. The addition of the RET to the Base makes the bitumen suitable for extremely heavy traffic conditions at 58 and 64 °C, very heavy traffic conditions at 70 °C, and standard traffic conditions at 76 °C. Adding 2% and 4% WCO to the PMB does not change the traffic grades of the binder at 58 °C. Still, adding 2% and 4%WCO to the PMB reduces its traffic grade to very heavy traffic conditions at 64 °C. On the other hand, the 6W has the same traffic grade as the Base, while

Table 5. High temperature performance grading of the samples.

Sample	Test temp. (°C)	As per AASHTO M 320		PG	As per AASHTO M 332		
		$G^*/\sin \delta$ (kPa)			$Jnr_{3,2}$ (kPa <sup>-1</sup> )	$Jnr_{diff}$	PG
		Unaged	RTFO-Aged				
Base	58	4.854	-	PG64	0.51	6.25	PG58-V
	64	2.090	4.777		1.15	9.52	PG64-H
	70	8.21	-		3.16	17.47	
PMB	58	8.482	-	PG76	0.15	15.38	PG58-E
	64	4.274	-		0.35	16.67	PG64-E
	70	2.214	-		0.9	21.62	PG70-V
	76	1.186	2.268		2.16	26.32	PG76-S
2W	58	5.560	-	PG70	0.28	12.00	PG58-E
	64	2.825	-		0.72	20.00	PG64-V
	70	1.480	2.757		1.84	31.43	PG70-H
4W	58	3.815	-	PG70	0.44	18.92	PG58-E
	64	1.992	-		1.00	22.09	PG64-V
	70	1.087	2.320		2.46	28.80	PG70-S
6W	58	2.631	-	PG64	0.53	15.22	PG58-V
	64	1.425	2.961		1.81	27.46	PG64-H
	70	0.785	-		3.75	22.95	
8W	58	1.555	4.139	PG58	1.06	19.10	PG58-H

8W is suitable for traffic conditions one grade lower than the Base. It has been determined that adding more than 6% WCO to PMB falls behind the Base in terms of both high-temperature performance level and traffic grading. In this context, it is recommended that the modification that will withstand very heavy traffic conditions at 64 °C and will not adversely affect the performance of the PMB can be obtained by adding 4% WCO to the PMB.

As mentioned in Chapter 2.4, it is possible to evaluate the stress sensitivity of the bitumen with the MSCR test. The  $J_{nr\_diff}$  parameter is examined in this regard. From Table 5, it can be checked whether the bitumen samples exceed the specification upper limit of 75%. Accordingly, all samples in the study were below the specification limit. Therefore, they will exhibit stable behavior by not showing a significant reaction to the stress changes in the traffic. Furthermore, for a relative evaluation of the effect of both temperature and additives on the stress sensitivity of bitumen,  $J_{nr\_diff}$  data are presented in Figure 5. However, a regular variation between  $J_{nr\_diff}$  values and additive content was not found. On the other hand, the Base is the most sensitive bitumen to stress changes, and adding WCO to PMB slightly increases the stress sensitivity of the PMB.

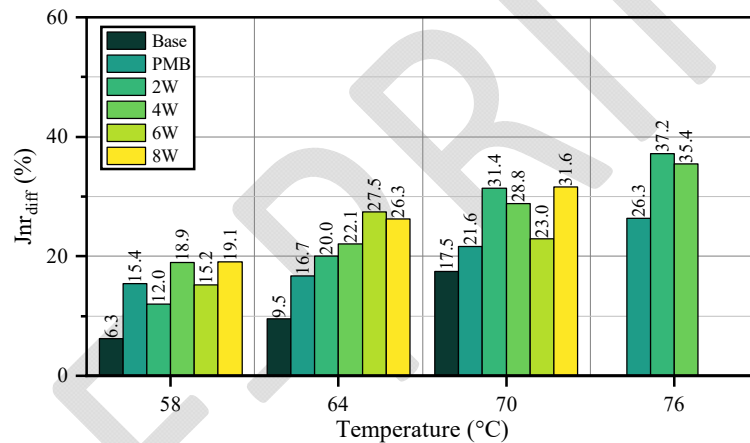


Figure 5 - Change of  $J_{nr\_diff}$  values at different test temperatures.

As a final evaluation of the data obtained from the MSCR test, the presence of the polymer network was examined. Accordingly, the  $J_{nr_{3.2}}$  and  $R_{0_{3.2}}$  data of bitumen samples at 58 °C and 64 °C were plotted on two separate graphs for different temperatures. The values for the bitumen samples were compared with the reference curves shown in Figure 2. The graphs of the evaluation are given in Figure 6. When Figure 6 is examined, it is seen that the data of the samples in the study are below the curve proposed by Salim et al. [41] at both temperatures. However, it has been shown in many studies that a polymer network is formed thanks to the chemical interaction of the functional groups in RET with asphaltenes [10, 25, 26]. Therefore, the value of PMB is expected to be above the reference curve. Thus, it is considered that the curve proposed by Salim et al. [41] is not suitable for evaluating the modified bitumen samples in this study. On the other hand, when the data of the bitumen samples are compared with the reference curve proposed by AASHTO M 332 [20] or Anderson [40], it is seen that the PMB sample remains above the curve for both temperatures.

This result meets the expectations and confirms the modification of the bitumen. At 58°C, all samples except the Base and 8W remained above the curve, indicating good elastic properties. However, it is seen that the polymer network in the binder cannot resist the increase in temperature, and there is a decline in its elastic properties. Adding more than 2% of WCO to the PMB binder in regions with a pavement temperature higher than 58 °C will adversely affect the elastic properties. It is expected that adding WCO, which has the effect of softening, to the bitumen will reduce the temperature resistance. As a result, it can be said that the limit temperature value at which the elastic properties begin to deteriorate is 58 °C for the WCO-added PMB.

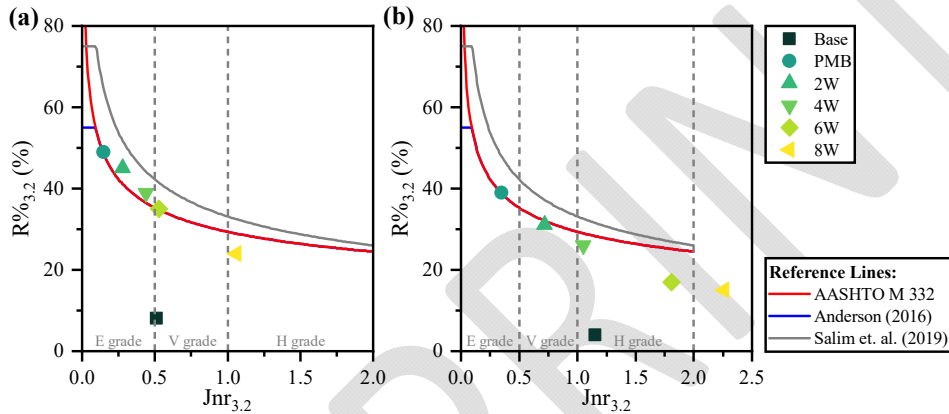


Figure 6 - Comparison of  $Jnr_{3,2}$  and  $R\%_{3,2}$  values of bitumen samples with reference curves for polymer network detection at different temperatures: (a) 58 and (b) 64 °C.

### 3.2. Low-temperature Cracking Behavior of Bitumen Samples

The  $S(t)$  and  $m$ -values of bitumen samples obtained from the BBR test are given in Table 6. When Table 6 is examined, it is seen that the addition of RET to the Base does not significantly affect the low-temperature performance. However, when WCO was added to PMB, the low-temperature performance increased with the increasing amount of WCO. Therefore, WCO-added PMB may be suitable in regions where cold climatic conditions prevail.

In order to investigate the behavior of bitumen at low temperatures in more detail, methods where  $S(t)$  and  $m$ -value are evaluated simultaneously, are included in this study as mentioned before. Accordingly, at first, the  $\lambda$  values of the bitumen samples at different test temperatures were calculated by dividing the  $S(t)$  by the  $m$ -value. The results are given in Figure 7. At all test temperatures, the  $\lambda$  values of the Base decreased with the addition of RET. For a RET-modified PMB, it was impossible to support this behavior with a study in the literature directly. On the other hand, a study for an SBS-modified PMB reported an increase in  $\lambda$  values compared to base bitumen [57]. Based on this result, it can be said that the RET additive will have a more positive effect on the low-temperature performance of the base bitumen than the SBS additive. Besides, when WCO was added to the PMB, a decrease in  $\lambda$  values was observed for all test temperatures. This decrease in  $\lambda$  values indicates that adding WCO to PMB will contribute to its low-temperature performance.

Table 6 - BBR test results and low-temperature performance grades of the bitumen samples.

Sample	Test temp. (°C)	Creep Stiffness $S(t) \leq 300$ MPa	m-value $m \geq 0.300$	Low-temperature PG As per AASHTO M 320
Base	-12	250.76	0.2996	PG -22
	-18	564.22	0.2089	
	-24	-	-	
PMB	-12	216.67	0.3140	PG -22
	-18	548.45	0.2280	
	-24	-	-	
2W	-12	152.89	0.3404	PG -22
	-18	402.56	0.2626	
	-24	609.10	0.2227	
4W	-12	103.59	0.3884	PG -28
	-18	247.37	0.3252	
	-24	469.14	0.2552	
6W	-12	56.40	0.4112	PG -28
	-18	119.69	0.3450	
	-24	303.98	0.2864	
8W	-12	37.03	0.4240	PG -34
	-18	71.18	0.3213	
	-24	266.98	0.3135	

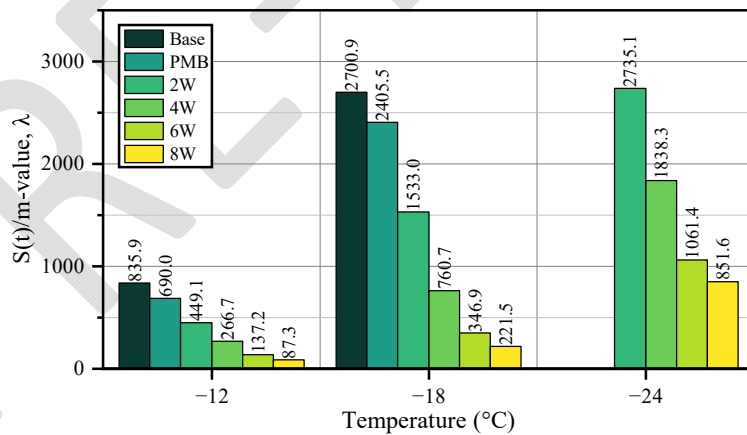


Figure 7 -  $\lambda$  values of the bitumen samples obtained from BBR tests performed at -12, -18, and -22 °C.



$\Delta T_c$  is the other parameter used in this study to investigate the low temperature behavior of bitumen samples in detail. Accordingly, the  $\Delta T_c$  values were calculated with the results obtained from the BBR test with the help of Equations (9), (10), and (11). However, unlike the traditional  $\Delta T_c$  calculation method, in this study, different combinations for  $T_1$  and  $T_2$  temperatures were used at the initial stage while calculating the  $\Delta T_c$  values. Accordingly, the results obtained are given in Table 7 for different temperature binaries. Since the critical values for Base and PMB samples were reached between -12 and -18 °C, BBR tests were not performed at a third test temperature. Therefore, different temperature binaries could not be created for these samples, and  $\Delta T_c$  calculation was performed by the conventional method. On the other hand, since BBR tests were carried out at three different temperatures for WCO-added PMB samples, it was possible to calculate  $\Delta T_c$  with different temperature binaries. When Table 7 is examined, it is understood that the selected temperature binary significantly affects the  $\Delta T_c$  value. In fact, when the 8W sample is evaluated, it exhibits m-controlled behavior between -12 °C and -18 °C and S(t)-controlled behavior between -18 °C and -24 °C. When the -12 °C and -24 °C range is considered as a whole, a result close to a balanced behavior is obtained. Similarly, for the other samples, it is seen that the temperature binary selected in the  $\Delta T_c$  calculation has a significant effect on the result obtained. Regarding this situation, Anderson et al. [22] stated that to determine the critical temperatures,  $T_1$  and  $T_2$  temperatures should be selected so that one meets the specification limit and the other does not. Subsequently, the critical temperature should be determined by interpolation in this range. They also stated that extrapolation could be used in cases where the critical temperature does not fall between the selected temperature binary. However, they recommended not to use extrapolation as much as possible.

Table 7 - Calculations of the  $\Delta T_c$  values at two different test temperatures.

Sample	$\Delta T_c$	Temperature binaries ( $T_1, T_2$ )		
		(-12, -18)	(-18, -24)	(-12, -24)
Base	-1.33	-	-	-
PMB	-1.13	-	-	-
2W	-1.06	-	-1.36	-1.73
4W	1.09	-	0.35	-0.48
6W	-3.25	-	-1.31	-1.21
8W	-11.96	-	9.86	0.76

Given the significant influence of the temperature binary on the  $\Delta T_c$  calculation, it is thought that this influence may be due to the equations used in the  $\Delta T_c$  calculation being derived by assuming that the effect of temperature variation on bitumen is linear. Therefore, in this study, different types of curve fitting were applied to the results obtained from BBR tests conducted at three different test temperatures to obtain the curve that simulates the behavior of bitumen with temperature change in the closest way. Accordingly, 32 trend lines with four different types (linear, logarithmic, power, and exponential) were generated for the  $S(t)$  and m-values of four samples. The properties of the generated trend lines are given in Table 8.

Table 8 - Determination of trend line type for the calculation of  $\Delta T_c$ .

Sample	Parameter	Trend Line Properties			Selected Critical Temp.
		Type	Equation	$R^2$	
2W	$T_{c,S(t)}$	Linear	$y = 0.05 \cdot x + 1.6242$	0.9492	-27.060
		Logarithmic	$y = 0.8773 \cdot \ln x + 0.0234$	0.9833	-26.390
		Power	$y = 0.9108 \cdot x^{0.3558}$	0.9733	-26.640
		Exponential	$y = 1.745 \cdot e^{0.0202 \cdot x}$	0.9327	-27.340
	$T_{c,m}$	Linear	$y = -0.0098 \cdot x + 0.4518$	0.9666	-25.490
		Logarithmic	$y = -0.171 \cdot \ln x + 0.7635$	0.9926	-25.037
		Power	$y = 1.5603 \cdot x^{-0.614}$	0.9992	-24.670
		Exponential	$y = 0.5122 \cdot e^{-0.035 \cdot x}$	0.985	-25.280
4W	$T_{c,S(t)}$	Linear	$y = 0.0547 \cdot x + 1.376$	0.9923	-30.130
		Logarithmic	$y = 0.9455 \cdot \ln x - 0.3356$	0.9999	-29.587
		Power	$y = 0.7332 \cdot x^{0.4077}$	0.9991	-29.808
		Exponential	$y = 1.5362 \cdot e^{0.0235 \cdot x}$	0.9836	-30.331
	$T_{c,m}$	Linear	$y = -0.0111 \cdot x + 0.5227$	0.9991	-30.063
		Logarithmic	$y = -0.19 \cdot \ln x + 0.864$	0.9839	-29.461
		Power	$y = 1.7353 \cdot x^{-0.595}$	0.9668	-29.103
		Exponential	$y = 0.5975 \cdot e^{-0.035 \cdot x}$	0.992	-29.685
6W	$T_{c,S(t)}$	Linear	$y = 0.061x + 1.0067$	0.9962	-34.105
		Logarithmic	$y = 1.0391 \cdot \ln x - 0.8584$	0.9748	-34.779
		Power	$y = 0.5032 \cdot x^{0.4982}$	0.9878	-34.510
		Exponential	$y = 1.2339 \cdot e^{0.0291 \cdot x}$	0.9999	-33.940
	$T_{c,m}$	Linear	$y = -0.0104 \cdot x + 0.5347$	0.9988	-32.567
		Logarithmic	$y = -0.179 \cdot \ln x + 0.8577$	0.9961	-32.548
		Power	$y = 1.4968 \cdot x^{-0.516}$	0.9876	-32.531
		Exponential	$y = 0.5914 \cdot e^{-0.03 \cdot x}$	0.9997	-32.634
8W	$T_{c,S(t)}$	Linear	$y = 0.0715 \cdot x + 0.6622$	0.9632	-35.384
		Logarithmic	$y = 1.2024 \cdot \ln x - 1.4791$	0.9178	-36.850
		Power	$y = 0.332 \cdot x^{0.615}$	0.9477	-36.254
		Exponential	$y = 0.9966 \cdot e^{0.0364 \cdot x}$	0.9831	-35.014
	$T_{c,m}$	Linear	$y = -0.0092 \cdot x + 0.5187$	0.8027	-33.772
		Logarithmic	$y = -0.166 \cdot \ln x + 0.825$	0.8742	-33.633
		Power	$y = 1.2678 \cdot x^{-0.452}$	0.9012	-34.255
		Exponential	$y = 0.5498 \cdot e^{-0.025 \cdot x}$	0.8361	-34.231

Equations were determined for each curve, and the critical temperatures ( $T_{c,S(t)}$  and  $T_{c,m}$ ) of the samples were calculated by using these equations. From the four different critical temperatures calculated, the one with the highest  $R^2$  value of the relevant equation, in other words, the one that most closely represents the behavior of bitumen with respect to temperature change, was selected. Table 8 shows that the selected trend line type varies depending on the bitumen sample and the investigated parameter. For example, the 2W sample shows a logarithmic behavior with respect to  $S(t)$ , while it shows an exponential behavior with respect to the m-value. On the other hand, the 4W sample exhibits logarithmic behavior with respect to the  $S(t)$  and linear behavior with respect to the m-value. Hence, the behavior of bitumen with temperature change can be represented by a unique trend line for each sample and parameter. Therefore, the traditional calculation method, in which this behavior is assumed to be linear, is considered unable to reflect the actual behavior of the bitumen sample completely.

After the procedures mentioned above, the selected critical temperatures and the  $\Delta T_c$  values calculated using Equation (11) are given in Table 9. When the obtained results are compared with Table 7, it is seen that there are significant differences in  $\Delta T_c$  values especially for WCO-added bitumen. Therefore, when evaluating WCO-added PMBs, using trend lines formulated by utilizing the results of tests conducted at three different temperatures, as in this study, will provide more realistic results.

Table 9 - The final  $\Delta T_c$  values obtained according to the calculations made in this study.

Sample	$T_{c,S(t)}$	$T_{c,m}$	$\Delta T_c$
Base	-23.33	-22.00	-1.33
PMB	-24.10	-22.98	-1.13
2W	-26.39	-24.67	-1.72
4W	-29.587	-30.06	0.48
6W	-33.94	-32.63	-1.31
8W	-35.014	-34.26	-0.76

In order to compare the effects of RET and WCO additives on the  $\Delta T_c$  parameters of bitumen samples, Figure 8 was prepared. When this figure is examined, first of all, it is seen that the addition of the RET to the base bitumen does not significantly affect the  $\Delta T_c$  parameter. Accordingly, it is possible to say that both base and PMB samples show a behavior close to the balanced behavior in terms of  $S(t)$ - or m-controlled behavior. On the other hand, the  $\Delta T_c$  results from PMBs are controversial. While there are studies in the literature where the  $\Delta T_c$  values of PMBs are very low [58], there are also studies with similar results [21] as in this study. With the addition of WCO to the PMB, no regular change was observed in  $\Delta T_c$  values. Generally, it is possible to say that PMBs with WCO addition show a balanced behavior. However, with  $\Delta T_c$  values, observing the low-temperature performance improvement provided by WCO is not entirely possible, which is clearly seen in the PG system and is well-accepted in the literature. As a result, this may be seen as a disadvantage of the  $\Delta T_c$  parameter.

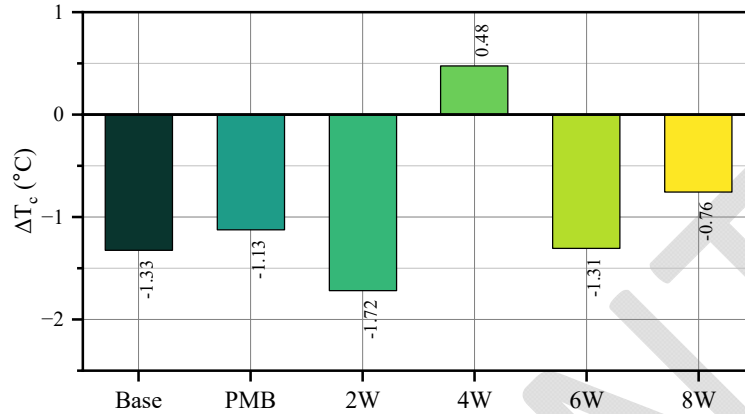


Figure 8 - The difference between the critical temperature found with the  $m$ -value and the critical temperature found with the  $S(t)$  value;  $\Delta T_c$ .

#### 4. CONCLUSIONS

In this study, 50/70 penetration grade base bitumen was modified with 1.5% reactive ethylene terpolymer (RET) and 0.2% polyphosphoric acid, and then 2%-8% waste cooking oil (WCO) was added. The effects of using WCO and RET on the high and low-temperature performance of modified bitumen were investigated with the MSCR and BBR tests. The results obtained from the experimental study are listed below:

- The addition of WCO to the PMB adversely affected the rutting resistance. With the addition of 8% WCO to the PMB, the  $Jnr_{0.1}$  and  $Jnr_{3.2}$  values increased by 586% and 607%, respectively.
- The elastic recovery performance of the PMB decreased with the increase in WCO content. With the addition of 8% WCO to the PMB, the  $R\%_{0.1}$  and  $R\%_{3.2}$  values decreased by 43.6% and 51%, respectively.
- Adding RET to base bitumen increased the  $S(t)$  and decreased the  $m$ -value. Accordingly, considering the Superpave performance criteria, the addition of RET negatively affects the low-temperature performance of base bitumen. However, the  $\lambda$  and  $\Delta T_c$  values of base bitumen decrease with the addition of RET. When these two parameters are evaluated, it can be said that adding RET improves the low-temperature performance of base bitumen by increasing its stress relaxation capacity.
- As mentioned above, the effect of RET addition on the low temperature of base bitumen varies depending on the parameter under consideration. Accordingly, novel parameters such as  $\lambda$  and  $\Delta T_c$ , rather than Superpave criteria, are considered more appropriate for evaluating the low-temperature performance of RET-modified bitumen.
- In parallel with adding WCO to PMB,  $S(t)$  values increased and  $m$ -values decreased. Additionally, with the addition of RET to PMB, the  $\lambda$  value decreased at all test

temperatures. These results show that adding WCO has significantly improved the low-temperature performance of PMB.

- When the low-temperature performance of bitumen samples was examined according to the  $\Delta T_c$  parameter, no apparent trend was observed. All of the prepared samples showed balanced behavior regarding  $S(t)$  or m-controlled behavior.

In line with the conclusions obtained from this study, it was determined that adding low amounts of WCO to 50/70 penetration-graded bitumen with 1.5% RET positively affected both the rutting and low-temperature performance of the base bitumen. Therefore, it has been determined that bitumen containing RET and low amounts of WCO can serve well under high and low-temperature conditions that pavements may be exposed to in many parts of Türkiye [59, 60]. In future studies, detailed experimental studies using bitumen containing RET and WCO in mix design will be useful in evaluating the behavior of RET and WCO in the asphalt mixture.

#### Acknowledgments

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