THE USE OF AN EXTENDED ROLLING THIN FILM AGEING METHOD AS AN ALTERNATIVE TO THE PRESSURISED AGEING VESSEL METHOD IN THE DETERMINATION OF BITUMEN DURABILITY

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Abstract

The South African Bitumen specification framework currently does not address durability aspects of bitumen adequately therefore the Road Industry identified the necessity for the development of an accelerated ageing protocol. The Pressurised Ageing Vessel (PAV) is an internationally accepted ageing method for the simulation of long-term binder ageing but the equipment is not readily available in South Africa. Alternatively, the Rolling Thin Film Oven Test (RTFOT) equipment is readily available and presented a more cost effective approach for the accelerated ageing of bitumen. The objective of the research outlined in this paper was to determine if the Extended Rolling Thin Film ageing method could be used as an alternative to the Pressurised Ageing Vessel method in the determination of bitumen durability.

With the PAV test used as a benchmark, the test conditions for the RTFOT test method were tailored to produce binder residues with similar “stiffness levels”. The empirical and rheological properties of the binder residues obtained support the proposed high temperature oxidation protocol. It is furthermore possible to calculate the rate of ageing to be used as a durability parameter at various temperatures.

1 INTRODUCTION

1.1 Bitumen Durability

When bitumen is exposed to air, it oxidises and the chemical composition as well as the physical properties change (Hagos, 2007). Ageing, associated with the temperature / time relationship, is well-known to impact on the durability of bitumen in surfacing applications. By varying the primary ageing factors like time and temperature, different ageing results can be achieved in a laboratory environment. Higher temperatures result in an increased rate of ageing and the level of ageing is more severe over a longer time period of exposure.

The three primary factors which can be varied to accelerate the rate of ageing of bitumen under laboratory conditions are time, temperature and air pressure. The secondary factors are increased bitumen viscosity due to ageing and the exposed surface area of the bitumen sample which reacts with oxygen during the test also impacts the rate of ageing (BiTVal report 2006, Oliver 2007 and Bahia, 2007).

The ideal outcome would be to simulate the ageing conditions yielding similar binder stiffness as observed in actual field ageing and similar binder chemistry as observed in actual field ageing.

1.2 South African Bitumen Specification Development

The South African bitumen specification dates back to 1951 when it was based on penetration. In 1994 the Road Grade nomenclature was adopted, classifying bitumen based on viscosity limits at 60°C with penetration and Softening point limits as optional. In 1995, there were concerns about SA specifications not addressing the compositional balance of bitumen chemical and physical properties. As a result, an investigation was conducted and the outcome was reported at CAPSA 1999 (van de Ven & van Assen 1999). In 1997 the SANS 307 classification reverted back to the penetration grading system, retaining viscosity as compulsory property. A Low Temperature Ductility (LTD) test and changes in properties such as penetration, softening point, viscosity and ductility after RTFOT ageing was focussed on addressing the compositional balance concerns. Although SANS 307 relied strongly on ductility at low temperature, the relevance of the low temperature ductility as a method to ensure a good compositional balance in bitumen was challenged. The sensitivity of the low temperature ductility test was investigated. Muller (et al 1999) demonstrated that the suitability of the ductility test and limits specified for South African penetration grade bitumens had to be reconsidered. Other physical properties should be investigated to supply additional insight into the low temperature performance and compositional balance of the penetration grade bitumens. The measurement of the low temperature force-ductility properties indicated that the bitumen's toughness changed with the variation in deformation speed and the temperature at which the tests were performed (both before and after the rolling thin film oven test) and the test was eventually abolished in 2002 (Standards South Africa, 2005).

Developments towards a Performance Graded (PG) system and PG concepts linked to the SA Standards were proposed by Van de Ven, Bahia and Jenkins (2004). A potential improvement to the SANS 307 is to have a direct approach in which grades are based on actual performance factors. However, this approach requires expensive and complex equipment to obtain performance related rheological properties. As a result, the idea of using a PG system based on the empirically estimated performance related properties evolved. Based on the PG classification system, it is suggested that grades are selected based on the application conditions including pavement temperature and traffic. The criteria for accepting bitumen in these grades are based on engineering properties that are performance related but derived from simple index properties such as penetration, softening points or viscosity. This way, a performance based system can be established without using expensive rheological equipment (van de Ven et al. 2004).

Bitumen durability plays an important role in bitumen specifications in various parts of world. In Europe and the USA, more emphasis is placed on asphalt binders whereas, Australia, South Africa and New Zeeland (NZ) the emphasis is more focused on sprayed seals. Although durability is an optional test in Australian specification, durability is viewed as an important property and has been incorporated into the NZ, European standards and the US PG system (Chin 2007).

1.3 Accelerated Binder Ageing Requirement for South Africa

The South African Bitumen Specification framework currently does not have a test method addressing the durability aspects of bitumen. The need therefore exists within the South African Road Industry to have a tool and mechanism to evaluate the durability of bitumen and assist with the "prediction of performance" related to the binder properties.

In the USA, the Pressurised Ageing Vessel (PAV) is used extensively in the SUPERPAVE performance grading, binder selection and classification systems. In an attempt to develop a South African durability protocol, the cost effectiveness and availability of equipment also needed to be considered. Comparing the durability and performance prediction systems utilised in the USA, Europe and Australia, it was found that the Australian approach which utilises the Rolling Thin Film Oven Test (RTFOT) was the most cost effective and appropriate method for accelerated ageing of bitumen for the South Africa Roads Industry.

A need therefore exists to determine if the more cost effective extended Rolling Thin Film ageing method could be used as an alternative to the Pressurised Ageing Vessel method in the determination of bitumen durability.

2. COMPARISON OF ACCELERATED AGEING METHODS

A review of available durability and ageing protocols was conducted to establish the type of equipment and requirements used internationally, the applicability to South African requirements and availability, cost efficiency, easy of use and reliable interpretation. It is recognised that different criteria and requirements exists for bitumen in the different applications such as seals, asphalt, crack seal and waterproofing applications. The main focus of this evaluation was however aimed at the first two applications due to the specific need identified in the South African Road Industry.

The accelerated ageing methods identified include the Pressurised Ageing Vessel (PAV) used in the SUPERPAVE PG grading system in the USA, which is a well known test method widely referenced and specified. The rotating cylinder ageing test (RCAT) is currently proposed for inclusion in the European specifications. In Australia, researchers at the Australian Road Research Board (ARRB) have developed a durability protocol and a seal performance prediction model utilising field data. The Australian method relies on a modified rolling thin film oven (RTFOT) test to accelerate the oxidative ageing of bitumen. The PAV test (used in Europe and the USA) is not regarded as suitable for Australia's sealing grade bitumens since binder hardening in seals is more rapid than in asphalt (Chin 2007).

Durability (resistance against ageing / conditioning) is a key requirement of bitumen in the asphalt pavement and seal applications. Test procedures are required for two ageing situations that occur in practice:

- The pre-treatment is required for the preparation of binder samples for further testing, in order to simulate the condition expected in practice. Binder conditioning and evaluation indicating the resistance to long-term hardening.

Binder Conditioning Regimes are split into two categories in Table 1; short-term ageing and long-term ageing. A shortened description of the published methods is given below.

Table 1 Bitumen Ageing Conditioning test methods
Source CEN TC 336 (2005) and BiTVal Report 2006

Test Method	Ageing	Reference
RTFOT (Rolling Thing Film Oven)	**Short Term Ageing**	EN 12607-1 / ASTM 2872
TFOT (Thin Film Oven)		EN 12607-1 / ASTM 1754
RFT (Rotating Flask Test)		EN 12607-1
Modified RTFOT*		**Await New Developments**
PAV (Pressurised Ageing Vessel)	**Long Term Ageing**	prEN14679 / AASHTO PP1-98
HiPAT (High Pressure Ageing Test)		Covered by PAV
RCAT (Rotating Cylinder Ageing Test)*		prEN 15323
LTRFT (Long Term Rotating Flask Test)		Development work shelved
ERTFOT163 (Extended RTFOT at 163°C) **ERTFOT100 (Extended RTFOT at 100°C)***		**New proposed protocol**

* (steel rod assisted)

2.1 Short-term ageing methods

2.1.1 Rolling Thin Film Oven Test

Short-term ageing of binders is best simulated by the rolling thin film oven test (RTFOT). The RTFOT is one of the most commonly used standardised tests to address the ageing related to hot mix asphalt mixing, transporting and paving conditions. The purpose of the test, in accordance with EN 12607-1 and ASTM D 2872, is to measure the combined effects of heat and air on a thin film of bitumen or bituminous binder in permanent renewal. The RTFOT has been adopted in South Africa (SANS 307), Australia (AS 2008), New Zeeland (TNZ M/1: 2007), Europe (EN12591) and the in the USA (Standard Specification for Performance Graded Asphalt Binder AASHTO Designation: MP1-98).

2.1.2 Alternative Short-term Ageing tests

Other test methods such as the thin film oven test (TFOT) EN 12607-2 and ASTM D 1754 and the rotating flask test (RFT) EN 12607-3 are also available but, due to limitations associated with the test, they have not been considered. As a result of the high viscosity of modified binders, one of the main problems with using the RTFOT for modified bitumens is that these binders will not form a thin film to expose fresh surface area inside the glass bottles during the test. The sample rolls into a ball and, in addition, some modifed binders have a tendency to roll out of the bottles. The modified rolling thin film oven test (MRTFOT) was developed to overcome these problems. The test conditions are identical to the standard

RTFOT with the exception of the fact that a set of 127 mm long by 6.4 mm diameter steel rods are positioned inside the glass bottles during oven ageing. In principle, the steel rods spread the binder into thin films through the action of shearing forces, therefore overcoming the problem of ageing high viscosity binders. The BiTVal authors state that: "Initial trials of the MRTFOT indicate that the rods do not have any significant effect on the ageing of conventional penetration grade bitumens". The principles described in the MRTFOT were adopted when the extended RTFOT at 100°C was evaluated as a potential accelerated ageing test.

2.2 Long-term ageing regimes

2.2.1 Pressure Ageing Vessel (PAV)

During the Strategic Highway Research Programme (SHRP) in the USA, the Pressure Ageing Vessel (PAV) was developed to simulate long-term, in-service oxidative ageing of bitumen in the field. The binder is first exposed to hardening of bitumen in the RTFOT or TFOT (simulating short-term ageing) and then followed by oxidation of the residue in a pressurised ageing vessel. The PAV test has also been standardised in the USA as AASHTO PP1-98 but in Europe the standard for the PAV is still in a draft form as prEN 14769.

2.2.2 Alternative Long-term ageing regimes

The rotating cylinder ageing test (RCAT) device was developed by the Belgian Road Research Centre. Test conditions can be varied and were selected in such a way to accelerate ageing without compromising the chemical processes that actually occur. This test truly reproduces the in-service ageing in the road surface (Belgian Road Research Centre, A63). The European standard for the RCAT is still in draft form as prEN 15323. There were no known equivalent standardised methods at the time of writing the BiTVAL report. A European standard for the test has since been submitted for CEN enquiry as prEN 15323.

RCAT is a device for ageing/conditioning bituminous binders. The RCAT tests are classified as tests simulating short-term ageing (STA) or tests simulating long-term ageing (LTA). It can be used for paving grade bitumen, modified bitumen and mastics. Depending whether the RCAT is used for short-term or long-term conditioning, a constant flow of **oxygen** (LTA) or **air** (STA) is fed into the ageing vessel. The testing cylinder rotates on two round drive bars in a ventilated oven at 1 revs/min or 5 revs/min respectively for LTA or STA conditioning during testing.

2.2.3 Australian ARRB durability test

The ARRB Durability Test accelerates field hardening under laboratory conditions. A 20 mm bitumen film is aged by means of air oxidation in an oven at 100°C. The durability is measured by the amount of days to reach a distress viscosity value. This distress viscosity is measured by means of a sliding plate viscometer at 45°C. The Australian method includes the addition of volatile cutters to achieve a very thin bitumen film for accelerated ageing. Many years of data correlation and validation have provided the Australian researchers with a reliable prediction model for seal applications. Sprayed seals are a key component of the Australian national road network, accounting for close to 90% of all weather surfacings in Australia. Therefore, like in South Africa, a large percentage of the Australian road network consists of seals rather than asphalt. The focus in Australia was therefore aimed at the determination of the rate of binder ageing in seals. It is considered that this durability test is not appropriate for indicating the long-term oxidation resistance of binders containing modifiers such as polymers or polyphosphoric acid (Oliver 2007).

3 DEVELOPMENT OF A SOUTH AFRICAN ACCELERATED AGEING TEST

It appears that researchers have identified that similar factors influencing binder ageing in asphalt applications also affect ageing in seal applications - only the mode of failure is different. By combining the principles employed in the abovementioned test methods, the basic philosophies which are valid for asphalt and seals application were combined in one test method. The idea of an extended RTFOT durability protocol was born.

In essence, the South African approach to the long-term laboratory ageing could be described as a hybrid approach. It utilises the RTFOT equipment which is more readily available than any of the other accelerated ageing equipment employed elsewhere. By modifying the RTFOT test condition as described in this paper, the Extended RTFOT at 163°C is shown to age bitumen residues to the PAV stiffness levels. When reducing the temperature to 100°C and with the assistance of a steel rod a continuous fresh surfaces area is created and resembles an RCAT-type approach but on a much smaller scale. The approach presented in this paper does not adopt the use of volatile solvents to achieve thin binder films like the ARRB durability test. The extended duration and the reduced temperature approach addresses the concerns related to relevance of the accelerated ageing test for binders use in seals as well as test conditions that closer resembles the maximum road temperatures – a philosophy similar to the PAV, RCAT and ARRB durability tests.

4 EXPERIMENTAL

4.1 RTFOT test conditions

The standard RTFOT involves rotating eight glass bottles, each containing 35 g of bitumen. The bottles are placed in vertically rotating carousel, while hot air is blown into each sample bottle. The air flow is set at a rate of 4000 ml/min. The vertical circular carriage rotates at a rate of 15 revs/min. Bitumen flows continuously in relatively thin films at a temperature of 163°C for 85 min around the inner surface of each container during the test (75minutes was specified in older versions of ASTM D 2872).

4.2 PAV test conditions

The PAV procedure adopted in the USA, entails ageing 50 g of bitumen in a 140 mm diameter container (giving a binder film that is approximately 3,2 mm thick) within the heated vessel, pressurised with air to 2,07 MPa for 20 hours at temperatures between 90 °C and 110 °C For the purpose of this research project a temperature of 100°C was selected.

4.3 Extended RTFOT test conditions at 163°C (ERTFOT163)

The ERTFOT163 involves rotating eight glass bottles, each containing 35 g of bitumen. The bottles are placed in vertically rotating carousel, while hot air is blown into each sample bottle. The air flow is set at a rate of 4000 ml/min. The vertical circular carriage rotates at a rate of 15 revs/min. Bitumen flows continuously in relatively thin films at a temperature of 163 °C around the inner surface of each container during the test. The test duration was extended to 325 minutes and samples were extracted at 205 minutes to establish a trend-line.

4.4 Extended RTFOT test conditions at 100°C (ERTFOT100sra)

The ERTFOT100sra involves rotating eight glass bottles, each containing 35 g of bitumen. The bottles are placed in vertically rotating carousel, while hot air is blown into each sample bottle. The air flow is set at a rate of 4000 ml/min. The vertical circular carriage rotates at a rate of 15 revs/min. At a temperature of 100°C around the inner surface of each container during the test bitumen does not flow continuously in thin films and an electroplated steel rod 129mm long with a diameter of 8mm is inserted into each glass bottle to ensure a thin film is continuously spread on the inside of the glass bottles. The test duration was extended for 48 hours. Samples were extracted at 24hours to establish a trend-line. The achievement of a thin binder film is steel rod assisted (=sra).

4.5 Binder samples

Bitumens from five sources were used in this research and the 60/70 penetration grade was selected based on the fact that it is used as an asphalt grade binder in South Africa. When comparing the viscosity range for 60/70 in South Africa it was possible to compare the 60/70 bitumen to the binder used extensively in seals in Australia.

60/70 penetration grade bitumen from a single source was used for the comparison of ageing methods in this paper. Similar trends were observed for 60/70 bitumen from other sources investigated as part of the development of a South Africa Accelerated Ageing Protocol

4.6 Evaluation of binder properties (before and after ageing)

4.6.1 Empirical testing

The standard test methods specified in SANS 307 for penetration grade bitumens were used to determine the penetration, softening point and viscosity values.

4.6.2 Rheological evaluation

The dynamic shear rheometer testing protocol, specified for evaluating binder properties from the PAV test in the PG grading specification, was used. The rheological properties were evaluated over a temperature range between 10°C and 40°C.

5 RESULTS AND DISCUSSION

5.1 Comparison of the Empirical Properties

The measurement of the SANS 307 properties at the various intervals reveals a clear trend which is established, indicating the affect of oxidative hardening over time. When the penetration values are compared in Figure 1, the rate of ageing is much more pronounced between the virgin bitumen (time = 0) and RTFOT ageing (time = 85 minutes). For the ERTFOT163, the relative age hardening is similar to the aged hardening observed for PAV ageing (Figure 1). Similarly the ERTFOT100sra reaches the similar PAV penetration values after 48 hours (Figure 2).

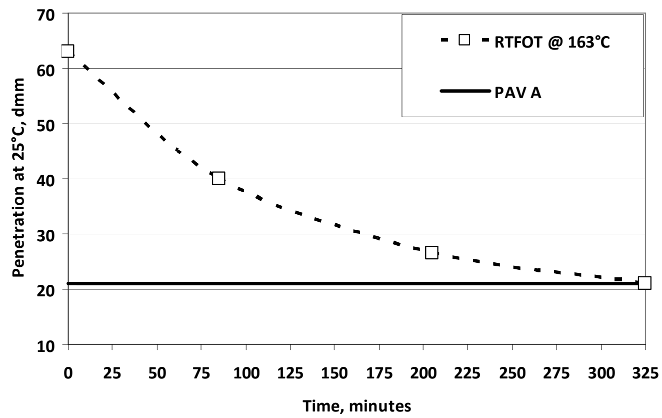

Figure 1 **Relative Ageing measured by means of Penetration values**

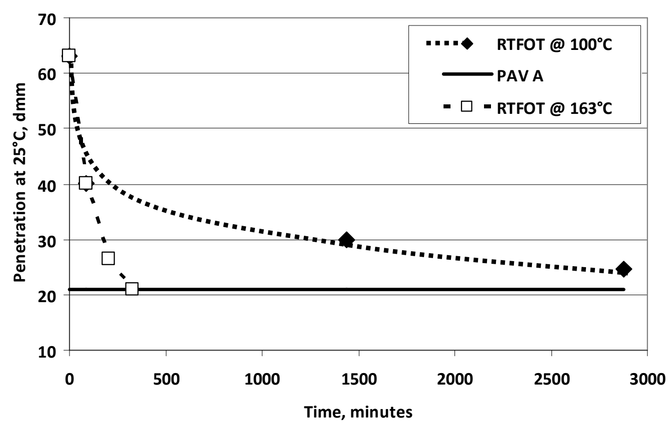

Figure 2 **Relative Ageing measured by means of Penetration values (ERTFOT163 vs. ERTFOT100 vs. PAV)**

The rate of ageing for ERTFOT163 calculated at 85, 205 and 325minutes provides a means of comparison for the ERTFOT100 calculated at 85, 1440 and 2880 minutes. The rate of ageing expressed at the change in the penetration value divided by the difference in time (hours) is graphically portrayed in Figure 3. The rate of reduction in penetration value reduces as the binder ages and will eventually reach a point where it will no longer change at 25°C.

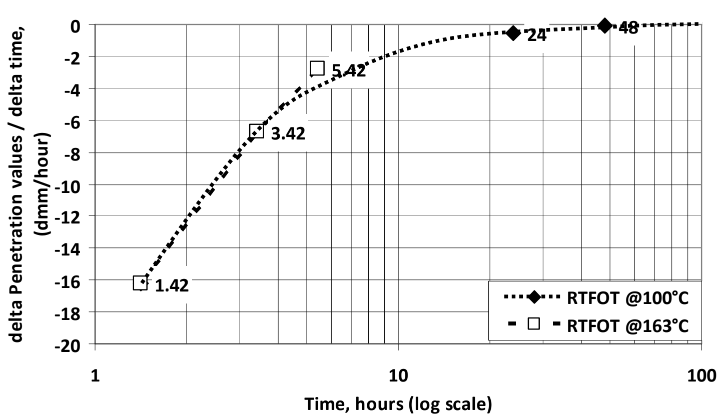

Figure 3 **Rate of ageing expressed as changes in Penetration values at 25°C (Extended RTFOT at 100°C and 163°C vs. PAV)**

Similarly, when the Softening Point values of the binder residues aged by means of the RTFOT at 163°C and 100°C are compared to softening point valued of binder residues aged by means of the PAV, the trends can be used to predict the relative rate of ageing at the two different temperatures.

The ERTFOT163 softening point value at 325 minutes and the ERTFOT163 softening point value at 48 hours are similar but both are higher than softening point value obtained by means of PAV ageing (Figure 4).

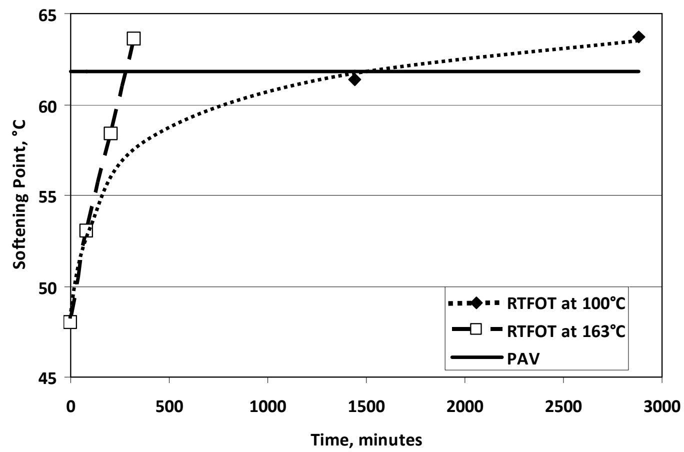

Figure 4 Relative Ageing measured by means of Softening Point (Extended RTFOT at 100°C and 163°C vs. PAV)

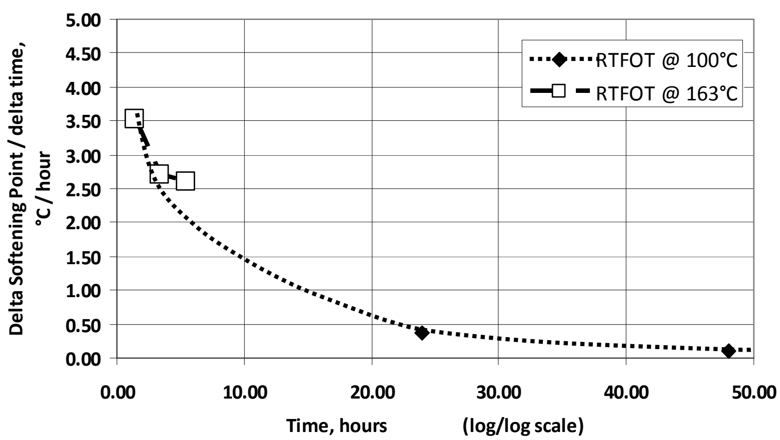

Figure 5 Rate of ageing expressed by changes in Softening Point values (Extended RTFOT at 100°C and 163°C vs. PAV)

As the binder ages and the binder become stiffer, the rate in the increase of the softening point reduces. Eventually there will no longer be a measurable change in the softening point value. By this time, the binder will no longer be flexible enough at low temperatures and the binder will be prone to cracking (Oliver, 1987 and Bahia 2007).

Similar trends are observed for the Brookfield viscosity at 60°C (Figure 5) and 135°C (Figure 6). The difficulty with viscosity measurements at these temperatures are that the aged binder does not act like a Newtonian fluid and, in terms of cracking behaviour would be irrelevant for assessment of the durability of binders (Oliver 2007).
In terms of the relevance of test conditions for determination of binder durability, the visco-

elastic behaviour of the binder residues needs to be considered at lower temperatures. The fundamental rheological properties determined by means of the dynamic shear rheometer provide a better understanding of the affect of ageing on the binder.

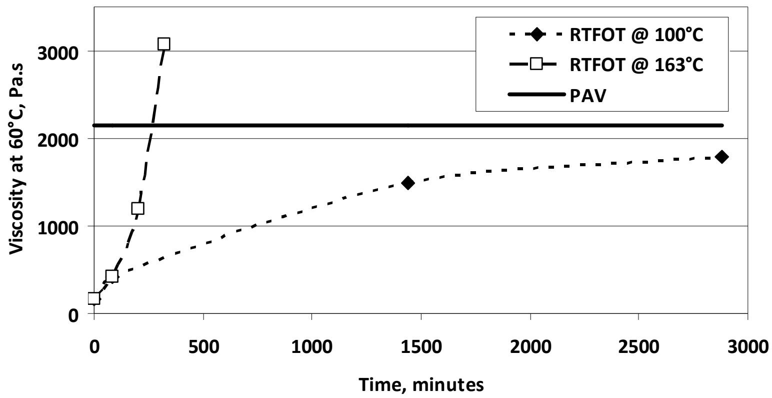

Figure 5 Rate of ageing expressed by changes in Viscosity values at 60°C (Extended RTFOT at 100°C and 163°C vs. PAV)

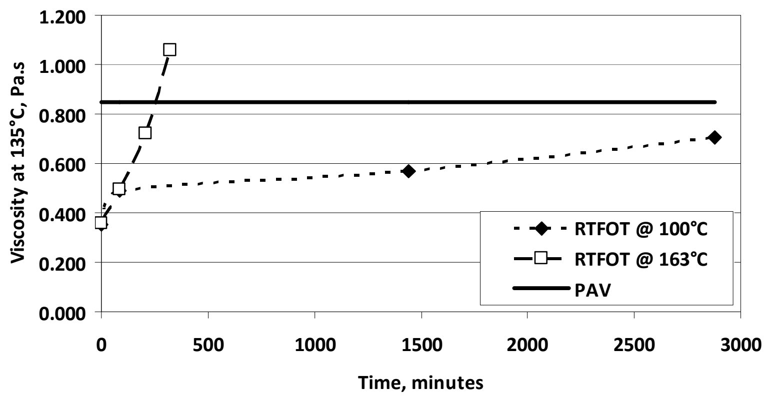

Figure 6 Rate of ageing expressed by changes in Viscosity values at 135°C (Extended RTFOT at 100°C and 163°C vs. PAV)

5.2 Comparison of the fundamental rheological properties

Comparing the fatigue factor {G* x sin(delta)} for the virgin binder and aged binder residues over the temperature range between 10°C and 40°C for the different ageing regimes (Figure 7), it is clear that the extent of ageing at 100°C and 163°C vs. the PAV aged binder residues are similar. The objective to reach the bench-mark stiffness (PAV value) was achieved with both the extended RTFOT at 163°C after 325 minutes and at 100°C at approximately 48 hours.

This trend is demonstrated across the whole temperature range but is more pronounced at the lower temperatures range 10°C to 25°C where the binder's elastic modulus will dominate because the binders are stiffer. Two options where considered to observe the relative rate of ageing between the ERTFOT163 and ERTFOT100. Figure 8 illustrates the trends for fatigue factor respectively at 10°C and 25°C with the magnitude of stiffness the differentiating factor.

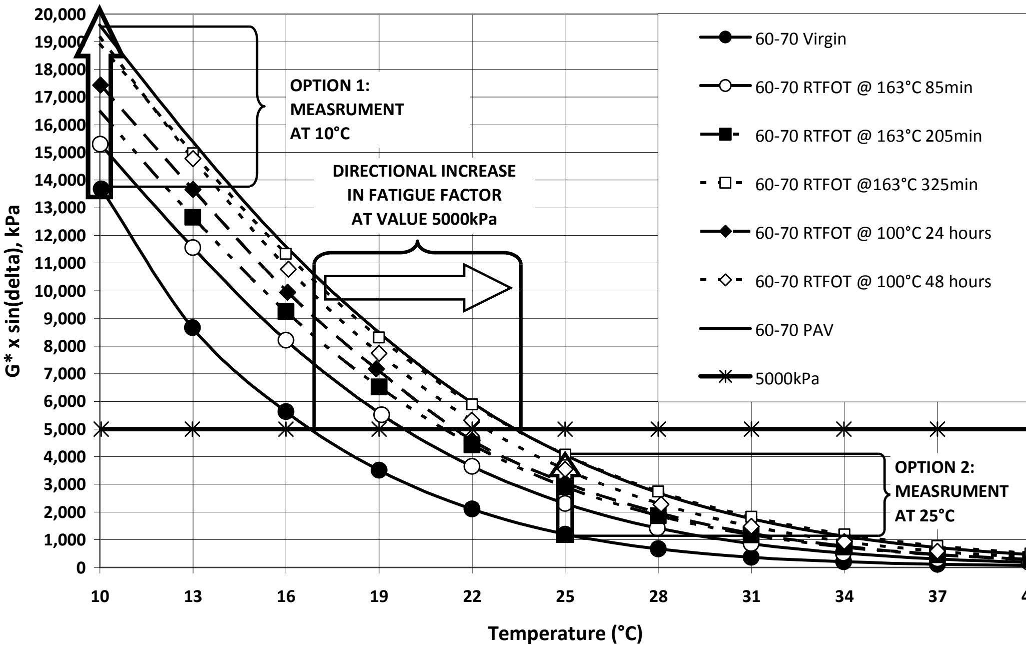

Figure 7 Comparison of the Complex Modulus (G*) between 10°C and 40°C

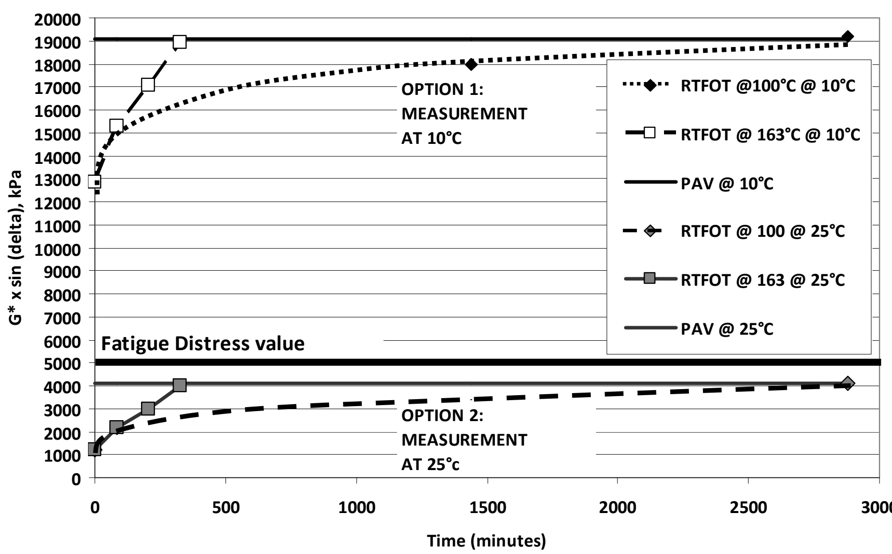

Figure 8 Accelerated Ageing measured with G* x sin (delta) at 10°C vs. 25°C (Comparing ERTFOT at 163°C, ERTFOT at 100°C vs. PAV)

At both 10°C or 25°C the rate of ageing at 163°C and 100°C show similar trends. Any one of the two measurement points (10°C or 25°C) can therefore potentially be selected to obtain a relative rate of ageing with the two extended RTFOT ageing procedures.

When the Fatigue Failure or distress value (5000kPa) from US PG system is used, the temperature gradually increases from 17°C for virgin bitumen to 23°C at PAV stiffness (Figure 7). This presents another method of comparative measurement keeping the distress value constant.

6 CONCLUSION

Despite the fact that the PAV and two accelerated RTFOT ageing procedures differ significantly with regards to the time, temperature and pressure, it is possible to achieve similar levels of stiffness with the extended RTFOT at 163°C. The ageing with the extended RTFOT at 100°C (steel rod assisted) shows a reduced ageing rate relative to the extended RTFOT at 163°C.

The trends from the results reported indicate the potential to calculate the rate of ageing using both the extended RTFOT methods. Various properties; Penetration at 25°C, Softening Point, the Fatigue Factor (obtained by means of DSR) has shown the potential to measure the relative changes in the stiffness of the residue binder properties. Viscosity at 60°C and 135°C present challenges related to the ability of the equipment to handle the increased stiffness of the aged samples and the testing temperatures are irrelevant for binder durability evaluation. The fundamental rheological properties determined by means of the DSR provide a better understanding of the affect of ageing on the binder at lower temperatures.

Each property has the potential to be used independently in order to report the changes observed during the ageing of bitumen. Similar to specifications criteria for the differences in these properties applicable to before and after RTFOT can then be employed to set ranges after 205 minute and 325 minutes for the RTFOT at 163°C. The objective is to have a RTFOT at 163°C addressing the short-term ageing (as currently indicated in SANS 307) and an extended RTFOT @ 163˚C after 205 and 325 minutes to simulate the long-term ageing (which is equivalent to the PAV ageing).

This paper provides evidence that the proposed "Extended" RTFOT ageing method can successfully be used as an alternative to the Pressurised Ageing Vessel.

It is postulated that the rate of ageing may present researchers with the potential to express the durability in terms of a rate of change in the properties which differs significantly from the current approach related to the interpretation of PAV ageing. Instead of expressing the durability in terms of a temperature where a distress stiffness level is reached, i.e. 5000kPa, it is suggested that the focus may be shifted to express the relative rate of ageing determined at one or another temperature, determined by the test property selected to measure the relative change as a result of ageing.

7 RECOMMENDATION

Ultimately the validation of the laboratory ageing with samples obtained from the field will provide the South African Roads Industry with a prediction tool that can possibly be incorporated in the South African Pavement Design Manual (SAPDM).

The potential application of the proposed extended RTFOT test method may add value as an additional norm to assist the South African Roads Industry with quality assurance and performance prediction in future. The intention is to use the proposed test method to assist practitioners with binder selection during the design and construction of a pavement. The extended RTFOT can be adopted in the SANS 307 bitumen specification in a similar manner that the PAV test is used in the Performance Graded binder classification and selection in the USA.

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KEY WORDS

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