DOKUZ EYLÜL UNIVERSITY

GRADUATE SCHOOL OF NATURAL AND APPLIED SCIENCES

SHORT AND LONG TERM AGING OF BITUMINOUS HOT MIXTURES

by

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October, 2020

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SHORT AND LONG TERM AGING OF BITUMINOUS HOT MIXTURES

A Thesis Submitted to the
Graduate School of Natural and Applied Sciences of Dokuz Eylül University In Partial Fulfillment of the Requirements for the Degree of Master of Science in Civil Engineering, Transportation Engineering

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İZMİR

M.Sc THESIS EXAMINATION RESULT FORM

We have read the thesis entitled **"SHORT AND LONG TERM AGING OF BITUMINOUS HOT MIXTURES"** completed by **ÇAĞLA DURMAZ** under supervision of **PROF. DR. BURAK ŞENGÖZ** and we certify that in our opinion it is fully adequate, in scope and in quality, as a thesis for the degree of Master of Science.

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SHORT AND LONG TERM AGING OF BITUMINOUS HOT MIXTURES

ABSTRACT

The major function of the road is to provide a good ride quality and serviceability during its service life. The predicted performance and service life of the asphalt pavement depend largely on the properties of bitumen used in the mixtures.

Increases in traffic volume, production errors and heavy climatic conditions cause deformations which are directly related to the aging of the bitumen. Aging is described as the hardening, loss of volatile components and oxidation of the bitumen. In previous researches, the bitumen has been aged separately and then added to the asphalt mixture, but in reality bitumen ages gradually in the asphalt mixture. In this study, the bituminous mixture aging was performed instead of bitumen aging in order to represent the aging in the field in the best possible way.

This thesis aims to evaluate the aging procedures of hot mix asphalt with the current standard (AASHTO R30) (Standard Practice For Mixture Conditioning of Hot Mix Asphalt Standard) as well as alternative methods (NCHRP 09-52, NCHRP 09-54 and RILEM) by way of indirect tensile strength test.

The level of aging has also been compared with the samples taken from recently constructed pavement surface and from the five years old pavement surface. Results depicted that, laboratory aging methods revealed the field aging properties on the unaged bitumen.

Keywords: Asphalt, oxidation, short term aging, long term aging, indirect tensile strength

BİTÜMLÜ SICAK KARIŞIMLARIN KISA VE UZUN DÖNEM YAŞLANDIRILMASI

ÖZ

Yolun ana işlevi, hizmet ömrü boyunca iyi bir sürüş kalitesi ve servis kolaylığı sağlamaktır. Asfalt kaplamanın öngörülen performansı ve hizmet ömrü, büyük ölçüde karışımlarda kullanılan bitümün özelliklerine bağlıdır.

Trafik hacmindeki artışlar, üretim hataları ve ağır iklim koşulları, bitümün yaşlanmasıyla doğrudan ilişkili deformasyonlara neden olur. Yaşlanma, bitümün sertleşmesi, uçucu bileşenlerin kaybı ve oksidasyonu olarak tanımlanır. Önceki araştırmalarda, bitüm ayrı olarak yaşlandırılmış ve daha sonra asfalt karışımına eklenmiştir ancak gerçekte asfalt karışımı içerisinde bitüm yaşlanmaktadır. Bu çalışmada, sahadaki yaşlanmayı en iyi şekilde temsil etmek için bitüm yaşlandırması yerine bitümlü karışım yaşlandırılması yapılmıştır.

Bu tez, sıcak karışım asfaltın yaşlandırma prosedürlerini mevcut standart (AASHTO R30) ve alternatif yöntemler (NCHRP 09-52, NCHRP 09-54 ve RILEM) ile değerlendirmeyi amaçlamaktadır. Yaşlanma etkisini incelemek üzere yaşlandırılmamış ve laboratuvar ortamında kısa ve uzun dönem yaşlandırılmış asfalt numuneleri üzerinde dolaylı çekme mukavemeti deneyleri gerçekleştirilmiştir. Asfalt numunelerinin yaşlanma seviyeleri daha sonra yaşlanma indeksleri kullanılarak değerlendirilmiştir. Yaşlanma seviyesi, yakın zamanda inşa edilen kaldırım yüzeyinden ve 5 yıllık kaplama yüzeyinden alınan örneklerle de karşılaştırılmıştır. Sonuçlar, laboratuvar yaşlandırma yöntemlerinin, yaşlandırılmamış bitümde sahada gerçekleşen yaşlanma özelliklerini ortaya çıkardığını göstermiştir.

Anahtar kelimeler: Asfalt, oksidasyon, kısa dönem yaşlanma, uzun dönem yaşlanma, dolaylı çekme mukavemeti

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CHAPTER ONE
INTRODUCTION

Asphalt has been used as a paving material in Europe since the 1850s and the hardening or stiffening of asphalt has been investigated since around 1900 in America (Krchma et al., 1991; Croney et al., 1974).

Asphalt roads have an important place in road transportation due to the increase in traffic demand over the years. Asphalt road should be able to meet this demand and provide smooth and comfortable travel, so it is important to keep road conditions as good as possible. The parameter that most affects the durability of the asphalt road is aging. The phenomenon known as aging makes the bitumen more viscous, sensitive and stiffer, resulting in deformations that accelerate asphalt deterioration. Asphalt aging or hardening is categorized as short-term aging and long-term aging depending on time.

Aging mechanism has two components: (Bell et al. ,1994)

1-Short term aging (STA)

2-Long term aging (LTA)

During storage of the mixture, mixing in plant, transporting to construction site, laying and compaction short term aging happens. This is probably thought to be caused by volatilization. Long term aging, which is considered predominantly to be caused by oxidation, occurs during its service life and while exposing it to the environment (Bell et al. ,1994) It is seen that, approximately 70% of oxidation occurs during short term aging process. Oxidation decelerates during the long term aging process and is only around 30% in 8 years. Rapid aging occurs during the preparation, laying and compaction of the mixture, which slows down at low temperatures during service life of the road.

Simulation of aging of asphalt and its mixture has been investigated over the years and various standards have been adopted for the aging behavior of the hot mix asphalt.

The present standard procedure to represent aging of bituminous mixtures is the AASHTO R30 protocol. According to AASHTO R30, mixture samples should be aged for 5 days at 85°C after they are conditioned to short-term aging for 4 hours at 135 °C. However, this standard protocol has only one temperature and different environmental circumstances or mixture feautures aren't taken into consideration. Therefore several researchers have conducted a study to develop a better simulation process for the hot mix asphalt.

In the thesis, the listed methods: American Association of State Highway and Transportation Officials R30 [AASHTO R30]; National Cooperative Highway Research Program 09-52 [NCHRP 09-52], National Cooperative Highway Research Program 09-54 [NCHRP 09-54] and The International Union of Laboratories and Experts in Construction Materials 206ATB [RILEM] methods were followed for short term aging and long term aging procedures. Laboratory specimens were divided into 3 groups under the names of unaged specimens, short term aged specimens and long term aged specimens. Indirect Tensile Strength test was applied to the specimens taken from the recently constructed and 5 years aged specimens field as well as to the laboratory prepared specimens to evaluate the aging behavior as well as to determine whether the current standard for aging is still valid.

CHAPTER TWO
FAILURES OF ASPHALT PAVEMENTS

The increase in axle loads, traffic jams, construction failures and environmental circumstances causes deformations. The commonly encountered failures such as rutting, fatigue and low temperature cracking and moisture susceptibility reduce the predicted performance of the roads. The deformations which were seen in bituminous pavements were examined and divided into groups by many researchers. The Strategic Highway Research Program (SHRP) A-417 (1994), classifies the deformations into 4 categories as: permanent deformations (rutting), fatigue cracking, low temperature cracking and moisture damage. In 2001, Brown examined aging in three categories, excluding aging from the general classification as follows:

1-Permanent Deformation

Rutting

Collapses

Swelling

Ondulation

2-Cracking

-Fatigue Cracking

-Low Temperature Cracking

3-Moisture Damage

In this study, above classification will be used because Brown's study is the most common deformation classification (Şengöz, 2003). Since the aging phenomenon constitutes the subject of this thesis, it is examined in detail as a separate section from the other deformations.

2.1 Permanent Deformation

Rutting is the most encountered kind of deformation in bituminous mixtures, which is considered very crucial both technically and economically. Permanent deformation is described as the collection of minor amounts of irrecoverable deformations in each implementation of the load acting on the pavement surface (Topal, 2001). These deformations are particularly problematic in countries with rough terrain, heavy traffic conditions and high temperatures such as Turkey.

Rutting in flexible pavement can be seen in two groups: due to sub layers or bituminous layers.

Although the use of stiff materials on the base and the subbase course partially reduces rutting due to the sub layers, this is considered as a structural deformation rather than material-related deformation. The unexpected weakening of the bituminous layer due to moisture may cause such deformation. Causes of deformation due to sub layers in general: depending on the insufficiency of sub layers thickness or the consolidation of the subgrade.

The rutting due to the sub layers is caused by excessive repeated stresses in the base or subbase layers beneath the asphalt layer. This rutting is usually seen in pavements that are not designed for real traffic conditions. It may also result from the use of low quality material, poor compaction of the material, poor drainage, and failure to take precautions against freezing and thawing effects. Figure 2.1 indicates the rutting caused by decreased air voids in the asphalt concrete layer from weak sub layers (Asphalt Institute, 2001).

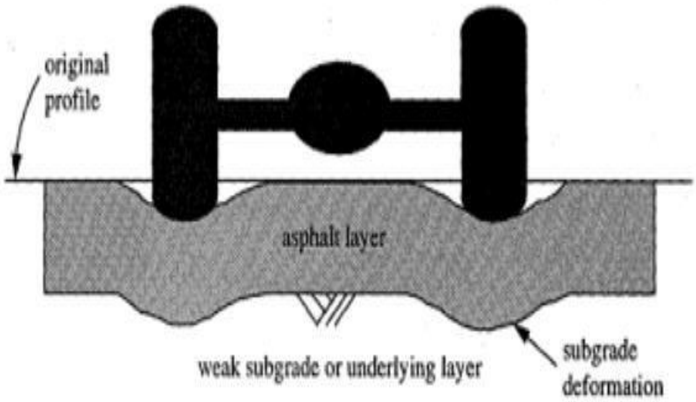

Figure 2.1 Rutting due to sub layers (Asphalt Institute, 2001)

The other type of rutting is seen in the bituminous mixture layers. It is called the flow rutting. This sort of rutting emerges in bituminous pavements which has low shear strength under repeated heavy loads. Swellings occur around the rutting. Rutting occurs due to insufficient bituminous mixture under the high pavement temperatures during the summer on the mixture surface.

Asphalt mixture researchers are commonly interested in the deformation such as rutting of the asphalt layers. It occurs due to repeated heavy loads. Figure 2.2 symbolizes the rutting caused by the asphalt mixture properties from weak mixture (Asphalt Institute, 2001).

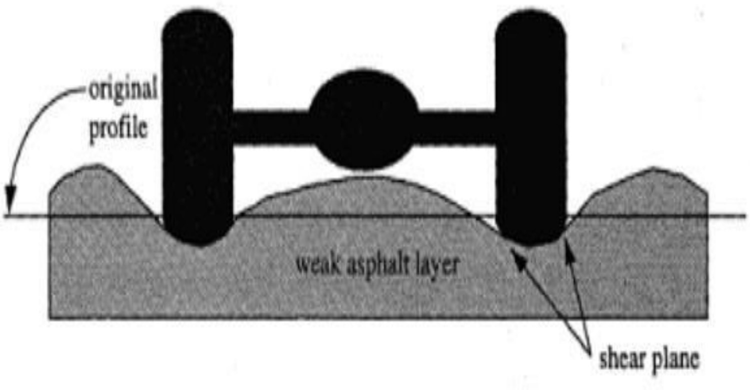

Figure 2.2 Rutting due to bituminous layers (Asphalt Institute 2001)

Although rutting is known as a bituminous problem, considering the bitumen and mineral aggregate together would be much more accurate in determining the rutting formation (Ağar & Umar, 1985).

Figures 2.3 and 2.4 demonstrate typical forms of rutting after long and short periods.

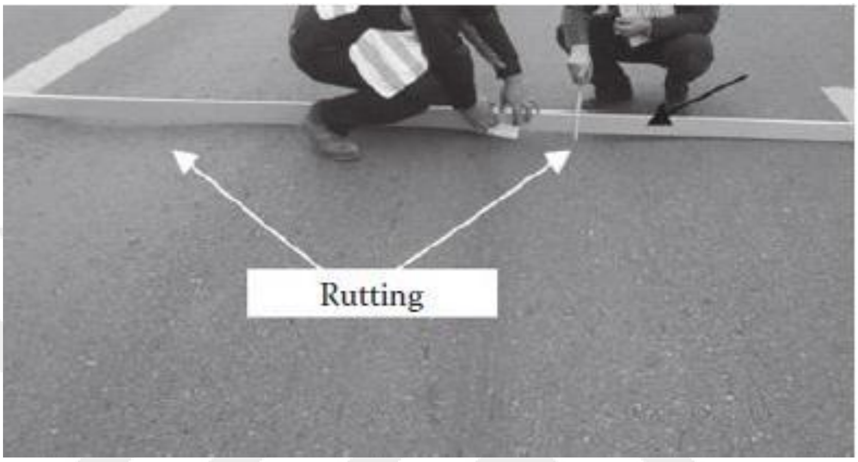

Figure 2.3 Typical rutting usually formed after a long period (Nikolaides, 2014)

Figure 2.4 Typical rutting quite often formed very early (Nikolaides, 2014)

Hot mix characteristics and road conditions affecting the rutting are given in Table 2.1.

Table 2.1 Factors causing rutting of asphalt mixtures (SHRP A415, 1994)

	Factor	**Change in Factor**	**Effect of Change in Factor on Rutting Resistance**
Aggregate	Surface Texture	Smooth to rough	Increase
	Gradation	Gap to continuous	Increase
	Shape	Rounded to angular	Increase
	Size	Increase in maximum size	Increase
Binder	Stiffness	Increase	Increase
Mix	Binder content	Increase	Decrease
	Air void content*	Increase	Decrease
	VMA	Increase	Decrease
Test or Field Conditions	Temperature	Increase	Decrease
	State of stress/strain	Increase in tire contact pressure	Decrease
	Load repetitions	Increase	Decrease
	Water	Dry to wet	Decrease if mix is water sensitive

In order to reflect the degree of deterioration of a pavement, the term Rutting Depth is used in many countries. Maintenance, coating and reinforcement methods are determined according to this depth. The deterioration assessment according to the ruth depth varies from country to country, but in most countries, it ranges from 1.0 to 2.5 cm. For example, in the UK, if the rut depth reaches 1 inch (2.5 cm) from the original pavement surface or 0.7 inches (1.3 cm) from the smooth edge at a distance of 2 meters from the shoulder edge, this means that the pavement failed as illustrated in Figure 2.5. (Uluçaylı, 1976).

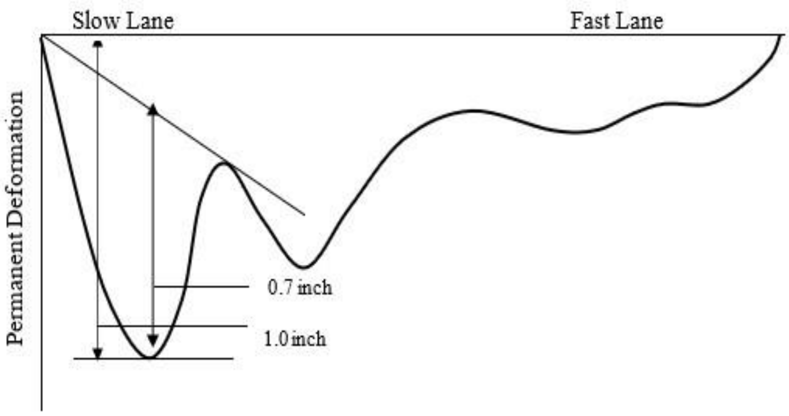

Figure 2.5 Permanent deformation condition in the U.K. (Uluçaylı, 1976)

As oxidation increases, asphalt concrete becomes more brittle and becomes more sensitive to cracking.

2.2 Cracking

The deformations that occur on the surface of the pavement with specific shape, through the impacts regarding traffic and environmental conditions are known as cracks. Cracks are classified into two important categories:

2.2.1 Fatigue Cracking

The fatigue cracks start with cracks under the pavement structure. As the compacted asphalt mixture exceeds the tensile strength, the crack formed under the asphalt pavement structure increases with repeated loads and spreads to the pavement surface. The highest tensile stress on the pavements occurs at the bottom of the pavement layer, in the direction of travel and in the range of two wheels as illustrated in Figure 2.6.

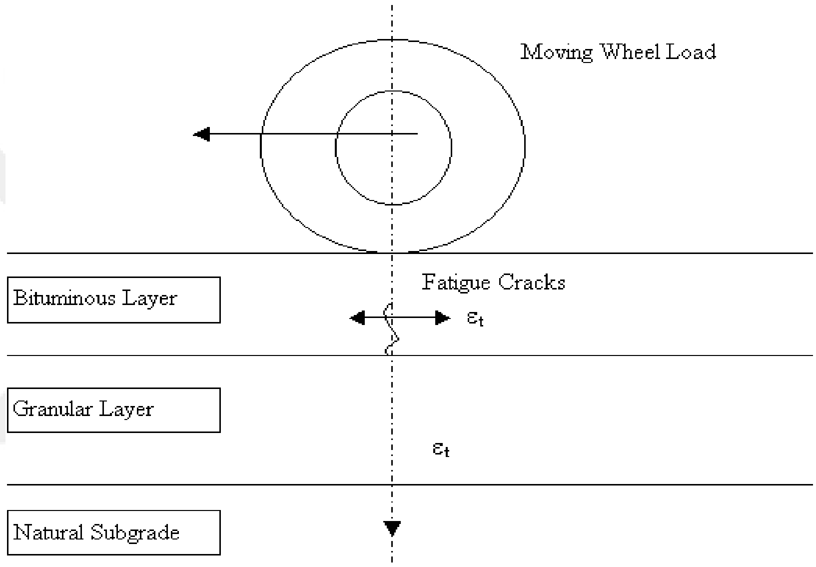

Figure 2.6 Fatigue cracking in bituminous pavements (Brown, 1980)

These types of cracks are connected to each other and occur in the whole or specific part of the pavement in alligator appearance and in blocks of different sizes. Fatigue cracks caused by settling or as a result of exceeding fatigue strength are formed irregularly along the axis of the road in the form of capillary cracks. With the effect of traffic, these irregular cracks gradually grow and then combine with the transverse cracks and turn into alligator appearance in blocks. Alligator cracks caused by settlement are formed as cornered and seen irregular. The alligator cracks caused by fatigue are generally uniform, smaller in size and seen as honeycomb.

The oxidation of the bitumen affects the fatigue properties of the bituminous mixtures. Oxidation increases the cohesion and the stiffness of the mixture. As a result of this, fatigue cracks are seen especially on thin bituminous pavement surfaces, although the formation of rutting is reduced (Uluçaylı, 1976).

The cracks arise from the traffic load, insufficient subgrade or as a consequence of the pavement deterioration. Tensile strength is seen under the pavement structure. When tensile stress exceeds, the tensile strength of the asphalt mixture cracks arise as illustrated in Figure 2.7. Cracks occur as a consequence of repeated loading. As illustrated in Figure 2.8, it appears randomly across the entire pavement. Lack of drainage may also cause cracks to occur. Alligator cracks are the most common deterioration in asphalt pavements with parking facilities in all deteriorations. The reason for the difficulty in providing drainage in the parking lots is the deterioration caused by the subgrade and base saturation (Lavin, 2003).

Figure 2.7 Fatigue cracking (Lavin, 2003)

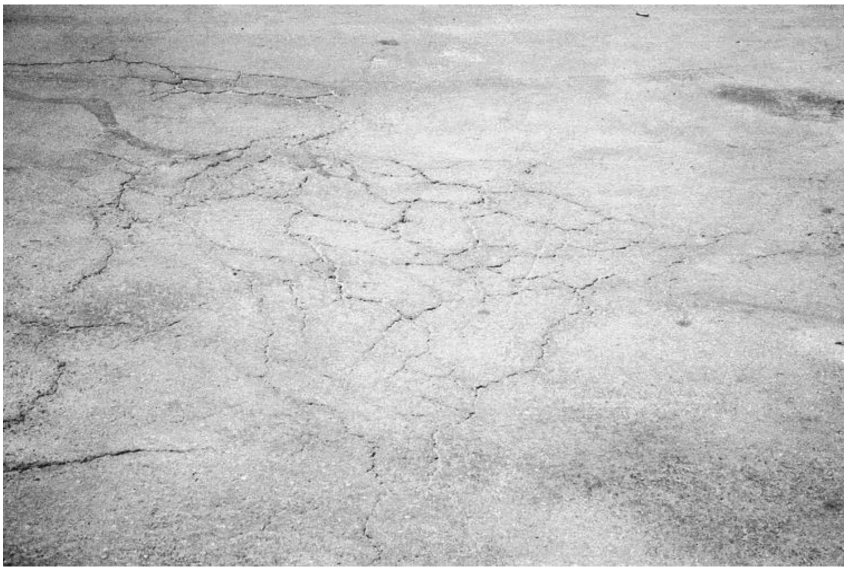

Figure 2.8. Subgrade failure (Lavin, 2003)

Low severity, medium severity and high severity fatigue cracks according to their severity are shown in Figure 2.9, 2.10, 2.11, respectively (Federal Highway Administration [FHWA], 2009).

Figure 2.9 Fatigue cracks with high severity (FHWA, 2009)

Figure 2.10 Fatigue cracks with medium severity (FHWA, 2009)

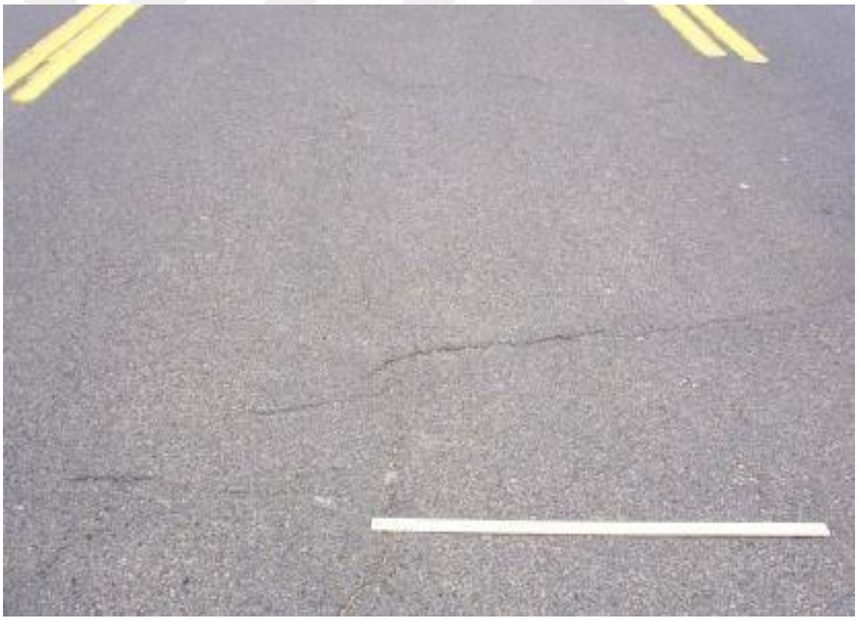

Figure 2.11 Fatigue cracks with low severity (FHWA, 2009)

Generally, fatigue cracking indicates that the bituminous pavement has reached a sufficient amount of design load implementations. Therefore, it is worn out and a planned improvement should be made. If fatigue crack arises when the pavement completes its service life, it is assumed to be the normal phase of the designment of the pavement. However, fatigue cracks seen before the service life may indicate that the pavement is exposed to more traffic than expected. As a result, the best ways to prevent fatigue cracking are as follows:

- Taking heavy traffic loads into account during the design
- Use materials that will not weaken with moisture.
- Use thicker pavements.
- Well compaction should be accomplished during the construction
- The content of the optimum bitumen should be higher
- More dense gradation should be used.
- More soften bitumen should be used (Uluçaylı, 1976; Asphalt Institute, 1996).

2.2.2 Low Temperature Cracking

As the air temperature decreases, bituminous mixtures try to shrink. As a result, the frictional force formed in the layer under the bituminous mixture prevents shrinkage of the bituminous mixture.

The researchers stated that bituminous mixtures had low temperature cracking properties on aggregate type, bitumen type, aging degree and air void ratio. Adding soft bitumens and rough aggregates in the asphalt mix causes a raise in the resistance of the asphalt mix to low temperature cracking. It is stated that there is a relationship between the bitumen stiffness and cracks at low temperatures. (Al-Qadi & Wang, 2009).

Such cracks are a serious problem during mixing; because they are difficult to evaluate and predict. The reasons for this difficulty are the aging characteristic and viscoelastic properties of the binder.

Low severity, medium severity and high severity; low temperature cracks according to their severity are shown in Figure 2.12, 2.13, 2.14, respectively (FHWA, 2009).

Figure 2.12 Low temperature cracks with low severity (FHWA, 2009)

Figure 2.13 Low temperature cracks with medium severity (FHWA, 2009)

Figure 2.14 Low temperature cracks with high severity (FHWA, 2009)

According to the studies, cracking temperature can be reduced by using low temperature sensitivity (high penetration index) bitumen. The soft bitumen is a better choice than the hard bitumen but is problematic at high temperatures (Benedetto & La Roche, 1998).

2.3 Moisture Damage

Slurry,which is seen in asphalt pavements, can be described as the weakening of the chemical bond between the bitumen and the aggregate in the presence of water is considered as a factor affecting the bituminous pavement stability. (Stuart, 1990).

Among the problems encountered in flexible pavements, consisting of aggregate and bitumen, moisture-associated damage plays a crucial role. This type of deterioration begins under the pavement layer or between the surface and binder layers with the highest water content and is seen as deep pits on the road surface, fatigue cracks and separation of aggregate grains from the road surface.

- Moisture damage of mixtures is examined in three classes:
- Deformations due to the decrease in the bitumen-aggregate bond (adhesive defects)
- Failures caused by the decrease in bitumen cohesion
- Degradation of aggregate particles due to environmental effects such as freezing (Terrel & Al-Swailmi, 1994).

Adhesive failure, as a result of the binding of the bitumen to lose the ability to adhere to aggregate grains, separation from the grain surface; cohesive failures appear as a consequence of the mass of the asphalt film, flowing due to external effects and fracture into thin film.

Brown (2001), stated that adhesion degradation without these two types of deformation occurs completely or partially in the intersection between bitumen and aggregate, and cohesion degradation is explained by rheological analysis of bitumen and aggregate node.

As the moisture sensitivity of the mixtures depends on the adhesion and cohesion ability of the bitumen, the researches are mostly focused on caused by weakening the asphalt-aggregate bond. Such failures are defined as slurry (Görkem, 2014).

CHAPTER THREE

DURABILITY

It is a feature that shows the asphalt mixture is able to resist the damaging air effects, water, temperature and traffic. These effects may include changes in asphalt properties (such as oxidation and evaporation), changes in the pavement and aggregate due to the water effect (freezing and thawing).

Durability generally improves with high asphalt content, dense (well-graded) aggregate gradation and well compacted, impermeable mixtures. An argument for increasing the amount of asphalt is that the asphalt film surrounding the aggregate grain becomes thicker. Thicker films provide increased resistance to aging and hardening. Another positive argument is that with the increase in the amount of asphalt, the size of the spaces that are interconnected from each other or the mixing and mixing process, the isolation of these pores make it more difficult for air and water to enter the asphalt mixture. However, thicker film is more prone to formation of rutting and deformation.

The mixture must include sufficient dose of asphalt in order to provide bitumen properties suitable to withstand the corrosive effects of traffic. Insufficient amount of asphalt can cause aggregate to break off the surface. If the asphalt has become brittle, wear may occur. If the asphalt is overheated in the hot mixing process, it will cause the asphalt to become brittle and worn out sooner.

A mixture with high asphalt content, whose voids are completely filled with asphalt, provides maximum durability, which is undesirable in terms of stability. When laid on the road, the pavement will groove and move under traffic. In this case, bleeding of the mixture can also be observed (Tunç, 2004).

3.1 Factors Affecting Durability

Dow (1903) first put forward the factors that affect the hardening of the bitumen and gradually lose its properties. In this study, weight and penetration properties of the bitumen recovered from hot mix asphalt were examined. Traxler (1961) examined the factors affecting the binding properties of the bitumen in four classes.

These are:

- Oxidation,
- Evaporation or loss of volatile substances,
- Polymerization (depending on active light),
- Concentration polymerization (depending on temperature).

Traxler stated that all the effects mentioned above have changed over time. In another study,there was an increase in factors, affecting the aging properties of the bitumen ,listing 15 of them (Traxler ,1963). Traxler's publication is based on empirical studies that support these effects. Later on, Petersen (1984) examined the factors related to aging of the mixtures in three basic classes:

- Reduction of oil particles in asphalt as a result of evaporation or absorption,
- Change in bitumen composition reacting with oxygen in the atmosphere
- Thixotropic effects of molecular structure.

Researchers, investigating aging and their effects have done studies based on the factors given by Petersen. Generally, the factors that lead to hardening of the bitumen over time are described below (Uluçaylı, 1998).

3.1.1 Volatilization

The volatile substances, which are found in the bitumen cause the bitumen to harden over time and / or temperature. If the bitumen is comparatively less volatile, the stiffening is expected to be less.

3.1.2 Oxidation

Oxidation is one of the significant factors for bitumen aging. Oxidation of bitumen due to the formation of organic molecules is defined as the coupling of the hydrocarbon components of the bitumen with the oxygen in the air (The Shell Bitumen Handbook, 1990). In this reaction, bitumen loses part of hydrogen and carbon.

If the chemical formula of bitumen is expressed as C_xH_y, these reactions are expressed as follows (Hunter, 1994).

$$C_xH_y + O \rightarrow C_xH_yO \quad (3.1)$$

$$C_xH_y + O \rightarrow CxH_{y-2} + H_2O \quad (3.2)$$

$$C_xH_y + 2O \rightarrow C_{x-1}H_y + CO_2 \quad (3.3)$$

The occurrence of these reactions leads to higher activation energy from top to bottom (heat and light). The first type of reaction occurs on the surface that is in contact with air and a shell is formed. If this shell is not damaged, it prevents the oxygen ingress and the oxidation event slows down. Hardening is delayed as the shell also prevents the volatile oils in the binder from evaporating. Therefore, the asphalt film on the aggregate grains is desired to be as thick as possible. However, it is necessary to estimate the optimum value of the film thickness to avoid excess bleeding and decrease in stability. (The effect of the thickness of the film, covering the aggregate grains on oxidation is included in the experimental studies). Oxidation process in refineries is done willingly to obtain “air blown asphalt”. This process is carried out in a controlled manner by blowing hot air into the residual asphalt in a special tank and a second type of reaction occurs. As a result of this reaction, the bitumen undergoes the following changes:

- Penetration value decreases,
- Softening point increases,
- Temperature susceptibility decreases (Uluçaylı, 1998).

3.1.3 Polymerization

Oxidation can be summarized with the reaction of oxygen and bitumen. The occurrence of this mechanism can be explained as follows; aging causes hardening of the surface, which creates surface cracks and enables oxygen to diffuse from these cracks which creates oxidation. Aromatics, resins and asphaltene fractions are affected by the oxidation, but not the saturates, according to Corbett & Mertz’s study, conducted in 1975 (Corbett & Merz, 1975). The amount of aromatics is reduced by aging since they turned into resins and then asphaltenes due to oxidation. As a result, it is possible to say that oxidation decreases the amount of aromatics and resins fraction, however this is just the opposite for the asphaltene fraction (Lesueur, 2009).

Polymerization is defined as the change of the hydrocarbon chain of the bitumen over time and the growth of molecules. These chains are thought to cause progressive hardening (Şengöz, 2003).

3.1.4 Thixotropy

Thixotropy is a very common phenomenon in bitumen. The bitumen gains rigidity over time when it is left at low temperature. This rigidity is not caused by evaporation or volatility of the volatile components of the bitumen, but by the colloidal character of the bitumen. By means of the colloidal structure, the bitumen gains a structure that resembles the sponge shape in cold weather, after a certain period of stagnation, in the form of a stretched web in three axes, which can only be seen in an electronic microscope. This structure is strengthened over time, giving the bitumen a stiffness in the face of external forces, similar to that of elastic bodies. However, this stiffness is lost as a result of a mechanical jolt. In order to achieve the same stiffness, the bitumen must be kept for a length of time. The rise in temperature also has effects equivalent to a mechanical shake on the thixotropy-dependent stiffness of the bitumen. Because of the temperature increase, the stiffness of the bitumen formed by micelles is disrupted and micelles begin to separate from each other. As a result, the bitumen loses its rigidity (Şengöz, 2003).

In summary, thixotropy is the ability of a colloidal melt to regain the same rigidity after a period of stagnation, providing that it loses its rigidity under mechanical or thermal effects and does not change all other physical conditions.

3.1.5 Seperation

Seperation is caused by the penetration of oily components into the mineral aggregate with a high degree of porosity from bitumen. Such hardening is a function of both the leaking inclination of the bitumen and the aggregate porosity. This causes hardening of the bitumen due to less oily components (Şengöz, 2003).

3.1.6 Syneresis

It is the occurrence of an oily asphalt film layer to the surface due to the change in the structure of asphalt (Şengöz, 2003).

3.2 Aging of Asphalt During Various Stages of Pavement Construction

The bitumen used in bituminous mixtures undergoes significant oxidation in processes carried out from the refinery until its start on the road. This greatly affects the bitumen's rheological behavior, performance, and durability (Şengöz, 2003).

During storage, the bitumen is in a large mass and can maintain its high temperature for days and weeks. During mixing with the aggregate, transportation and construction of the road, the bitumen has high temperature and thin film for a short period of time and a medium or low temperature film for a long period of service life (The Shell Bitumen Handbook, 1990).

The properties of the bitumen in these four cases are listed below.

3.2.1 Aging of Asphalt During Storage

Very little oxidation occurs in the bitumen tank at high temperature and in a large mass. The asphalt surface that is exposed to oxygen is very small compared to the whole mass. However, a certain amount of oxidation happens if the bitumen passes through the pipe on the tank and falls at a distance from the binder surface. This is because oxygen can easily react with asphalt because the asphalt surface falling from a pipe into the tank is larger. (The Shell Bitumen Handbook, 1990).

3.2.2 Aging of Asphalt Aging During Mixing

During the mixing period, all aggregate grains and fillers are covered with an asphalt film with a thickness of 5 to 15μ. In this case, the mixture in the mixer is very sensitive to aging caused by oxidation and evaporation. Aging during mixing should be taken into account for the selection of a suitable bitumen for the mixtures (Şengöz, 2003).

Aging during mixing with the aggregate depends on many reasons such as time of mixing, temperature and asphalt film thickness. This damage due to the mentioned factors can be minimized by controlling them carefully. The amount of bitumen and temperature control is particularly critical (The Shell Bitumen Handbook, 1990).

Figure 3.1 shows the aging index value depending on the film thickness. The index of aging is defined as the ratio of the aged bitumen viscosity to the unaged bitumen viscosity.

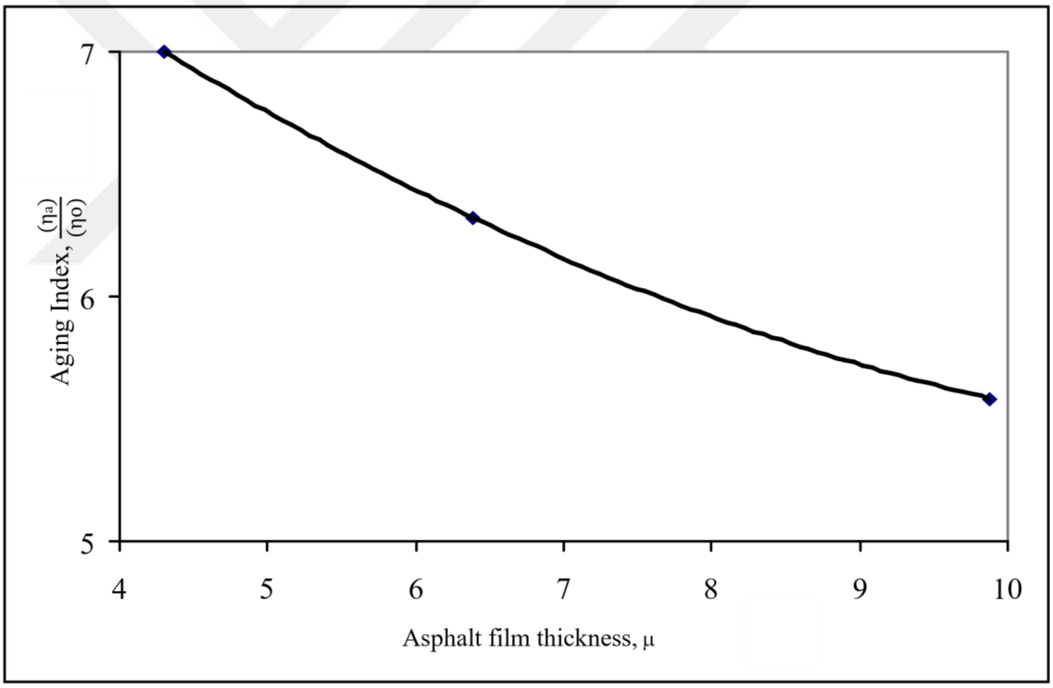

Figure 3.1 The graph of aging index depending on asphalt film thickness (Griffin et al., 1955)

As it is seen from the figure, the index of aging increases as the asphalt film thickness surrounding the aggregate grains decreases (Griffin et al., 1955).

The type of mixer that is used is one of the factors affecting aging during mixing. Studies have shown that aging is less in mixtures prepared in drum type mixers compared to mixtures prepared in five type mixers (Haas, 1973). This is the result of less mixing time in drum mixers. The study conducted by Shell Bitumen UK revealed that the total decrease in penetration was 5% to 15% lower and increase in the softening point was 4ºC less in the mixture prepared in a drum type mixer under equal mixing temperatures, compared to the mixture prepared in a batch type mixer (The Shell Bitumen Handbook, 1990).

3.2.3 Aging of Asphalt During Transporting to the construction site, laying and compaction

Aging during storage of the mix depends on the mixture temperature, asphalt film thickness and oxygen exposure time as mentioned in the previous sections. When a mixture is discharged into the storage silo, air enters and some of this air remains in the voids in the material. During storage, some of the oxygen in the air reacts with bitumen. However, if no additional oxygen is introduced into the silo, oxidation stops after a while.

The loading and unloading lids of the silos used for the storage of mixtures must be airtight. Otherwise, the silo will be working like a chimney, constantly passing air through it. As a result, the material oxidizes and cools.

In addition, the silo should be filled as far as possible so that there is no free air on the ceiling. Otherwise, the air remaining on the silo roof will cause reaction on the surface of the mixture. As a result of this reaction, carbon dioxide (CO_2) will be released and as the carbon dioxide is heavier than air, it will act as a cover in the upper part of the mixture and decrease the oxidation rate.

The study in the United States has revealed that additional oxidation will not occur during transportation and paving if the oxidation in the silo is limited with the incoming air (Brock, 1986). This is due to the fact that during loading into the vehicle there is no significant fresh air intake into the mixture. Thus, there will be little additional oxygen input for oxidation. Investigations have shown that if the mixture is poured directly from the mixer to the conveying means, the amount of hardening that will occur during transportation, is similar to the amount generated during silo storage (The Shell Bitumen Handbook, 1990).

3.2.4 Aging of Asphalt During Service Life

Figure 3.2 shows the years of aging index values of the bitumen. During short-term aging, approximately 70% of oxidation occurs; It is seen that oxidation slows down during long-term aging and occurs only around 30% in 8 years.

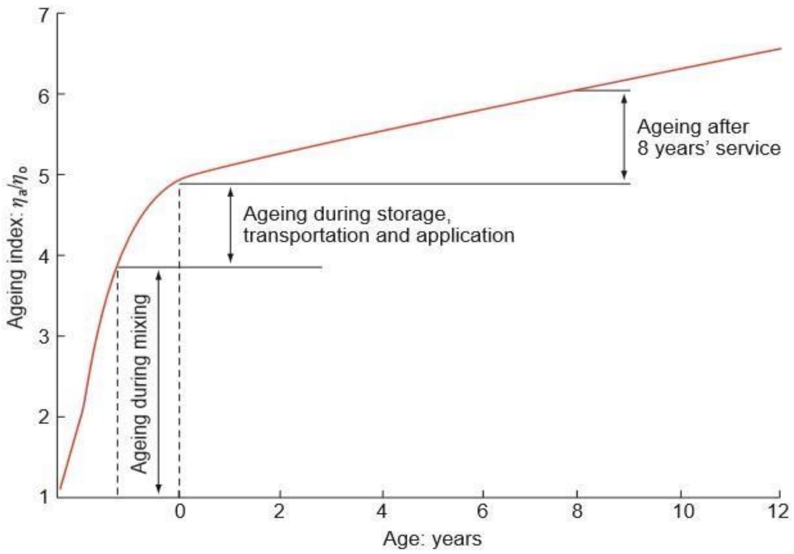

Figure 3.2 Relative viscosity increase after mixing with the aggregate (The Shell Bitumen Handbook, 1990)

As it can be understood from the figure, rapid aging occurs when preparing, laying and compacting the mixture, which slows down at low temperatures on the road.

Aging,which is observed during the service life depends on factors as the degree of compaction (air void ratio), the asphalt film thickness and permeability surrounding the grains of the aggregate (Şengöz, 2003).

3.3 Laboratory Aging of Bituminous Mixtures

Aging of bituminous mixture can be implemented in the laboratory by various methods. In this section, aging methods of bituminous mixtures will be examined under three main headings: oxidation, extended heating, ultraviolet/infrared treatment methods. After the improvement of the bitumen recycling process on mixtures, the researchers used the bitumen recycling method to explain the aging effects of the mixtures. The studies of the researchers on the mixtures are given below (Şengöz, 2003).

3.3.1 Extended Heating Methods

Before the extended heating methods were conducted, the mixture was exposed to extremely high temperature for a specified time.Then,appropriate experiments such as compression and recovered binder tests are done (Airey, 2003).

Pauls & Welborn (1952) aged 2 inches (50.4 mm) cylindrical sand mixtures of Ottawa in the forced draft oven for different periods at 163ºC and measured their compressive strength. They also determined the consistency of the bitumen recovered from the mixtures.

Plancher et al. (1976), 25 * 40 mm size of the mixture in the oven by aging, lime, bitumen evaluated the effects of oxidation-related aging properties. Experiments have shown that the modulus of elasticity of lime-doped mixtures varies less than that of non-doped mixtures.

Before recovering the bitumen Kemp & Predoehl (1981) conditioned the sand mixtures of Ottowa for 1200 hours at 60ºC in the forced draft oven. Since more bitumen is obtained than the sand mixtures of Ottowa, they preferred TODT for the aging of the bitumen.

The mixtures were aged in a forced draft oven at 100ºC by Hugo & Kennedy (1985). This process was carried through for 4 and 7 days in a dry and in the presence of 80% moisture. After the treatments, 4-inch (100 mm) cores were taken from the samples. Consistency tests were applied on the recovered bitumen. Von Quintas et al. (1988) used forced draft ovens to represent short-term aging. After the uncompacted mixtures were aged at 135ºC for 8, 16, 24 and 36 hours, viscosity and penetration tests were applied on the recovered bitumen and the conclusions were compared with the viscosity and penetration values of the original bitumen. Von Quintas et al. (1988), have also made research on the mixtures with long term aging properties. In addition, three compacted samples were subjected to pressurized oxidation at 60ºC under 100 psi pressure for five to ten days.

Von Quintas et al. (1988) suggested the method of aging by using an oven according to pressure oxidation as a result of their studies. In addition, he stated that the aging method should be studied more in the oven and the temperature values should be evaluated very well in order to choose the best method. In addition, most of the methods used in aging asphalt mixtures in the laboratory included aging compacted asphalt samples. But, Von Quintas et al. (1988) investigated to represent the short term aging of loose mixtures. In this procedure, the loose mixture was heated in a forced draft oven for 8, 16, 24 and 36 hours at 135°C. While this procedure presented aging levels similar to field levels, there was a significant distribution in laboratory data. Von Quintas et al. (1988) searched long term aging on asphalt mixes which was aged for 2 days at 60 ° C and then for 3 days at 107 °C. Bell (1989) claimed that the asphalt mixtures with high air void content can display sample deterioration because of higher temperatures.

The SHRP-A-003A project which is related to researches done by Von Quintas *et al*. (1988), involves developing short and long term aging methods. In 1994, laboratory experiments and field researches related to aging have been performed by Chris A. Bell et al. Within SHRP and the experimental methods were evaluated (Bell et al., 1994a, 1994b, 1994c). They proposed keeping the loose mixture in an oven for 4 hours at 135°C to represent the short term aging process. The process was performed to simulate the aging which occurs during preparation, laying and compaction of the mixture in the laboratory (Bell *et al*., 1994; Monismith *et al*., 1994). In addition, the mixture should be mixed hourly to age the mixture homogeneously in this process. To represent the long term aging procedure which occurs throughout the service life of the pavement, they proposed that the short-term aging mixture should be kept in an oven for 120 hours (5 days) at 85°C after compaction. Keeping the compacted mixture in the forced draft oven for 2 days at 100°C has the same effect, but it is preferable to work at low temperature, given that the sample may deform at high temperatures.

A short term aging procedure was improved by Scholz (1995) for continuously graded mixtures. Although the process is similar to the SHRP short term aging protocol, the temperature is either 135°C or the compaction temperature, whichever is superior, and the aging time is restricted to 2 hours. In addition, Scholz (1995) improved a long term aging process for compacted asphalt mixture samples. It is the same as long term aging condition of the SHRP STOA protocol. In this process, compacted specimens are aged in an oven for 120 hours at 85°C (Brown & Scholz, 2000).

AASHTO R30 (2002), is the current standard recommended by the American Association of State Highway and Transportation Officials (AASHTO) to represent short and long term aging of asphalt mixtures. According to that standard, loose mixture specimens should be conditioned at 135°C for 4 hours in order to represent short term aging effects.As for long term aging simulaion, the specimens should be compacted with Marshall compactor first and then compacted specimens should be conditioned at 85°C for 120 hours to represent long term aging.

Bonaquist (2011), made experimental studies depending on National Cooperative Highway Research Program (NCHRP) Project 09-43. In order to simulate bitumen absorption and aging during construction, a conditioning time at T_c was determined (Compaction Temperature) as 2 hours on the other hand, to represent aging over the service life, a representatively higher temperature and longer aging time, which is not more than 16 hours, were determined to evaluate the moisture susceptibility and the rutting resistance.

Epps Martin et al. (2014), carried out NCHRP Project 9-49 involving laboratory conditioning experiments. In view of the difficulty of determining the T_c in the field, 2 hours at 135°C was recommended as the standard laboratory conditioning protocol instead of 2 hours at T_c. For long term aging, a significant effect was observed in the increase of the mixture stiffness due to long term aging both in the laboratory and field. Additionally, it was pronounced that, aging occurs more in summer than winter due to higher temperatures. When simulating the first summer of field aging, the loose samples should be exposed to short term aging condition at 135°C for 2 hours and after compacting the samples they should be aged for 4 weeks to 8 weeks at 60°C for long term aging.

The NCHRP 09-52 project (2018), which has been completed recently provided a comprehensive review of the existing AASHTO R30 protocol in addition to the recommendations of the NCHRP 09-43 and NCHRP 09-49 projects. In the NCHRP 09-52 Project, 9 field sites were selected in the United States to compare laboratory conditioned specimens with field specimens. In the light of the findings obtained from 9 field regions, the long term aging procedure recommended by AASHTO was found appropriate, whereas in the short-term aging procedure, 2 hours were recommended instead of 4 hours (Yin et al., 2018).

Partl et al. (2012), performed a research in accordance with RILEM (The International Union of Laboratories and Experts in Construction Materials 206-ATB) to reproduce recycling asphalt pavement (RAP) in laboratory oven aging of loose asphalt mixture samples. The mixtures were produced in a plant and nine different laboratories.

Laboratory samples were firstly aged for 4 hours at 135°C. For the long term aging process, both the plant produced and the short term laboratory produced loose asphalt mixtures were aged in a forced draft oven for 1, 2, 5, 7 and 9 days at 85°C. Partl et al. showed that, binding oxidation continued for up to nine days, but oxidation rate decreased with the increasing conditioning time .Therefore, 4 hours at 135°C as short term aging condition and 9 days at 85°C as long term aging condition were recommended to simulate aging process.

Kim et al. (2018), made research depending on the NCHRP project 09-54. In order to represent long term aging, a procedure was developed, calibrated and validated with field mixtures obtained from nine field projects which represent the wide variety of climates found in the United States and Canada. This aging process was applied on the loose mixture.The temperature was determined as 95°C. Aging durations were determined by location, years of aging and depth. The proposed procedure provides a series of laboratory aging time maps for aging of the field area of 4, 8 and 16 years at depths of 6, 20 mm and 50 mm below the surface to determine the duration. Generally, a period of 4 years and a depth of 20 mm is taken into account for calculations.

Within the scope of the thesis, developed temperature methods regarding laboratory aging are taken into consideration.

3.3.2 Oxidation Methods

Oxidation tests involve applying high temperature and pressure oxidation to laboratory aging samples (Airey, 2003).

In the method developed by Kumar & Goetz (1977), samples were conditioned at 60°C for 1, 2, 4, 6 and 10 days. In the research conducted by Kumar & Goetz (1977), film thickness and permeability were measured. Permeability in dense mixtures is a measure of aging resistance, the ratio of the thickness of the film to permeability in open graded mixtures is a measure of the resistance against aging. In the research carried out by Goef & Lufsey (1965), they emphasized that permeability is important in determining the aging effect rather than void content.

Kim et al. (1986) & Oregon, aged their mixtures by pressure oxidation in the laboratory. In the experiment, the samples were aged for 1, 2, 3 and 5 days under 100 psi pressure at 60ºC and 0.7 MPa. The aging effects were assessed by resilient modulus and fatigue strength tests. In addition, the modulus ratios of the specimens (modulus ratio; is the ratio of the resilient modulus value of the aged specimens to the resilient modulus value of the unaged specimens) were calculated based on the aging time. The data obtained show that the rate of modulus increases as aging increases.

In the procedure, developed by SHRP programme, the samples are compacted after the short term aging. It includes oxygen blowing for 5 days at 60°C or 85°C.

Khalid & Walsh (2002) improved a test for accelerated aging of porous asphalt. The technique has shown the aging effect improved by the SHRP long term aging process, but have observed that longer aging times are required since lower temperatures are used in the test (Khalid & Walsh, 2000).

3.3.3 Ultraviolet / Infrared Treatments

Ultraviolet / infrared treatment includes exposing laboratory aging samples to ultraviolet radiation (Airey, 2003).

Hveem et al. (1963) prepared asphalt mixtures using Ottawa sand. The asphalt mixtures were tested in a semi compacted form. Infrared radiation was applied to the samples to create heat at 60ºC and air flow at 41ºC. Studies have shown that this process, which lasts approximately 1000 hours, is equal to the 5-year aging of the road.

Hugo & Kennedy (1985), applied ultraviolet light for 54 hours and 14 days to the mixture in two separate periods. After the treatments, viscosity tests were conducted on the recovered bitumen.

Tia et al. (1988) applied a large number of aging methods for various periods at 60°C, including convection oven aging, forced draft oven aging and ultraviolet light aging.

They compared ultraviolet light and oven aging methods on compacted samples. Their studies have shown that the aging level is the same in both experimental methods. They also stated that the use of forced draft oven together with ultraviolet light methods represents better aging.

CHAPTER FOUR

EXPERIMENTAL

4.1 Materials

4.1.1 Bitumen

50/70 penetration grade bitumen obtained from Tupras Aliaga refinery was used in the experimental studies. The penetration, softening point, ductility, specific gravity, flash point tests are conducted to evaluate the different feautures of the bitumen. The results of the experimental studies are given in Table 4.1.

Table 4.1 Laboratory test results for 50/70 base bitumen

Test	Results	Spec. Limits	Test Method
Penetration Test (0.1 mm)	65	50 - 70	ASTM D5-06
Softening Point Test (°C)	51	46 - 54	ASTM D36-06
Viscosity (mPa.s) @ 135°C	0.412	3000 mPa.s (max.)	ASTM D4402-06
Viscosity (mPa.s) @ 165°C	0.104	-	
Ductility @ 25°C, cm	100		ASTM D113
Specific Gravity	1.030	-	ASTM D70
Flash Point	+260	230 (min.)	ASTM D92
Penetration Index (PI)	0.35	-	-
Rolling Thin Film Oven Test (RTFOT)			ASTM D2787-12
Change of mass (%)	0.160	0.5 (max)	-
Penetration after RTFOT	53	50 (min)	ASTM D5
Retained Penetration after RTFOT (%)	82	50 (min.)	ASTM D36-06
Softening Point after RTFOT (%)	58	48 (min.)	ASTM D36-06

4.1.2 Aggregates

In the experimental studies, limestone aggregates obtained from Derebeton İzmir Quarry were used and specific gravity, Los Angeles abrasion, sand equivalence, fine aggregate angularity, flat and elongated particles ,sodium sulfate soundness tests were applied on the aggregate groups to determine the properties of the aggregate that is used. The results of the tests applied on the aggregate groups, the used specifications and limit values are given in Table 4.2. Aggregate gradation was determined in accordance with the Type 1 wearing course of specification of general directorate Turkish Highway.

Table 4.2 Physical properties of limestone aggregate used in the tests

Test	**Results**	**Spec. Limits**	**Test Method**
Specific Gravity (Coarse Agg.)			ASTM C127-07
• **Bulk**	2.704	-	
• **Saturated surface dry (SSD)**	2.717	-	
• **Apparent**	2.739	-	
Specific Gravity (Fine Agg.)			ASTM C128-07
• **Bulk**	2.691	-	
• **SSD**	2.709	-	
• **Apparent**	2.739	-	
Specific Gravity (Filler)	2.732	-	
Los Angeles Abrasion (%)	2	45 (max.)	ASTM C131
Flat and Elongated particles (%)	7.5	10 (max.)	ASTM D4791-10
Sodium Sulfate Soundness (%)	1.47	10-20 (max.)	ASTM C88-05
Fine Aggregate Angularity (FAA)	47.85	40 (min.)	ASTM C1252-06

Table 4.3 Gradation for limestone aggregate

Sieve Size/No.	Gradation (%)	Spec. Limits
19 mm	100	100
12.5 mm	92	88-100
9.5 mm	73	72-90
No.4	44.2	42-52
No.10	31	25-35
No.40	12	10-20
No.80	8	7-14
No.200	5.3	3-8

4.2 Optimum Bitumen Determination Through Marshall Mix Design

In Marshall mix design method, at least three test samples are prepared for each combination of aggregate and bitumen percentage in accordance with ASTM D 1559. The aggregate mixture to be used within the scope of the test is dried in an oven at 105 ± 5 ° C. After this process, 1150 grams of aggregate mixture that is prepared according to the determined gradation is kept in the oven for a while in order to reach the mixing temperature. The bitumen and aggregates that reach the mixing temperature are put in the mixing bowl and mixed thoroughly. The bitumen-aggregate mixture obtained after the mixing process is placed in Marshall molds with a diameter of 101.6 mm and a height of 76.2 mm. The mixture, which cools down to the specified compaction temperature, is placed in the Marshall compactor and 75 blows are hit on both sides for the wearing course Type-1 Turkish Specification. After compaction, the sample that is cooled to room temperature is placed in the jack and removed from the mold.

Before the experiment, the samples are kept in a 60 ° C water bath for 2 hours in order to bring them to the desired measurement temperature. At the end of this period, the samples are removed from the water bath, dried and placed in the Marshall test device. The flow meter is placed in its place and the device has been started. The loading speed of the device is 50.8mm / min. should be. The experiment continues until the stability and flow values on the device screen become stable. At the end of the experiment, the stability and flow data are recorded. According to all experimental and measurement results, average heights of samples, bulk specific gravity (Dp), percentage of void (Vh), percentage of void in mineral aggregate (VMA), percentage of void filled with asphalt (VFA) are calculated for each bitumen percentage. The following graphs are drawn.

-% Bitumen - Dp
-% Bitumen - Stability
-% Bitumen - Flow
-% Bitumen - Air void
-% Bitumen - VMA
-% Bitumen – VFA

While determining the optimum bitumen percentage, it is checked whether other values are in accordance with the specification criteria based on 4% air void for the wearing course.

4.3 Laboratory Aging Procedures

As explained previously in section 3.3.1, short and long term oven aging was carried out with extended heating methods within the scope of the thesis. The aging methods used in the experiments are AASHTO R 30, NCHRP 09-52, NCHRP 09-54 and RILEM, as stated in Chapter 1.

The samples were divided into three groups to represent the aging of asphalt in the laboratory: unaged specimens, short term aged specimens and long term aged specimens. Specimens were kept in a room that is airtight without being exposed to aging to see the impact of aging clearly. These specimens will be named as unaged specimens. The unaged process involves neither short term nor long term aging processes. It should be taken into consideration that, the long term oven aging procedures also involve the short term forced draft oven aging treatment.

As indicated before, AASHTO R30 is the current standard that involves short term aging of loose mixture in the forced draft oven for 4 hours at 135°C and long term aging of compacted specimens for 120 hours at 85°C. Initially 3 specimens were kept in the forced draft oven for 4 hours at 135°C to represent short term aging (Figure 4.1). Following this process, the short term aged specimens were compacted with the Marshall compactor as shown in Figure 4.2 and kept in the oven, heated up to 85°C for 5 days to represent long term oven aging as shown in Figure 4.3. The summary of the experimental studies is given in the flowchart in Figure 4.4.

Figure 4.1 Short term conditioning of AASHTO R30 method in a forced-draft oven (Personal archive, 2019)

Figure 4.2 Compacted specimens of AASHTO R30 method after short term conditioning (Personal archive, 2019)

Figure 4.3 Long term conditioning of AASHTO R30 method in a forced-draft oven (Personal archive, 2019)

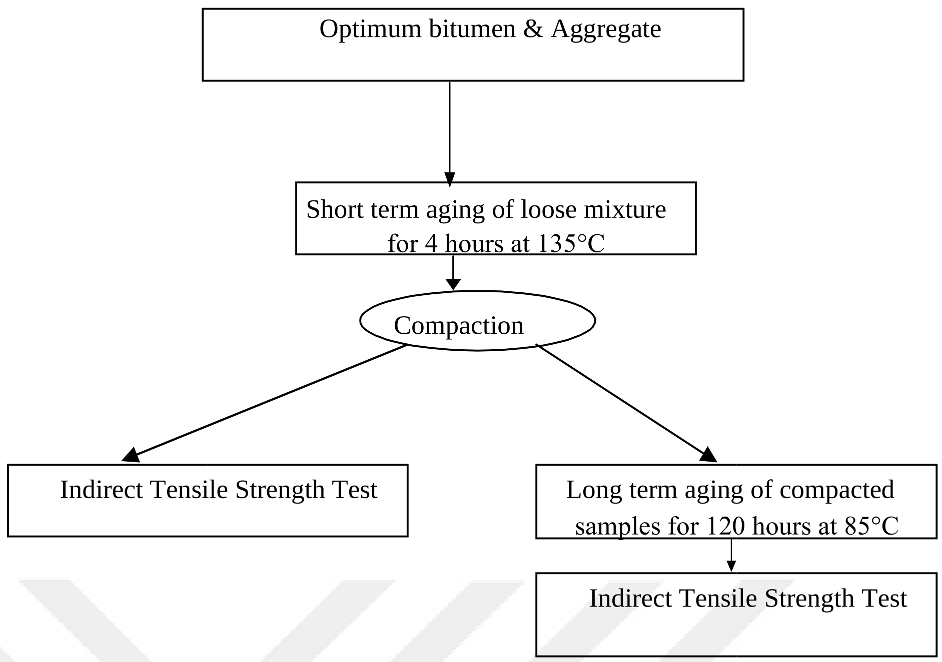

Figure 4.4 Flow chart of the current laboratory aging standard (AASHTO R 30)

NCHRP 9-52 method involves short term forced draft oven conditioning of loose mixture for 2 hours at 135°C and long term aging of compacted specimens for 120 hours at 85°C. 3 specimens were kept in a forced draft oven at 135°C for 2 hours. After that the short term aged specimens were compacted with the Marshall compactor and kept in the oven heated up to 85°C for 5 days to represent long term oven aging.

RILEM method involves short term conditioning of loose mixture for 4 hours at 135°C and long term aging of loose mixture for 9 days at 85°C. 3 specimens were kept in a loose mixture state at 135°C for 4 hours for short term aging as shown in Figure 4.5. Then, for long term aging, they were kept in the oven at 85°C for 9 days in a loose mixture state (without compaction after short-term aging) as demonstrated in Figure 4.6.

Figure 4.5 Short term conditioning of RILEM method (Personal archive, 2019)

Figure 4.6 Long term conditioning of RILEM method (Personal archive, 2019)

Regarding NCHRP 09-54 method, the same short term conditioning was applied as in AASHTO R30 (4 hours at 135°C). For this reason, specimens of AASHTO R30 method and NCHRP 09-54 method were prepared and put in the oven at the same time. Firstly, 3 specimens were kept in a loose mixture for 4 hours at 135°C for short term aging. Following the short term aging procedure, the specimens were long term aged as a loose mixture without compaction.

Long term aging protocol involves the aging of the loose mixture and 95°C is determined as an aging temperature (Kim et al., 2018). The aging was performed in the forced draft oven. The aging duration was determined by the following formula:

$$t_{oven} = \sum_{i=1}^{N} D * A * \exp\left(-Ea/(R * Ti)\right)/24 \quad (4.1)$$

where:

t_{oven} = required oven aging duration at 95°C to reflect field aging (days);

D= depth correction factor;

A= frequency factor;

E_a = activation energy

R= universal gas constant (this value is taken as 0.0831kJ/mol.k)

Ti= pavement temperature at the depth of interest ,at the hour of interest (Kelvin**)**

In this study the depth correction factor (D), the frequency factor (A) and activation energy (E_a) are obtained corresponding to pavement layer of 20 mm depth from Table 4.4.

Table 4.4 Climatic aging index coefficients (Kim et al., 2018)

Pavement Layer	**Depth Correction Factor (D)**	**Pre-exponential Factor (A)**	**Activation Energy (E_a)**
Surface Layer (6mm)	1.0000	1.40962	13.3121
20- mm Depth	0.4565	1.40962	13.3121
Deeper Layers (below 20 mm)	0.2967	1.40962	13.3121

The pavement temperature (Ti) was determined by following the equation developed by Witczak:

$$Ti = Ta * [1 + 1/(z + 4)] - 34/(z + 4) + 6 \quad (4.2)$$

where:

T_i = hourly pavement temperature (°F),

Ta= hourly air temperature (°F),

z= depth below the pavement surface (this value is taken as 0,79 inch corresponding to 20 mm depth)

For the calculation of T_i, the 4 main districts (Dikili, Aliaga, Menemen, Buca) of Izmir City were taken into account and the hourly air temperature values (Ta) were taken from the General Directorate of Meteorology belonging to the years of 2015, 2016, 2017 and 2018. The 4 year period is based on the recommendation of the NCHRP 09-54 method.

T_{oven} values were then calculated yearly for each district as presented in Table 4.5 which indicates that, average duration of 4 days should be utilized ,regarding the simulation of long term aging process.

Table 4.5 The T_{oven} values for 4 main districts

District/Years	2015 (days)	2016 (days)	2017 (days)	2018 (days)	Total (days)
Dikili	1.035	1.043	1.037	1.057	4.17
Aliaga	1.039	1.052	1.039	1.062	4.19
Menemen	1.039	1.052	1.039	1.063	4.19
Buca	1.038	1.052	1.040	1.061	4.19

Table 4.6 summarizes the name of each above-mentioned methods as well as the conditions (duration and temperature level) of short and long term aging methods.

Table 4.6 The methods used for aging

Method	Aging					
	Short Term Aging			**Long Term Aging**		
	Temperature (°C)	Duration (hrs)	Mixture Type	Temperature (°C)	Duration (days)	Mixture Type
AASHTO R30	135	4	Loose	85	5	Compacted
NCHRP 09-54		4	Loose	95	4*	Loose
NCHRP 09-52		2	Loose	85	5	Compacted
RILEM		4	Loose	85	9	Loose

***value determined by calculations**

4.4 Indirect Tensile Strength (ITS)

The indirect tensile strength (ITS) test was conducted on the laboratory prepared samples to evaluate the short and long term aging condition. It is the most common test used for evaluating the aging characteristics of asphalt mixtures, which directly affect the performance of the asphalt pavements (Anderson et al., 2003). Indirect tensile strength test is often preferred in civil engineering because of its simplicity. The ITS results give information about the laboratory mix design test and allow to predict resistance to failures, such as cracking and rutting (Zanievski & Srinivasan, 2004).

The same test was also applied on the field core samples taken from the recently constructed wearing course as well as from the pavement surface with a service life of 5 years. In order to make definite evaluations, the type of the aggregate, the gradation as well as the type of the bitumen used in the field was chosen the same as in the laboratory aged specimens.

The cylindrical laboratory prepared asphalt specimens and the field core specimens were correctly placed on the loading frame. The load was implemented (at loading rate of 51mm/min) in the vertical diametrical plane and created a tensile load in the horizontal direction of the specimen. The maximum load was read and saved on the device which is illustrated in Figure 4.7 (ASTM D 6931).

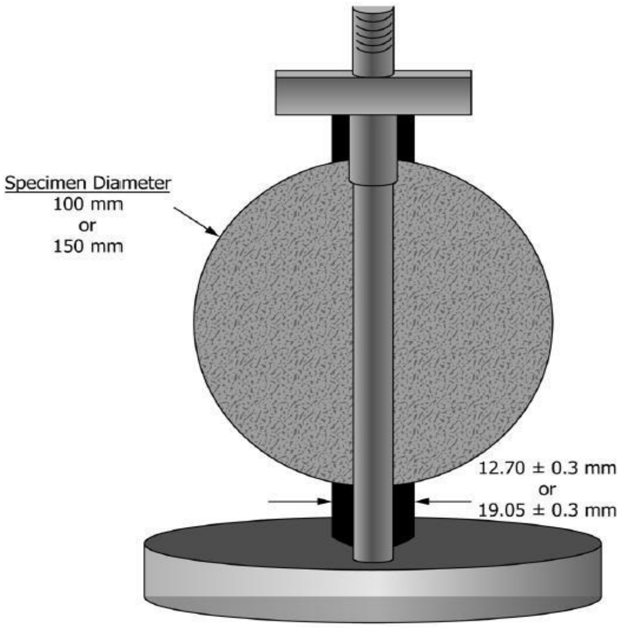

Figure 4.7 Diagram of an ITS loading fixture (ASTM D 6931, 2012)

The standard test method for indirect tensile (IDT) strength of bituminous mixtures (ASTM D6931) was used to conduct this process.The below steps are followed in order:

- The height of the specimen was measured and then recorded as illustrated in Figure 4.8

Figure 4.8 Asphalt height measurement with caliper (Personal archive, 2019)

- The diameter of the specimen was drawn by chalk to provide the correct position as shown in Figure 4.9.

Figure 4.9 Drawing specimens with chalk (Personal archive, 2019)

- Then the specimen was put in a plastic bag and the air was soaked by a pipette as shown in Figure 4.10 and placed into water for 2 hours at 25°C as shown in Figure 4.11.

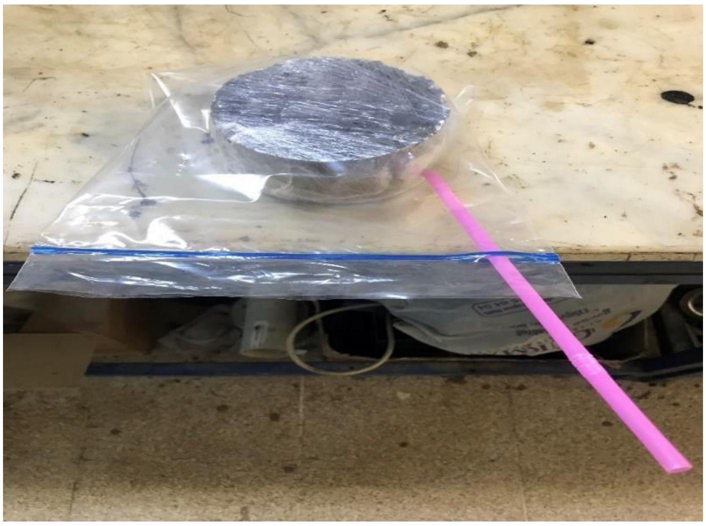

Figure 4.10 Vacuuming the specimen and putting it in a heavy duty leak-proof plastic (Personal archive, 2019)

Figure 4.11 Water bath at 25 °C (Personal archive, 2019)

- After the specimen was taken out of the water and the plastic bag, it was placed properly to the device.

- After placing the specimen into the device, a vertical compression load is applied and when the maximum value is read it is recorded. Figure 4.12 shows the correct placement of the specimen into the device.

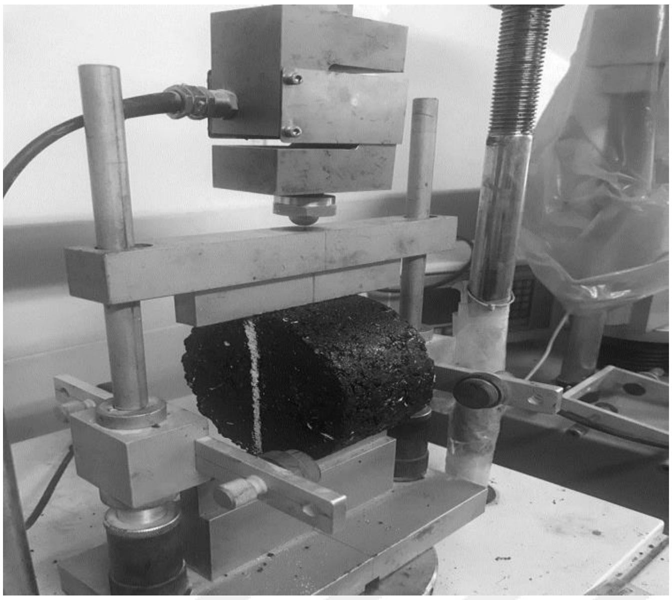

Figure 4.12 A true assemble of specimen for ITS test (Personal archive, 2019)

The below equation is used to calculate the ITS:

$$St = (2000\ x\ P_{max})\ /\pi t D \tag{4.3}$$

where;

S_t = Indirect tensile strength (ITS), kPa

P = Maximum load, N

t = Specimen height immediately before test, mm

D = Specimen diameter, mm

Aging indices were defined for the assesment of the aging effects of the laboratory prepared asphalt mixtures. The ratio between the ITS values of short and long term aged specimens and the ITS values of unaged specimens were calculated as short term aging index (SAI) and long term aging index (LAI) (Equation 4.4, 4.5).

$$\text{SAI} = \frac{(\text{ITS value of short term aged specimens})}{(\text{ITS value of unaged specimens})} \tag{4.4}$$

$$\text{LAI} = \frac{(\text{ITS value of long term aged specimens})}{(\text{ITS value of unaged specimens})} \quad (4.5)$$

The results of the indirect tensile test of specimens taken from the field and prepared at the laboratory were compared to specify the level of aging. The most accurate laboratory aging method which represents aging in the field well is aimed to be determined.

CHAPTER FIVE
RESULTS AND DISCUSSION

5.1 Viscosity Test Results

In accordance with ASTM standard, the ranges of viscosity values that are used to determine the mixing and compaction temperatures are 0.17 ± 0.02 Pa.s and 0.28 ± 0.03 Pa.s. The viscosity values of the asphalt used in the mixture at 135°C and 165°C were measured in order to specify the mixing temperatures and compaction temperatures. The values obtained are plotted on the graph as shown in Figure 5.1. On the line formed by combining these two points, the mixing and compaction temperature ranges corresponding to the above viscosity values were determined. These ranges are given in Table 5.1.

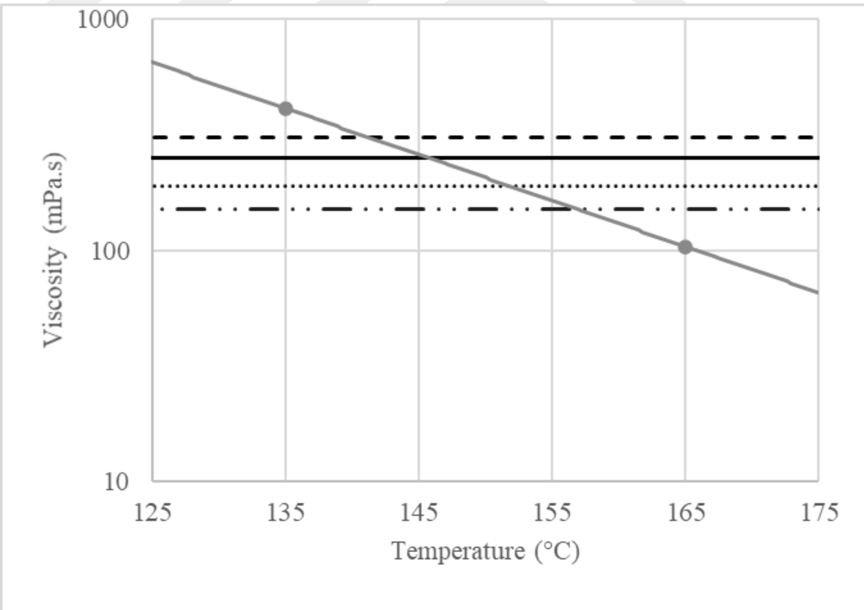

Figure 5.1 Temperature-viscosity relationship of the bitumen

Table 5.1 Mixing temperatures and compaction temperatures for base bitumen

	Mixing Temperatures (°C)	**Compaction Temperatures (°C)**
Base Bitumen	**152-158**	**141-146**

5.2 Marshall Mix Design Results

The Marshall Stability test was performed on the specimens, which include different bitumen content to specify the optimum bitumen content in accordance with ASTM D1559. In the experimental study, the optimum bitumen content which corresponds to 4% air void (depending on the maximum and minimum air void level presented in the General Directorate of Highways Specifications) was selected as 4.80% (by weight of aggregate). The detailed outcomes of the Marshall Design as well as the bitumen content versus Unit weight (Dp), Stability, flow, air void (Vh),void filled with asphalt (VFA), void in mineral aggregate (VMA) are presented in Table 5.2.

The parameters (stability, flow, VFA, VMA) corresponding to 4.80% optimum bitumen content are presented in Table 5.2. As indicated in Table 5.2, all parameters are within the specification limits.

Table 5.2 Marshall mix design test results

Parameter	Result	Specification for Wearing Course
Optimum Bitumen Content (%)	4.8	4-7
Stability (kg)	1135	900 (min)
Flow (mm)	2.60	2-4
VFA (%)	71	65-75
VMA (%)	14	14-16

5.3 Indirect Tensile Strength Test Results

5.3.1 Results of Laboratory Aged Specimens

The ITS results of unaged, short term and long term oven aged specimens regarding all methods are presented in Figure 5.2. The details of the calculations can be found in Appendix section. For all the investigated aging methods, it was observed that, the ITS values of the specimens increased from unaged condition to long term aged condition. The rise in indirect tensile stress values is considered as an indicator of the aging effect.

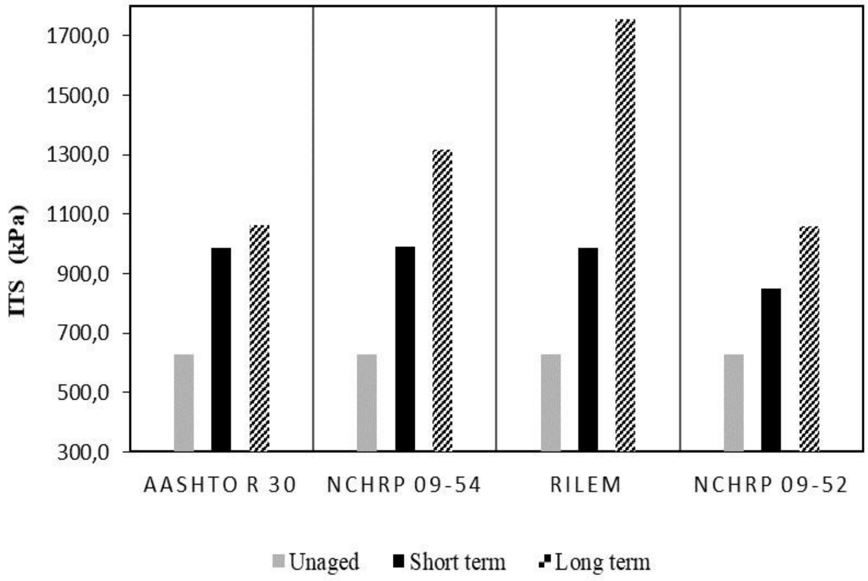

Figure 5.2 ITS results of aging methods

Among all the short term aged specimens subjected to investigated methods , the short term aged sample by the method of NCHRP 09-52 yielded the minimum (although the variation is not significant) ITS values since the NCHRP 9-52 method involves 2 hours of oven aging of loose samples. The short term aged samples produced by other methods demonstrated similar ITS values.

In case of long term aged specimens, the long term aged specimen by RILEM Method yielded the highest ITS value indicating pronounced effect of aging. As expected, long term aged specimens by traditional method (AASHTO R30) and NCHRP 09-52 yielded similar ITS values.

Short term aging index (SAI) and long term aging index (LAI) were calculated for the evaluation of aging characteristics of the specimens. Figure 5.3 represents the indices corresponding to each method.

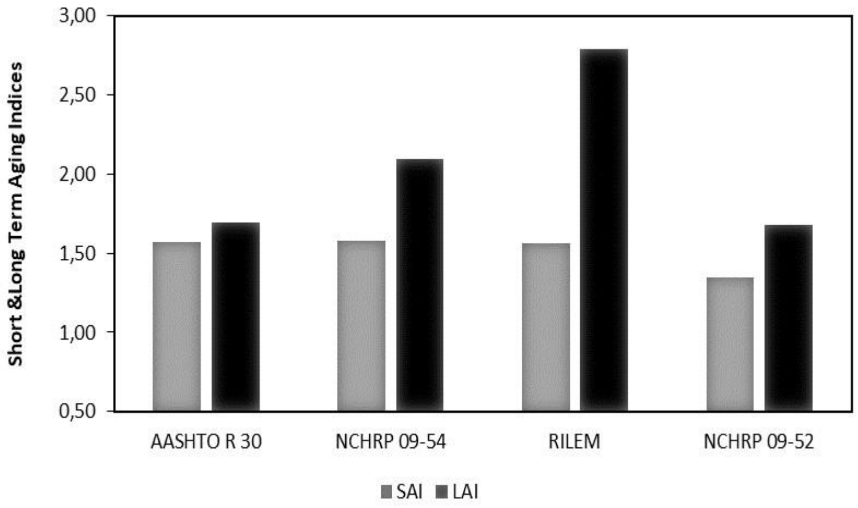

Figure 5.3 Short & long term aging index results of all aging methods

Depending on Figure 5.3, the following conclusion has been reached: the lower the aging index values, the less aging effect that the bituminous mixture exposes. Among the short term aged specimens, the short term aged specimen by NCHRP 09-52 method yielded less aging indices. The short term indices regarding AASHTO R30, NCHRP 09-54 and RILEM method yielded similar aging index values. Long term aging index values indicate that, RILEM method produced higher long term index values. Besides, NCHRP 09-52 and traditional method yielded similar long term index values.

5.3.2 Results of Field Core Specimens

The field core specimens were taken to evaluate the short and long term aging conditions of the laboratory prepared specimens from the recently constructed wearing course and from the surface of a wearing course with a service life of 5 years. It should be mentioned that, the type of the aggregate, the gradation as well as the type of the bitumen used in the field is the same as in the laboratory aged specimens. The ITS values of both field core samples and the laboratory aged specimens are shown in Figure 5.4.

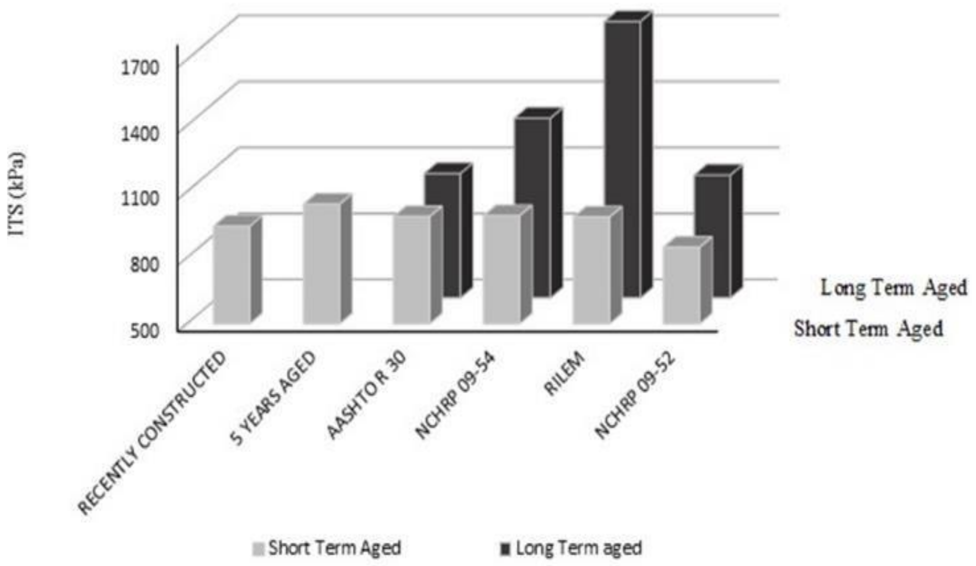

Figure 5.4 ITS Results of field core specimens

As indicated in Figure 5.4, the ITS value regarding the sample, taken from the 5 years old pavement surface is higher than the asphalt core ,taken from the recently constructed pavement surface. As expressed before, the increment in the ITS value is an indicator of the aging effect. When comparing the specimen ,taken from the recently constructed pavement with the laboratory short term aged specimens (simulating the aging process during mixing with aggregate, laying and compaction), the ITS value of short term aged sample by AASHTO R30, NCHRP 09-54 and RILEM Method has demonstrated similar values with the ITS value of recently constructed sample.

When comparing the ITS values of the long term laboratory oven aged samples with 5 years old field specimen, the ITS value of long term aged specimen by AASHTO R30, NCHRP 09-52 yielded similar values with the ITS value of sample taken from 5 years aged pavement surface.

CHAPTER SIX
FINDINGS AND CONCLUSIONS

This research is aimed to evaluate the aging procedures of asphalt mixture with the current standard (AASHTO R30) as well as the alternative methods (NCHRP 09-52, NCHRP 0954 and RILEM) by way of indirect tensile strength test. The level of aging has been also compared with the samples taken from recently constructed pavement surface and from the 5 year old pavement surface.

The below conclusions can be drawn from this research:

1. For all the investigated aging methods, the indirect tensile stress (ITS) values of the samples increased from unaged condition to long term forced draft oven aged conditions. The increase in ITS values is considered as an indicator of the aging effect. The increment in ITS is not favorable since the asphalt concrete with higher ITS value is more susceptible to fatigue cracking although it is more rigid, brittle and resistant to bending. Besides, fatigue life of samples with higher ITS values reduces at a certain level of stress.

2. Comparing all the short term forced draft oven aged specimens, all of the investigated methods yielded similar ITS values owing to the fact that, all the short term oven aging conditions regarding each method do not exhibit significant difference. Although this variation is not significant, the sample short term aged by the NCHRP 09-52 method had the minimum ITS values since NCHRP 09-52 involves less oven aging treatment (2 hours at 135°C) in comparison with other methods.

3. In case of long term aged specimens, RILEM method yielded the highest ITS value indicating pronounced effect of aging. The long term aged samples produced by traditional method (AASHTO R30) and NCHRP 09-52 yielded similar ITS values (although their short term oven aging procedures regarding the two methods differ) due to the fact that, both methods cover exactly the same long term oven aging

treatment (85°C; 120 hours). It may be concluded that, 2 hours or 4 hours short term forced draft oven aging of loose samples at 135°C does not yield significant variation in the ITS values of long term aged samples.

4. Considering the findings from the short and long term aging indices, which were calculated for the evaluation of aging characteristics of the samples, it is possible to consider that, lower aging index values exhibit less effect of aging. Among the short term aged samples, the one aged by NCHRP 09-52 method yielded less aging indices. The short term indices regarding AASHTO R30, NCHRP 09-54 and RILEM method yield similar aging index values. Long term aging index values indicate that, RILEM method produced higher long term index values. Besides, NCHRP 09-52 and traditional method yielded similar long term index values.

5. The short and long term forced draft oven aging condition of the laboratory prepared specimens were compared with field core specimens in order to evaluate the simulation of oven aging procedures. The comparison of ITS values of the specimens taken from the recently constructed pavement surface and the ITS values of laboratory short term aged specimens (simulating the aging process during mixing with aggregate, laying and compaction), indicate that, the AASHTO R30, NCHRP 09-54 and RILEM methods demonstrate similar values. Therefore; it may be concluded that, the 2-4 hours forced draft oven aging at 135°C can be suggested as short term aging condition. Taking into consideration the ITS values of the long term laboratory oven aged specimens with 5 years old field specimen, the ITS value of long term aged specimen by AASHTO R30, NCHRP 09-52 yielded similar values.

6. Although the parameters regarding NCHRP 09-54 method has been gathered and calculated for Izmir Region, a significant variation is observed between the ITS values of long term laboratory aged specimens and ITS of the 5 years old field core samples. Therefore, 120 hours (5 days) oven aging of compacted specimens at 85°C can be recommended as a short term aging condition.

This method includes the usage of one type of aggregate and 50/70 penetration grade bituminous binder. Further studies can be made by the use of various aggregate type as well as bitumen.

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APPENDICES

APPENDIX A: MARSHAL MIX DESIGN

Table A.1 Marshall mix design data for virgin bitumen

Specimen No.	Bitumen %		Specimen Height (mm)				Weight in air (gr.)	Weight in water (gr.)	SSD weight (gr.)	Volume	Bulk specific gravity	Max. teo. specific gravity	Voids (%)	VMA	VFA	Flow (mm)	Stability (kgf)	Correlation fact.	Corr. Stability (kgf)
	Wa	**Wb**	**1**	**2**	**3**	**Avg.**	**A**	**C**	**B**	**V**	**Dp**	**Dt**	**Vh**	**%**	**%**	**mm**	**kgf**		**kgf**
1	3.5	3.4	64.1	64.2	64.1	64.1	1172.7	684.2	1189.4	505.2	2.321	2.569				2.1	1045	0.985	1029
2	3.5	3.4	63.9	63.8	63.9	63.9	1171.4	685.1	1183.7	498.6	2.349	2.569				1.80	1034	0.99	1024
3	3.5	3.4	63.4	63.2	63.2	63.3	1180.9	688.0	1192.6	504.6	2.340	2.569				2.52	1101	1.005	1107
Avg.											2.337	2.569	9.04	16.01	43.5	2.14			**1053**
1	4.0	3.8	62.9	62.9	62.8	62.9	1175.2	686.2	1179.3	493.1	2.383	2.551				2.21	1192	1.015	1210
2	4.0	3.8	62.8	62.9	62.9	62.9	1183.1	691.2	1185.8	494.6	2.392	2.551				2.39	1079	1.015	1095
3	4.0	3.8	62.9	62.4	62.2	62.5	1185.4	693.4	1188.4	495.0	2.395	2.551				2.30	1181	1.025	1211
Avg.											2.390	2.551	6.32	14.52	56.5	2.30			**1172**
1	4.5	4.3	62.0	61.6	61.9	61.8	1190.3	695.3	1191.4	496.1	2.399	2.534				2.85	1192	1.043	1243
2	4.5	4.3	61.9	61.8	61.9	61.9	1170.9	687.0	1172.0	485.0	2.414	2.534				2.73	1309	1.04	1361
3	4.5	4.3	61.9	61.8	61.7	61.8	1180.3	695.0	1181.2	486.2	2.428	2.534				2.05	1201	1.043	1253
Avg.											2.414	2.534	4.73	14.08	66.4	2.54			**1286**
1	5.0	4.8	61.9	61.9	61.7	61.8	1187.1	701.1	1187.6	486.5	2.440	2.516				2.65	980	1.043	1022
2	5.0	4.8	61.6	61.7	61.6	61.6	1197.4	705.2	1197.5	492.3	2.432	2.516				2.59	936	1.049	982
3	5.0	4.8	61.7	61.5	61.7	61.6	1177.8	688.2	1178.1	489.9	2.404	2.516				2.72	853	1.049	895
Avg.											2.426	2.516	3.61	14.08	74.3	2.65			**966**

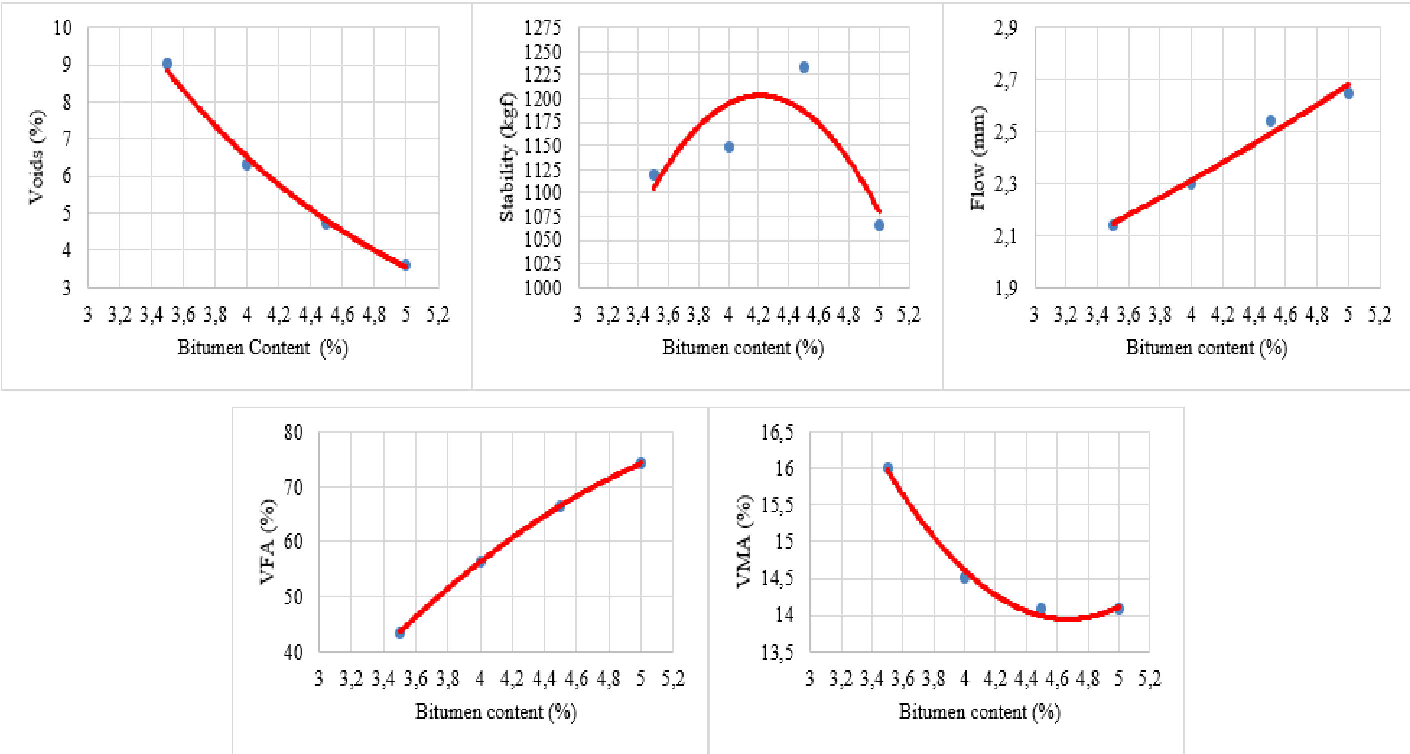

Figure A.1 Marshall mix design graphs for virgin bitumen

APPENDIX B: INDIRECT TENSILE STRENGTH

Table B.1 Indirect tensile strength test results of laboratory aged specimens

	METHOD	Specimen No	Specimen Thickness (mm)				Diameter	Load	Load	ITS	Average
			1	2	3	Ave.	(mm)	(kgf)	(N)	(kPa)	
Unaged		1	62.7	62.4	62.1	62.4	101.6	616	6040.90	606.60	628.60
		2	61.2	61.4	61.5	61.4	101.6	626	6138.96	626.96	
		3	62.4	62.2	62.5	62.4	101.6	662	6492.00	652.25	
		Average									
Short-Term Aged	AASHTO R30 (4hrs, 135 °C)	1	60.4	60.2	60.4	60.3	101.6	1012	9924.33	1030.70	987.01
		2	62.4	62.1	62.4	62.3	101.6	955	9365.35	942.14	
		3	62.3	62.2	62.5	62.3	101.6	1002	9826.26	988.19	
		Average									
	NCHRP 09-54 (4hrs, 135 °C)	1	60.4	60.1	60.2	60.2	101.6	864	8472.95	881.42	991.15
		2	60.2	60.5	60.2	60.3	101.6	1101	10797.12	1121.96	
		3	62.1	62.2	62.1	62.1	101.6	981	9620.32	970.07	
		Average									
	RILEM (4hrs, 135° C)	1	61.6	61.4	61.5	61.5	101.6	963	9443.80	962.18	985.10
		2	61.3	61.4	61.4	61.4	101.6	997	9777.23	998.43	
		3	61.5	61.2	61.7	61.5	101.6	995	9757.62	994.70	
		Average									
	NCHRP 09-52 (2hrs, 135 °C)	1	62.1	62.2	62.2	62.2	101.6	856	8394.49	846.10	847.95
		2	62.2	62.2	62.1	62.2	101.6	864	8472.95	854.19	
		3	62.5	62.3	62.7	62.5	101.6	858	8414.11	843.56	
		Average									
Long-Term Aged	AASHTO R30 (5days, 85 °C)	1	62.1	61.8	62.1	62.0	101.6	1013	9934.14	1004.20	1044.20
		2	61.6	61.3	61.4	61.4	101.6	1021	10012.59	1021.24	
		3	61.8	61.9	61.8	61.8	101.6	1114	10924.61	1107.18	
		Average									
	*NCHRP 09-54 (3days, 95 °C)	1	62.1	61.8	61.9	61.9	101.6	1357	13307.62	1346.36	1315.83
		2	63.0	63.2	63.1	63.1	101.6	1323	12974.20	1288.36	
		3	61.8	62.1	61.9	61.9	101.6	1323	12974.20	1312.77	
		Average									
	*RILEM (9 days, 85 °C)	1	63.2	63.3	63.2	63.2	101.6	1777	17426.42	1726.83	1755.10
		2	62.4	62.2	62.3	62.3	101.6	1769	17347.96	1744.80	
		3	62.6	62.4	62.5	62.5	101.6	1824	17887.33	1793.68	
		Average									
	NCHRP 09-52 (5 days, 85 °C)	1	61.3	61.4	61.4	61.4	101.6	1159	11365.91	1160.53	1091.84
		2	62.8	62.8	62.9	62.8	101.6	1082	10610.80	1058.37	
		3	62.7	62.5	62.7	62.6	101.6	1077	10561.76	1056.62	
		Average									

Table B.2 Indirect tensile strength test results of field core specimens

	Specimen No	Specimen Thickness (mm)				Diameter	Load	Load	ITS
		1	2	3	Average	(mm)	(kgf)	(N)	(kPa)
RECENTLY CONSTRUCTED	1	61.4	61.7	61.5	61.5	101.6	925	9071.15	923.72
	2	62.2	62.4	62.5	62.4	101.6	998	9787.04	983.30
	3	61.5	61.6	61.4	61.5	101.6	950	9316.32	949.20
	4	61.8	61.6	61.7	61.7	101.6	922	9041.73	918.23
5 YEAR AGED	1	63.2	63.3	63.3	63.3	101.6	1075	10542.15	1044.10
	2	62.5	62.4	62.4	62.4	101.6	1076	10551.96	1059.02
	3	61.9	61.7	61.9	61.8	101.6	1033	10130.27	1026.56
	4	61.5	61.6	61.4	61.5	101.6	1048	10277.37	1047.11