

Investigation of Different Laboratory Aging Methods of Bituminous Mixtures

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Abstract

The predicted performance and service life of the pavement depend largely on the properties of bitumen used in the mixtures. The most important feature of bitumen, which has profound effect on the performance of the road is durability. The durability of bitumen is expressed as the resistance to aging. 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. The aim of this paper is to evaluate different proposed laboratory aging methods (NCHRP 09-52, NCHRP 09-54 and RILEM) in relation with the current Standard AASHTO R30 (Standard Practice For Mixture Conditioning of Hot Mix Asphalt standard) and to make comparison with the samples performance taken from the field in terms of Indirect Tensile Strength (ITS). 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. Based on the results, 2 hours forced draft oven aging at 135°C is recommended as short term aging condition because, 2 hours or 4 hours short term forced draft oven aging did not yield significant variation in terms of ITS values. Additionally, 120 hours (5 days) oven aging of compacted samples at 85°C can be recommended as long term aging condition.

Keywords

aging, oxidation, short term aging, long term aging, hot mix asphalt

1 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 the United States [1, 2]. Keeping the aging occurrence under control is crucial for asphalt samples because it affects the rheological, morphological, and microstructural properties of the sample [3–5]. Traxler [6] listed 5 factors affecting the aging of asphalt in order of importance:

1. Oxidation
2. Volatilization
3. Time
4. Polymerization
5. Condensation

Traxler [7] increased the above factors to 15 in 1963. Four of these 15 items were believed to contribute most to hardening. In 1984, Petersen [8] reduced them to three major factors regarding the aging of asphalt mixtures:

1. Loss of the oily components of bitumen by volatility or absorption by porous volatilization

2. Changes in the chemical composition of bitumen molecules from reaction with atmospheric oxygen-oxidation
3. Molecular structuring that produces thixotropic effects-steric hardening

Later, most of the researchers carried out studies based on these three factors.

There are two components of aging to consider [9]:

1. Short term aging (STA)
2. Long term aging (LTA)

Short-term aging occurs during storage of the mixture, mixing in plant, transporting to construction site, laying and compaction. This is probably thought to be caused by volatilization. Long-term aging, which is thought to be predominantly caused by oxidation, occurs during its service life and while exposed to the environment [9].

Fig. 1 shows the aging index values depending on the years of bitumen. It is seen that, approximately 70 % of oxidation occurs during short term aging period. Oxidation

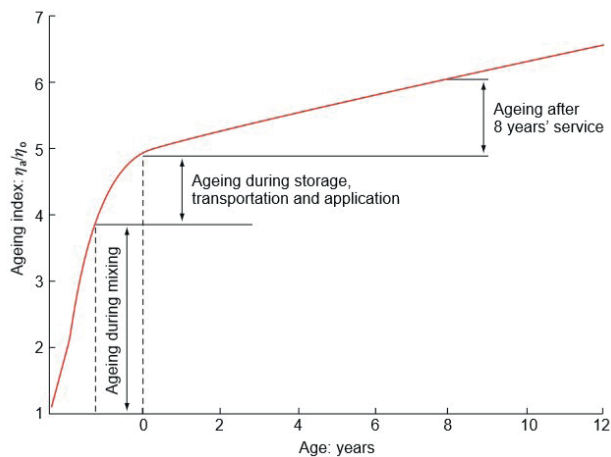


Fig. 1 Aging of asphalt during mixing, subsequently during storage, transportation, and in service [10]

decelerates during the long term aging period and is only around 30 % in 8 years. As can be seen from the figure, rapid aging occurs during the preparation, laying and compaction of the mixture, which slows down at low temperatures during service life of the road.

2 Previous studies on asphalt-aggregate aging

There have been several research studies conducted to develop laboratory aging procedures of asphalt mixtures. These studies have identified many interrelated factors such as; source of bitumen, bitumen type, absorption, production temperature and silo storage time etc. [11, 12]

The results of their studies have depicted that, laboratory aging procedures brought in similar properties to the bitumen with the field aging [6, 13–19].

Rolt [20] have investigated the effect of field aging on asphalt mixture properties. Based on the results of the study, it was concluded that, the influences of exposure time and temperature had a substantial effect on the characteristics of mixture aging, while the influence of pavement thickness, pavement density and bitumen content were negligible. In a study by Rondón et al. [21], hot mix asphalt was exposed to environmental impacts in Bogota, Colombia for 42 months. The test results showed that, the increase in mixture stiffness, rutting resistance and fatigue resistance was observed in the first 29 months of environmental exposure and the opposite situation was observed between 30 and 42 months. In a study performed by Houston et al. [22], it was observed that, pavement service temperature and pavement air void content significantly affected the aging mixture in the field and aging increased with warmer service temperatures and higher air void contents in the mixture.

In a study by Aschenbrener and Far [23], reheating was found as a significant effect on the aging of asphalt mixtures. They recommended laboratory-produced samples should be conditioned for 2 hours at field compaction temperature to simulate asphalt aging and absorption during plant production.

Bell et al. [9] evaluated the relation between field aging and laboratory aging in terms of short term oven aging (STOA) and long term oven aging (LTOA) protocols. The results from the resilient stiffness modulus indicated that, aging of loose mixtures for 4 hours at 135°C represents a short-term aging whereas aging of compacted samples for 120 hours at 85°C represents long term aging.

AASHTO R 30 is the current standard recommended by the American Association of State Highway and Transportation Officials (AASHTO) [24] in order to simulate short and long term aging of asphalt mixtures at laboratory. According to that standard, loose mixture specimens should be conditioned for 4 h at 135°C in order to simulate short term aging effects, while, for long term aging simulation, the samples should be compacted with Marshall compactor first and then compacted specimens should be conditioned for 120 hours at 85°C to represent long term aging.

Sirin et al. [25] investigated the viscoelastic behavior of the asphalt mixtures by altering the temperature and the duration of the aging procedures. Based on their findings, aging conditions have a considerable effect on the dynamic modulus of the samples. Also, they depicted that, conditioning at 85°C for 5 days, which is recommended in AASHTO R30 method, is not sufficient to simulate field aging in hot climatic region for compacted asphalt mixture specimen.

Bonaquist [26] 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 for the evaluation of moisture susceptibility and rutting resistance.

Epps Martin et al. [27] 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

field and laboratory. Additionally, it was pronounced that, aging occurs more in summer than winter due to high temperatures. In order to simulate the first summer of field aging, the loose samples should be subjected to short-term aging condition for 2 hours at 135°C and after compacting the samples they should be conditioned at 60°C for 4 weeks to 8 weeks for long term aging.

The more recently completed NCHRP 09-52 project provided a comprehensive review of the existing AASHTO R30 guidelines in addition to the recommendations of the NCHRP 09-43 and NCHRP 09-49 projects. In the NCHRP 09-52 Project, nine field sites were selected in the US to compare laboratory conditional samples with plant mixed samples. Findings from nine site regions depicted that, long term aging procedure, which proposed by AASHTO is suitable, however, STOA aging protocol of 135°C for 2 hours of the loose mixture is recommended instead of 4 hours [28].

Partl et al. [11] conducted a study in accordance with RILEM (The International Union of Laboratories and Experts in Construction Materials 206-ATB) with the aim of reproducing recycling asphalt pavement (RAP) by laboratory aging of loose mixture samples. Asphalt mixtures were produced in a plant and nine different laboratories. Laboratory mixes were firstly aged at 135°C for 4 hours. For the long-term aging process, both the plant-produced and the short-term laboratory-produced loose mixtures were conditioned in the oven at 85°C for 1, 2, 5, 7 and 9 days. Results showed that, binding oxidation continued for up to nine days, but oxidation rate decreased with 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 was recommended to simulate aging process.

Kim et al. [29] made research depending on the NCHRP project 09-54. In order to simulate long term aging of asphalt mixtures, 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. A loose mixture aging at 95°C was used as an aging protocol in an oven. 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.

The aim of this study is to compare the different proposed laboratory aging methods with the samples taken from the field for short and long term aging and to determine whether the current standard for aging is still valid. To represent the most accurate short and long term aging, the recently constructed and 5 years aged samples were taken from the field. ITS test was conducted on the samples both taken from the field and prepared at laboratory to evaluate the aging behavior.

3 Experimental

3.1 Materials

The base bitumen with a 50/70 penetration grade was obtained from Aliaga/Izmir Oil Terminal of the Turkish Petroleum Refinery Corporation. In order to characterize the properties of the base bitumen, conventional test methods such as: penetration test, softening point test, ductility test, etc., were performed. These tests were conducted in conformity with the relevant test methods that are presented in Table 1.

Table 1 50/70 Bitumen binder properties

Test	Result	Spec. Limits	Standard
Penetration Test (0.1 mm)	65	50-70	ASTM D5-06/ EN 1426
Softening Point Test (°C)	51	46-54	ASTM D36-06/ EN 1427
Viscosity (mPa.s) @ 135°C	0.412	3000 mPa.s (max.)	ASTM D4402-06
Viscosity (mPa.s) @ 165°C	0.104	-	ASTM D4402-06
Ductility @ 25°C, cm	100	-	ASTM D113
Specific Gravity	1.030	-	ASTM D70
Flash Point	+260	230 (min.)	ASTM D92 EN 22592
Penetration Index (PI)	0.35	-	-
Rolling Thin Film Oven Test (RTFOT)			ASTM D 2787-12
Change of mass (%)	0.160	0.5 (max)	-
Penetration after RTFOT	53	50 (min)	ASTM D5 EN 1426
Retained Penetration after RTFOT (%)	82	50 (min.)	ASTM D36-06/ EN 1427
Softening Point after RTFOT (%)	58	48 (min.)	ASTM D36-06/ EN 1427

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 in order to have an accurate comparisons.

Coarse and fine limestone aggregates were procured from Dere Beton/Izmir quarry. In order to find out the properties of the limestone aggregate used in this study, specific gravity, Los Angeles abrasion resistance, sodium sulfate soundness, fine aggregate angularity and fat and elongated particles tests were conducted. The results are presented in Table 2. Grading of aggregate was chosen in conformity with the Type 1 wearing course of Turkish Specifications. Table 3 presents the final gradation chosen for limestone aggregate.

Table 2 Limestone physical properties

Test	Results	Spec. Limits	Test Method
Specific Gravity (Coarse Agg.)			
- Bulk	2.704	-	ASTM C127-07
- Saturated surface dry (SSD)	2.717	-	
- Apparent	2.739	-	
Specific Gravity (Fine Agg.)			
- Bulk	2.691	-	ASTM C128-07
- 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 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

3.2 Test methods

3.2.1 Marshall Mix Design

The Marshall Stability method was used to find out the optimum bitumen content for gradation specified in Table 3, in accordance with ASTM D 1559 [30]. Series of test specimens are prepared for a range of different bitumen content so that the test data curves (stability, flow, air void level, bulk specific gravity, voids in mineral aggregate (VMA), voids filled with asphalt (VFA) versus bitumen content) show a well defined optimum value. Consequently, the optimum bitumen content is determined by the bitumen content corresponding to the median of designed limits of percent air voids in the total mix (i.e. 4 %).

3.2.2 Aging procedures

In order to simulate the aging of asphalt in laboratory, the samples were divided into three groups: unaged samples, short term aged samples and long term aged samples. The unaged process does not involve neither short nor long term aging processes. The name of the methods, the conditions (duration and temperature level) of short and long term aging processes are presented in Table 4. It should be noted that, the long term oven aging procedures also involves the short term forced draft oven aging treatment.

As indicated before, AASHTO R30 involves short term forced draft oven aging of loose mixture at 135°C for 4 hours and long term aging of compacted samples at 85°C for 120 hours. NCHRP 9-52 method involves short term

Table 4 Aging methods

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

forced draft oven conditioning of loose mixture at 135°C for 2 hours and long term aging of compacted samples at 85°C for 120 hours. RILEM method involves short term conditioning of loose mixture at 135°C for 4 hours and long term aging of loose mixture at 85°C for 216 hours. Regarding NCHRP 09-54 method, the same short-term conditioning was applied as in AASHTO R30 (4 hours at 135°C). For long term aging, a loose mixture aging at 95°C was used as an aging temperature. The aging was made using a forced draft oven. The picture of the core samples taken from field is given in Fig. 2, while samples produced at laboratory condition are given in Fig. 3 as loose mix and compacted samples.

The duration of aging were determined by the following equation (Eq. (1)):

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

where:

- t_{oven} required oven aging duration at 95°C to reflect field aging (days),
- D depth correction factor,
- A frequency factor,
- Ea activation energy,
- R universal gas constant (this value is taken as 0.0831 kJ/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 (Ea) is obtained corresponding to pavement layer of 20 mm depth from the Table 5.

The pavement temperature (Ti) was determined by following equation (Eq. (2)) developed by Witczak [31]:

$$Ti = Ta \cdot [1 + 1 / (z + 4)] - 34 / (z + 4) + 6, \quad (2)$$

where:

- Ti 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 Ti , the 4 main districts (Dikili, Aliaga, Menemen, Buca) of Izmir City was taken into account and the hourly air temperature values (Ta) were taken from the General Directorate of Meteorology for 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 6 which indicates that, average duration of 4 days should be utilized regarding the simulation of long term aging process.

3.2.3 Indirect tensile strength test (ITS)

ITS test was performed in order to evaluate the short and long term aging condition of the laboratory prepared samples. The test is one of the most popular tests used for evaluation of the aging characteristics of asphalt mixtures, which directly affect the performance of the asphalt pavements. ITS test is often used 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 [32].



Fig. 2 Core samples taken from field



Fig. 3 Laboratory produced loose and compacted samples

Table 5 Climatic aging index coefficients [29]

Pavement layer	Depth correction factor (D)	Pre-exponential factor (A)	Activation energy (Ea)
Surface Layer (6 mm)	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

Table 6 The Toven 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

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 ITS testing is conducted on laboratory aged and field taken samples depending on ASTM D 6931 [33]. Based on this standard, each sample is put in a plastic bag and placed into water for 2 hours at 25°C as conditioning. The air should be soaked by a pipette before this procedure. Following, the cylindrical laboratory prepared asphalt samples and the field core samples were correctly placed on the loading frame. The load was applied (at loading rate of 51 mm/min) in the vertical diametrical plane and creates a tensile load in the horizontal direction of the sample. The maximum load was read and saved on the device as outlined in.

In order to compute the ITS, the following equation (Eq. (3)) is used:

$$St = (2000 \times P_{max}) / \pi t D, \tag{3}$$

where;

- St* indirect tensile strength (ITS), kPa
- P* maximum load, N
- t* specimen height immediately before test, mm
- D* specimen diameter, mm

Aging indices were defined to evaluate the aging effects of laboratory prepared asphalt mixtures. The ratio between the ITS values of short and long-term aged samples and the ITS values of unaged samples was calculated as short-term aging index (SAI) and long-term aging index (LAI) (Eqs. (4)–(5)).

$$SAI = \frac{(\text{ITS value of short term aged specimens})}{(\text{ITS value of unaged specimens})} \tag{4}$$

$$LAI = \frac{(\text{ITS value of long term aged specimens})}{(\text{ITS value of unaged specimens})} \tag{5}$$

4 Results and discussions

4.1 Marshall Mix Design results

The Marshall Stability test was conducted on the samples, that contain different bitumen content in order to determine the optimum bitumen content in accordance with ASTM D1559 [31]. In the experimental study, the optimum bitumen content corresponding to 4 % air void (depending on the maximum and minimum air void level presented in

the General Directorate of Highways Specifications) was selected as 4.60 % (by weight of aggregate). Test results are presented in Table 7. As indicated in Table 7, all parameters (stability, flow, VFA, VMA) corresponding to 4 % air void level are within the specification limits. The influence indication of nanomaterials on basic properties of 50/70 asphalt binder is tabulated in Table 7.

4.2 Indirect tensile strength test results

4.2.1 Results of laboratory aged specimens

For all the investigated aging methods, it was observed that, the indirect tensile stress (ITS) values of the samples increased from unaged condition to long term aged condition as illustrated in Fig. 4. The increase in indirect tensile stress values is considered as an indicator of the effect of aging.

Among the all 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 sample 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.

Table 7 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

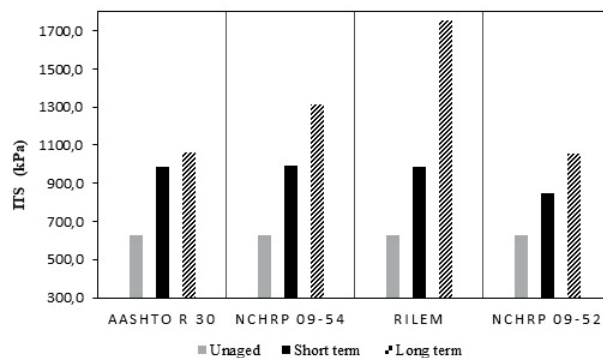


Fig. 4 ITS results of aging method

Short term aging (SAI) and long term aging indices (LAI) were calculated for the evaluation of aging characteristics of the samples. Fig. 5 represents the indices corresponding to each method.

Based on Fig. 5, it can be said that, the lower the aging index values, the less aging effect the bituminous mixture exposes. Among the short term aged samples, the short term aged sample 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 indicates that, RILEM Method produced higher long term index values. Besides, NCHRP 09-52 and traditional method yielded similar long term index values.

4.2.2 Results of field core specimens

The ITS values of both field core samples and the laboratory aged specimens are presented in Fig. 6.

As indicated in Fig. 6, the ITS value regarding to 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 to effect of aging. When comparing the specimen taken from the recently constructed pavement with the laboratory short term aged specimens

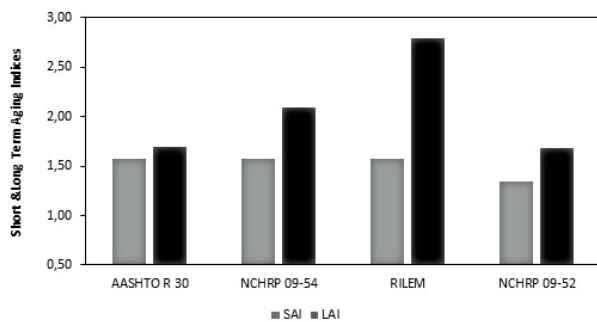


Fig. 5 Short and long term aging index results of all methods

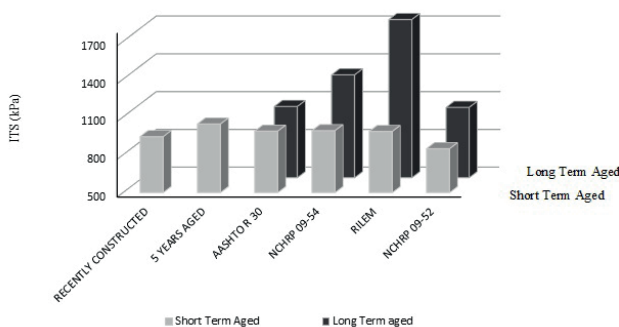


Fig. 6 ITS results of field core specimens

(simulating the aging process during mixing with aggregate, laying and compaction), the ITS value of sample short term aged specimen 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 specimens with 5 years old field specimen, the ITS value of long term aged specimen by AASHTO R30, NCHRP 09-52 and yielded similar values with the ITS value of sample taken from 5 years aged pavement surface.

5 Conclusions and recommendations

This study is aimed to evaluate the aging procedures of hot mix asphalt with current standard (AASHTO R30) 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 been also compared with the samples taken from recently constructed pavement surface and from the 5 years old pavement surface.

The following conclusions can be withdrawn from this study:

- For all the investigated aging methods, the indirect tensile stress values of the samples increased from unaged condition to long term forced draft oven aged conditions.
- Among the all short term forced draft oven aged samples, all of the investigated methods yielded similar ITS values due to the fact that, all the short term oven aging conditions regarding each method do not exhibit significant difference.
- 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. Therefore, the 2 hours forced draft oven aging at 135°C can be recommended as short term aging condition.
- 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.
- A significant variation is observed between the ITS values of long term laboratory aged specimens and 5 years old field core samples, when NCHRP 09-54 method is used. Therefore, AASHTO R30 and NCHRP 09-52 methods are recommended as long term aging methods.

This study covers the utilization of one type of aggregate and 50/70 penetration grade bituminous binder. More research can be made by using different aggregate type as well as bitumen.

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