Effect of Ageing In Various Bituminous Mixes

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Abstract- *The short-term aging properties of neat bitumen were investigated using the rolling thin film oven test (RTFOT) to simulate aging during mixing, compaction and laying of asphalt mixtures, though the actual time of short-term aging in the field varies depending on haulage distances or paving times. The empirical tests, which include penetration and softening points, were conducted to ascertain the binder consistency. The RTFOT was conducted at 1630 C for 70min, 85min, and 100min for bitumen 80/100-penetration grade.*

Results from the study indicated that aging resulted in oxidation of the bitumen with increase in the stiffness of the binder. It was observed that aging increased the viscosity, decreased the binder penetration and increased the softening point of the neat bitumen. The results from the study also indicated that the magnitude of the short-term aging depends on the binder source, and aging time, as with longer aging time, the binder hardness and viscosity increases, thereby decreasing the penetration and increasing the binder softening point.

Keywords- asphalt mixtures, empirical tests, the rolling thin film, aging time, softening point

I. INTRODUCTION

1.1. Background

The durability properties in terms of resistance to ageing of the bitumen binder, is the key factor for the binder characterization in asphaltic mixes, and hence pavement performance. While enormous advances have been made in the use of the BSMs in pavement construction and rehabilitation, the problem of binder hardening in both the plant mix process and in-service conditions remains an area where further research is warranted, (Jenkins, 2000), (Overby et al. 2004), (Gueit et al., 2006), and (Serfass et al., 2008)). Limited research that has been done in the past has indicated that BSMs can age significantly during in-service pavement life (Overby et al., 2004), (Peterson et al., 2000) & (Serfass et al., 2008). In that respect, binder ageing has been a concern of the practitioners globally; hence the need to investigate this behaviour.

1.2.Objectives

The objectives of Task 11 include; - Investigation of binder ageing potential of BSMs in short-term, during mixing and compaction and long-term during pavement in-service period. - Evaluation of rheological properties of foamed bitumen and bitumen emulsion, during laboratory production to determine short-term ageing and recovered bitumen from cores extracted from in-service pavement to determine the long-term ageing. - Investigation of differential ageing (if any) for foamed bitumen and bitumen emulsion after short-term and long-term ageing. - If warranted from the findings, make appropriate recommendations regarding the severity of ageing of different binder types and whether this needs to be considered during the mix design phase.

1.3. Scope of the work

Task 11 focuses primarily on the age hardening behaviour of BSMs. It is known from hot-mix asphalt that age hardening occurs during mixing, construction, and long-term in-service conditions in the pavement. The same principle was used to investigate the ageing behaviour of BSMs as follows: - The short-term ageing was investigated using different type of bitumen from different refineries. The fresh and foamed bitumens' rheological properties properties were tested i.e. viscosity, penetration and softening point with respect to time after circulation of bitumen in the laboratory foam plant (WBL10). The same rheological properties investigated on the residual of bitumen emulsion (after evaporation of moisture). - Further short-term ageing was investigated from compacted and cured specimens, then bitumen was recovered and rheological properties were retested. The long-term ageing was investigated for the cores extracted from the pavement i.e. after five to ten years of trafficking. - The bitumen was recovered (foamed bitumen and bitumen emulsion) using the Abson method according to ASTM D1856-95a, after cold centrifuge with minimum force of 3000 time gravity. - The rate of age hardening on BSMs is discussed and recommendations provided.

II. FACTORS INFLUENCING AGEING BEHAVIOUR OF BSMs

2.1. Background

The foamed bitumen and bitumen emulsion is produced after altering bitumen viscosity properties. The altering of bitumen provides desirable binder, which will allow a stability of mixes in a wide range of mineral aggregates to be effectively used in the pavement construction. Foamed bitumen produced through injection of small quantities of cold moleculised water as a fine mist, into hot bitumen (170o C–180o C) in an expansion chamber at high air pressure.

The emulsifying agent induces electric charge on the dispersed bitumen droplets, which provide stability of the bitumen droplets under water continuous phase. Shell Bitumen, (2003) and Serfuass et al., (2008) indicated that bitumen droplets when break during water evaporation disperse thin film of bitumen in aggregates which provide adhesion properties in the mineral aggregates. At present, the analysis of the variables that influence foam production are presented by Brennen (1983) who identified three factors viz,

- The amount of foam produced (expansion and half-life)
- The amount of water in the foam (usually 2-3%)
- The foaming temperature of bitumen (17OOC-18O0 C).

2.2. Factors influencing age hardening

The age-hardening process of the BSMs might be affected by several factors, all operating at the same time. In assessing these factors, Serfuass et al. (2008), Page et al. (1985) noted that the most critical variables to consider are the characteristics of mixes themselves. Hardening of the original bitumen for the HMA is different from bitumen stabilized mixtures. High temperatures and presence of air during plant mixing, mixing time, and construction are the variables that can cause age hardening of the HMA.. The comparative manufacture and construction process for BSMs and HMA are indicated in Table K.1.

TABLE K.1: The comparative manufacturing process and construction of BSMs and HMA, (Jenkins, 2000)

Parameters	Bitumen emulsion	Foamed bitumen	Hot mix asphalt
Bitumen temperature during mixing	50°C – 70°C	170°C -180°C	140°C -180°C
Aggregates temp	Ambient 25°C	Ambient (25°C) Half warm (40°C-99°C)	Hot (140°C – 200°C)
Moisture content during mixing	60%-70% OMC	70% - 85% OMC	Dry
Type of coating of aggregates	Thin coating coarse particle and cohesion of mix with fines mortal	Partial coating of large particle with spot welding of mix with fines mortar	Coating of larger particle with controlled film thickness
Construction and compaction temp	Ambient (25°C) or Half warm (40°C-95°C)	Ambient or Half warm (40°C-95°C)	140°C–160°C

2.3. The Binder dispersion and voids content

The access of oxygen into the thin film of binder in BSM-foam or BSM-emulsion is linked to the air-void content in the mixture. The high surface area of bitumen in BSM-foam and uneven distribution of the bitumen over the different granular fractions might be the factors resulting in premature ageing. Indeed, as the bitumen disperses preferably in the mortar fraction, the binder is distributed very selectively (in droplets) on a larger aggregates.

Tuffour et al. (1993) expressed the dependence of binder aging (measured by the variation in the value of physical or rheological properties with time) on the variables mentioned above:

$$p(t) = f(T,t,l,K) \text{--------------- Equation (1)}$$

Where,

p(t) = physical or rheological properties of bitumen,T = environmental temperature

t = time ,l = length of diffusion path, K = intrinsic reactivity of bitumen

III. EXPERIMENTAL PROGRAM

3.1. Materials

3.1.1 Bitumen Sources and Grade

Four types of straight bitumen (pen-grade) from two different refineries and slow setting emulsion were procured for the laboratory investigation on this study. Two penetration-grade binders i.e. 80/100 and 60/70, were sourced from NATREF in Gauteng, and two penetrationgrades i.e. 80/100 and 60/70 were sourced from CALTEX in Western Cape. These bitumen types are commonly used for the foaming process in South Africa. The selection was made to compare ageing behaviour of bitumen from different refineries. The slow stetting emulsion (ANi B SS-60) commonly used for recycling was procured from COLAS-SA in Western Cape.

3.1.2 Field cores: Sources and Locations

The pavement with construction history of both foamed bitumen and bitumen emulsion were listed for the experimental investigation. To enable comparison of different environmental conditions, 40 cores were sourced from Western Cape, 36 cores from Gauteng (cored by CSIR) all from South Africa. An additional 6 cores were obtained from Saudi Arabia, transported to Stellenbosch to be included in this study. The lists of pavement sections included in this study are indicated Table K.2.

Table K.2: List of pavement section selected for field ageing studies

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Bitumen emulsion	Foamed bitumen
- N7 TR 11/1 Near Cape Town	- P243/1 near Vereeniging
- MR 27 – Near Stellenbosch	- N7 TR11/1 Near Cape Town
- Grassy park in Cape Town	- Grassy Park in Cape Town
	- Shedgum road in Saudi Arabia

Table K.3 indicates the source of cores and locations, including pavement section, mix type,cores numbers, and location of extractions

3.2. Binder extraction and recovery

3.2.1 Background

The binder characteristics in the mixture cannot easily be examined, without full separation of bitumen from aggregates. Full extraction therefore, has to be performed to recover the bitumen from the mix or RAP to enable examination of its rheological properties. Full extraction implies the use of a particular solvent, and equipment to separate bitumen from aggregates.

3.2.1.1. Recovery of Bitumen from mixes.

Figure K.1. Centrifuge extraction Figure K.2. Abson recovery method

The conventional methods for qualitative extraction and recovery of asphalt from paving mixture are done by centrifuge extraction, vacuum extraction, reflux extraction etc. (ASTM 2003). Different common solvents have been used in the binder extraction and recovery procedures, i.e. e.g. trichloroethylene (TCE), methylene chloride, benzene, 1-trichloroethane (TCA), etc. However due to the carcinogenic and environmental hazardous nature of all chlorinated solvents, the use of these solvents is phasing out (EPA, 2003), and (Collins-Garcia et al. 2000). The extraction and recovery of bitumen has an important influence on the binder characteristics.

3.2.1.2. Recovery of bitumen from Emulsion

Several methods exist for the recovery of bitumen from emulsion. These methods are divided into two types.

1) Thin film oven method,
2) Evaporation method ‘’heating en mass” or a chemical reaction under defined operation conditions.

3.2.1.3. Extraction and Recovery Methods

In this study, due to the availability of equipment, the extraction and recovery of the bitumen from the compacted specimens and extracted field cores was done using standard Abson Method according to ASTM D 1856-95a.

The process consists of cold centrifuge with minimum force of 3000 time gravity. Centrifuge cups were used to collect filler. Trichloroethylene solvent used to separate binder from aggregates. The centrifuge process however observed to be unable to capture all filler; therefore three to four repeat centrifuge processes were necessary. The tendency of foamed bitumen and bitumen emulsion to adhering to the filler particles, create challenges for total separation of mix using the centrifuge. This results in supper filler being recovered with bitumen.

Figure K.1 and K.2 shows the Abson equipment used in the extraction and recovery process possessed by SOILAB in the Western Cape.

The bitumen contents after core extraction calculated from the differences in mass of aggregates, moisture content, and fines in the extracts as follows:

Equation (2)

$$\text{Bitumen content, \%} = \left[\frac{(w_1 - w_2) - (w_3 + w_4)}{(w_1 - w_2)}\right] x100$$

Where:

W1 = mass of test portion ,W2 = mass of water in the test portion

W3 = mass of the extracted coarse aggregates ,W4 = mass of the fine aggregates in the extracts

IV. TESTS RESULTS

4.1. Rheological properties

The **Penetration Test** was performed at 25o C, with 100g of loading weight and needle at a 5 second penetration, according to ASTM D5-IP49. One set of tests was done per sample of recovered bitumen, and repeat set of tests on another sample. The average of the two replicates are recorded in the tables of results. It is important to note that penetration test protocol should be followed precisely; a slight variation can cause significant differences in the results.

The **Softening Point test** was performed according to ASTM D36. The sample and the ring and ball set-up were placed in the 800ml flask and conditioned at 5o C for 15 min. The heating mantle was used to heat the water at a constant rate of temperature change of 5o C per mininute until the bitumen softens and moves with the ball bearing 25mm below the ring. The temperature of the water is recorded and the difference in temperature between two rings should not be more than 1o C.

The **Viscosity Test** was done according to ASTM D4402 using a Brookfield Model DV-I viscometer with thermocel temperature control system. The selected spindle was SC-29 for the 60o C and SC-21 for the 135o C. Temperature range was 60o C and 135o C to determine the viscosity of bitumen at field conditions and mixing temperature. The spindle was equilibrated in the clean Thermocel chamber for 15 minutes. 13g of sample for 60o C and 8g for 135o C was poured in the tube and placed in the Themorcel (with spindle temporally removed). The spindle is hooked to the viscometer and inserted into the bitumen and left to reach equilibrium for further 15 minutes. The spindle and torque speed is selected from the digital panel and viscometer started. The torque of the viscometer is between 2 to 98% of full scale, the reading with the higher torque percentage recommended for more accurate. Three readings are recorded at an interval of 60 seconds and averaged. However, due to sensitivity of the readings, the spindle is left to rotate for 15 minutes and thereafter the readings are taken. The unit of dynamic viscosity is Pascal-Second (Pa.s) which is 1N.sec/m2 . The cgs unit is the gm/cm.s, which is Poise or Centipoise cP, at a given temperature,

1 Pa.s = 1000 mPa.s = 10 Poise = 1000cP
Kinematics viscosity (ע) is the ratio of the dynamic viscosity (η) to the density (δ) of a liquid such that;

$$\nu = \eta / \delta \text{ --------------- Equation (3)}$$

Where,
ע = Dynamic viscosity in Pa.s
η = Kinematic viscosity in mm2/s
δ = Density in kg/l at the temperature under consideration

The unit of kinematic viscosity is mm2 /s or cgs = cm2/s, which is stoke conveniently converted centistokes as follows,
1 mm2 /s = 0.001 cm2 /s = 1 centistoke

The SABS 307 standard specification for the penetration, softening point and viscosity of bitumen are indicated in Table K.4.

4.1.1 Laboratory test results

4.1.1.1. Penetration, Softening point and Viscosity.

The consistency of foamed bitumen after different periods of circulation (i.e. 1hr, 4hrs, 8hrs), was done to simulate a short term age hardening of foamed bitumen during mixing period. The age hardening of base bitumen and foamed bitumen at different circulation times is indicted in Table K.5

PENETRATION INDEX (PI)

The penetration Index (PI) of bitumen from rheological properties is determined to show the characteristics in temperature susceptibility. The PI is determined by the relationship between bitumen penetration value and the softening point as indicated in Eq 4, (Shell bitumen, 2003).

Equation (4)

Where,
SP = Softening point Pen = Penetration at 25o C

4.1.2 Field cores test results

4.1.2.1. Penetration, Softening point and Viscosity

The consistency of recovered bitumen from foamed bitumen and emulsion mixes (cores) was investigated for long-term ageing behaviour. The descriptions of the investigated pavement sections are detailed below:

Grassy park road in Cape Town, 5th Avenue and 3rd Avenue road sections were rehabilitated in 1999 by recycling 200mm CTB with addition of 1.5% foamed bitumen and 2% emulsion respectively. 1% cement was also added on both sections during recycling. The surface was covered by a 40mm asphalt wearing course, (UWP, 1999).

Table K.11: Recovered bitumen properties from Shedgum Road

Properties	ST 08 (RR1) BWP				ST06 (LL3) OWP			
	Layer 1 TOP	Layer 2	Layer3	Layer 4 BOTTOM	Layer 1 TOP	Layer 2	Layer 3	Layer 4 BOTTOM
Penetration 25°C [dmm]	22	24	22	21	16	18	15	17
Softening point [°C]	68.0	64.0	68.0	68.4	76.0	71.0	74.0	75

Table K.12: Recovered bitumen properties from Grassy Park Foamed BSM section

Properties	Robot		Straight section		
	BWP	OWP	YL	OWP	BWP
Penetration 25°C [dmm]	49	15	37	5	outlier
Softening point [°C]	55.8	80	51.2	70.7	44.6
Viscosity at 60°C [Pa.s]	810	N/A	411	NA	108.6
Viscosity at 135°C [Pa.s]	0.932	5.65	0.532	2.283	0.278

Note: The result of more than 80 penetrations is considered outlier as the value might have been a result of residual solvent in the recovered bitumen sample.

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Table K.13: Recovered bitumen properties from Grassy Park Emulsion section
Table K.14: Bitumen properties used in Grassy Park roads rehabilitation, UWP (1999)

The extraction and recovery of bitumen from field cores extracted from N7 TR 11/1 Near Cape Town, and P243/1 near Vereeniging from Gauteng are on going. It is anticipated that after completion of tests on these cores a broader knowledge on long-term age hardening of BSMs will be provided.

As part of the ongoing tests, three temperature buttons were installed in the N7 road section on different BSM layers. The aim of installing the buttons was to record temperature and humidity variation in BSM layer for a period of one year. The effect of temperature and humidity variation in the BSMs can then be correlated with the age hardening behaviour of the recovered bitumen from BSMs on the same sections.

V.ANALYSIS AND DISCUSSION OF RESULTS

5.1. Laboratory investigation on short term ageing, for base bitumen versus foamed bitumen

Figure K.3: Penetration versus ageing time (hr) of base bitumen versus foamed bitumen at temp. 170o C-180o C

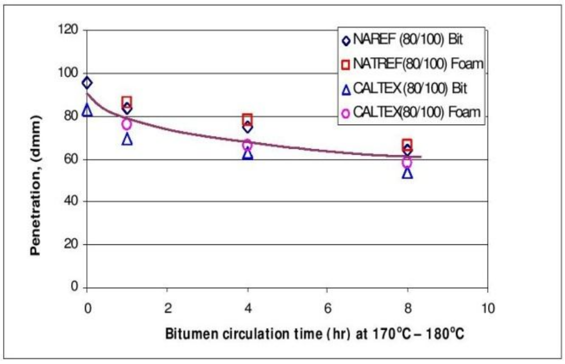

Figure K.3: Penetration versus ageing time (hr) of base bitumen versus foamed bitumen at temp. 170°C-180°C

The consistency of foamed bitumen with respect to mixing time i.e. 1 hr, 4hr, and 8hr, for 80/100 penetration binder, is presented in Figure K3. It is clear from Figure K3 that base bitumen for both NATREF and CALTEX refineries undergoes age hardening of approximately 30% in short term, for a period of 8 hours heated at 170o C – 180o C. Similar behaviour occurs for the foamed bitumen that is produced after ageing of the base bitumen i.e. the foaming process does not alter this trend. Shell bitumen (2003) has reported the same effect of age hardening on base bitumen of the HMA for the short-term ageing.

VI. COMPARISSION OF AGING CHARECTERSTICS

comparison of aging characteristics of warm mix asphalts involving natural and synthetic water containing additives

When comparing the aging characteristics of hot and warm mix asphalts from a technical point of view, it can intuitively be expected that a warm mix asphalt would be less subjected to aging-induced failures due to lower application temperatures. Since the side effects of warm asphalt technology should be investigated distinctly. This study addresses the aging investigation of properties of bituminous mixtures containing two (i.e., natural and synthetic zeolite) water based additives available on the market.

6.1. INTRODUCTION

Conventional Hot Mix Asphalt (HMA) covers the majority of the field pavement practices around the world. Since the human being has noticed changes in temperature of the atmosphere, concern about global warming issues has led the researchers to look for applications with lower temperatures. In addition to ecological issues, economic matters also were taken into account by lowering application temperatures. The less the application temperatures, the less fossil fuels is used to apply a bituminous layer. This issue is also important from the sustainable development point of view. In this regard, new technologies in pavement engineering was born. These new technologies are generally called Warm Mix Asphalt (WMA).

6.2. EXPERIMENTAL

6.2.1. Materials

Table 1. The properties of the base bitumen.

Test	Specification [[Q6: Q6]]	Results	Specification Limit
Penetration (25 °C; 0.1 mm)	ASTM D5-97 EN 1426	63	50 to 70
Softening Point (°C)	ASTM D36-95 EN 1427	49	46 to 54
Viscosity at (135 °C), Pa.s	ASTM D4402-06	0.51	-
Thin Film Oven Test (TFOT); (163 °C; 5 hr)	ASTM D1754-97 EN 12607-1		
Change of Mass (%)		0.07	0.5 (max.)
Retained Penetration (%)	ASTM D5-05 EN 1426	51	50 (min.)
Softening Point after TFOT (°C)	ASTM D36-95 EN 1427	51	48 (min.)
Ductility (25 °C), cm	ASTM D113-07	100	-
Specific Gravity	ASTM D70-03	1.030	-
Flash Point (°C)	ASTM D92-01 EN 22592	260+	230 (min.)

The bitumen used in this study was 50/70 penetration grade bitumen which was provided from TUPRAŞ, Izmir refinery. This grade of penetration is commonly used in Izmir due to climatic conditions. In order to characterize the properties of the base bitumen, conventional test results for base bitumen conducted at the DokuzEylul University (DEU), Highway Materials Laboratory are given in Table

6.2.2. Experimental plan

Based on the regarding modification temperature and mixing times for natural and synthetic zeolites, WMA bitumens were produced just before the mixing with aggregates process. Within the scope of this study, both natural and synthetic zeolites were added directly to the bitumen itself since it is possible to add the water based additive into the mixture. Although some studies say that adding zeolites directly to the bitumen will result in a more consistent mixture9, it is understood that actually the proper way of using zeolites as WMA additives is to add them directly into mixture during mixing phase in order to provide adequate expansion of bitumen.

The experimental curves were plotted for base and additive containing bitumens. Curves for various bitumen types are presented in Figure 1. Acceptable equiviscous bitumen temperatures for mixing was chosen as the range corresponding to 0.17±0.02 Pa.s and acceptable temperatures for compaction was chosen as the range matching 0.28±0.03 Pa.s.

Following the production of natural and synthetic zeolite modified bitumens, WMA mixtures were prepared based on the determined mixing temperatures. The optimum bitumen content was determined by the Marshall mix design method. The optimum bitumen content for base bitumen, natural and synthetic zeolite modified bitumens were respectively determined as 4.76%, 4.56% and 4.32% by weight of the aggregates. The aggregates were placed in an oven adjusted for proper temperature the day before, to be completely dried and ready for mixing.

All specimens were tested for Indirect Tensile Strength (ITS) after being cured. To perform this task, ASTM D6931-12 [[Q5: Q5]] "The standard test method for indirect tensile strength of bituminous mixtures" was taken into account. The ITS test was conducted by the Marshall stability and flow apparatus. The loading rate was set to 51 (mm/min) in case for ITS. To be adequate and unbiased, three specimens for each additive content were prepared and tested randomly. The ITS results can give the evaluation keys in terms of low temperature and fatigue cracking of asphalt pavements. Studies introduce ITS result as a good indicator in predicting the laboratory rutting potential of asphalt mixtures[11]. This test is widely used in investigation of moisture induced damages of bituminous mixtures.

The raw data recorded from the test device should be processed using the following Equation 1 to obtain ITS values:

$$S_t = (2000 \times P) / (\pi \times t \times D) \quad (1)$$

Where:

S_t = Indirect tensile strength (ITS), kPa,P = Maximum load, N t = Specimen height immediately before test, mm,D = Specimen diameter, mm.

VII. CONCLUSIONS

The age hardening of BSMs and fundamental characteristics of BSMs associated with short-term and long-term age hardening have been investigated through laboratory testing. Based on the data of the study, the following conclusions are drawn:

- The time bitumen is kept in circulation in the laboratory plant at eleveted temperature before making of BSM-foam contributes to the ageing of the binder, especially after 8 hours. The effects of ageing are more notable for the softer bitumen (80/100) than hard bitumen (60/70), with 30% drop of penetration for 80/100 bitumen, compared to drop of 12-17% on the 60/70 bitumen.
- It is apparent from the study that some short-term age hardening of foamed bitumen during mixing occurs. However, the trend follows that of the age hardening of the base bitumen. Nevertheless, the foaming process in

itself does not alter the bitumen properties. For the bitumen emulsion, no age hardening occurred during mixing time because elevated temperature is not used in the mixing process

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