

UNIVERSITY OF NOTTINGHAM
SCHOOL OF CIVIL ENGINEERING



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Development of the Saturation Ageing
Tensile Stiffness (SATS) Test for
High Modulus Base Materials

by

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to my dedicated family

Abstract

This thesis describes the development of a combined ageing/moisture sensitivity laboratory test to evaluate the performance of High Modulus Base (HMB) materials. The Saturation Ageing Tensile Stiffness (SATS) test consists of initial vacuum saturation of cylindrical specimens prior to placing in a high temperature (85°C) and pressure (2.1 MPa) environment in the presence of moisture for an extended period of time (65 hours). The stiffness modulus measured after the test divided by the stiffness modulus measured before the test (retained stiffness), and the specimen saturation measured again after the test (retained saturation), are used as an indication of the sensitivity of the compacted mixture to combined ageing and moisture effects. The test was used to reproduce in the laboratory the loss of stiffness modulus (60% reduction or more) and binder ageing (10 years or more) observed from the trial site where High Modulus Base (HMB) had been used.

The test protocol was developed with the specific intention to be applied on HMB materials which have relatively high air voids content and relatively low binder content. However, a trial with denser and richer mixtures (e.g. EME materials) also showed a promising result indicating that the new protocol can be applied to a wider range of materials.

A series of SATS tests using HMB materials with two different aggregate types (Acidic and Basic) and four different binders, successfully demonstrated that the protocol can distinguish more durable materials in a qualitative manner. The protocol also showed that the performance of poorly performing materials can be dramatically improved by adding certain additives, providing a potential solution to the field application of problematic materials.

For the limited range of materials covered in this study, ageing of the binder did not significantly alter the moisture sensitivity of the mixture.

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Declaration

The work described in this thesis was conducted at the University of Nottingham, School of Civil Engineering, Nottingham Centre for Pavement Engineering between February 2001 to September 2004. I declare that the work is my own and has not been submitted for a degree of another university.

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Chapter 1. Introduction

1.1 Background

Recent research has shown that deterioration in thick, well constructed flexible pavement structures (typical of UK trunk roads and motorways) is confined to the surfacing, in the form of rutting and surface-initiated cracking. If timely treatment is applied, this deterioration has been found not to significantly affect the structural integrity of the pavement, which has led to the concept of “long life” pavements [Nunn, 1997]. In parallel with this, there has been a general trend in the UK to use progressively stiffer base materials, driven by the need to minimise costly maintenance interventions, frequently in combination with one of the new generation of thin surfacing materials.

Initial road trials of High Modulus Base (referred to as **HMB**) materials containing a 15 Penetration bitumen (10/20 paving grade), known as HMB15, were undertaken in the UK at five sites [Nunn and Smith, 1997]. Results demonstrated that HMB15 behaved in a similar way to conventional base macadams, provided that appropriate mixing, laying and compaction temperatures were maintained. Due to the high stiffness modulus of this material it was estimated that cost savings of approximately 25% could be achieved compared to laying conventional Dense Bitumen Macadam (DBM).

However, on-going monitoring of an initial trial site, located at the Transport Research Laboratory (TRL), showed that the stiffness modulus had unexpectedly dropped by approximately 60% after about 8 years. Since the HMB15 base layer at this site had not been surfaced or trafficked it was thought that the drop in stiffness modulus was due to moisture and air which had been allowed to permeate into the material. As a comparison, other investigations have been performed on several in-service pavements containing HMB materials. Results showed that these pavements generally performed better than the TRL trial site, although a small number also showed an unexpected drop in stiffness modulus. Investigations on cored samples from the site also clearly demonstrated a certain level of visual deterioration, mainly

in the upper base layer. Some of the results from one such core investigation are presented in Appendix A.

Due to these concerns over long-term durability (ageing and moisture) the Highways Agency (HA) temporarily suspended the use of HMB materials containing 15 Penetration (10/20 paving grade) and 25 Penetration (20/30 paving grade) bitumens and established a research programme, led by Scott Wilson Pavement Engineering (SWPE) and the Nottingham Centre for Pavement Engineering (NCPE), to investigate the durability of long life pavements, which is thought to be the key reason for the decrease in stiffness modulus.

1.2 Objectives

The main objective of the research programme was to identify/develop a laboratory testing procedure able to reproduce the material properties observed in the HMB trial site, with a specific interest in long-term durability (to ageing and moisture sensitivity) of bituminous materials.

In long-term service conditions, it is likely that the pavement is undergoing the both aspects of ageing and moisture damage together (i.e. materials damaged by water infiltration while the binder in the material is being aged). However, from the literature review (see Chapter 2) and the questionnaire survey undertaken (see Appendix B), it became clear that the existing durability protocols are designed to investigate one aspect only (i.e. ageing test for ageing sensitivity, moisture sensitivity test for moisture damage) and are thus not ideal to reproduce the material properties from the trial site.

Therefore the prime concern was to develop/investigate a protocol which would combine the effect of both aspects (ageing and moisture damage) together in one process. More specifically, the protocol needed to be able to simulate a 60% or more reduction in mixture stiffness due to moisture damage, combined with a 10 or more years of 'long-term' binder ageing. This specific target was agreed based on the investigation on the HMB trial site, as described in the previous section. The

research was mainly focused on HMB materials with nominally identical properties to those from the HMB trial site.

As a result of this research, it is expected to present a unique and practical simple test protocol for predicting/evaluating long-term durability performance of HMB materials. The developed test protocol can be used to observe whether a severely aged binder makes the mixture particularly more susceptible to moisture damage. Eventually the protocol will be used to screen out potentially problematic materials (under the combined effects of ageing and moisture damage) and furthermore to find a way to improve it.

1.3 Scope of Research

The scope of this thesis consists of a literature review (Chapter 2) followed by seven chapters, each detailing aspects of the work undertaken, and a final chapter (Chapter 10) in which the main conclusions and recommendations for future work are presented. As the main objective of this research was to develop a testing protocol, the literature review is mainly focused on an introduction to currently available test protocols worldwide. Also a brief introduction to the basic terms used in this research is presented, providing the reader with the necessary background knowledge to this study. Chapter 3 presents several preliminary durability trials, selected from the test methods review. A representative material is manufactured and tested using the selected protocols and the results are reviewed. In Chapter 4 details of a test protocol, named SATS (Saturated Ageing Tensile Stiffness), which has been selected as the most promising approach from the previous chapter, is presented. Some preliminary trials using the SATS protocol are presented and results are reviewed. Also the key factors of the protocols are studied and several modifications are undertaken to understand the aspects observed in the chapter. Results are reviewed and a standard form of the testing protocol (referred to as 'standard SATS') is proposed. In Chapter 5 the developed testing protocol (standard SATS) is applied to a range of HMB bituminous mixtures. Details of the mixture combinations are presented and the results are analyzed. A statistical analysis is performed on a series of SATS test data (using the control mixtures) to observe the test variability. The mixture variation is also investigated including effects of certain

additives and mixture volumetric change (e.g. air voids, binder content). Chapter 6 describes a further investigation to understand the mixture behaviour and effects of key parameters in the SATS protocol. As a result, an updated version of the testing protocol (referred to as ‘modified SATS’) is proposed in Chapter 7 clearly demonstrating a better function. The durability of a limited range of bituminous mixtures is investigated using the modified protocol (modified SATS) and the results are reviewed. Chapter 8 presents a parallel investigation on binders recovered from the specimens used in the previous chapters. The Dynamic Shear Rheometer (referred to as DSR) results are presented and reviewed with the corresponding results from the SATS test series. In Chapter 9, the ‘nominal’ retained saturations presented in the previous chapters and the ‘actual’ retained saturations (considering the volumetric change during the process) are compared and the differences are reviewed. Also the ‘true’ moisture sensitivity of the material is identified by extracting ageing and pressure factors. The results are compared to the ‘nominal’ retained stiffnesses and the differences are reviewed.

Chapter 2. Literature Review

2.1 Pavement Structure

A pavement is a structure which separates the wheels of vehicles from the underlying foundation material. Pavements over soil are normally of multi-layer construction with relatively weak materials below and progressively stronger ones above [Croney and Croney, 1998]. This modern concept of pavement construction was pioneered by the Romans. As shown in Figure 2.1, the multi-layer concept used by the Romans is not very different to the typical modern flexible pavement layout shown in Figure 2.2.

Pavements can be considered to consist of three main layers, the surfacing, the base, and the foundation. In the case of asphalt pavements, the surfacing is generally divided into the surface course and the binder course which are laid separately. The base is the main structural element in the pavement. The foundation of a pavement essentially comprises two layers. The upper layer is termed the subbase and is usually formed of good quality granular material. The subbase provides a structural layer which distributes loads to the subgrade and provides a working platform for construction traffic and a compaction platform onto which bituminous materials can be laid and compacted. The lower section, the subgrade, is the natural soil or fill material which provides the surface upon which the pavement is constructed [Whiteoak, 1990]. The interface between these two layers is called the formation. Where the soil is considered to be very weak, a capping layer may also be introduced additionally between the subbase and the soil foundation [Croney and Croney, 1998].

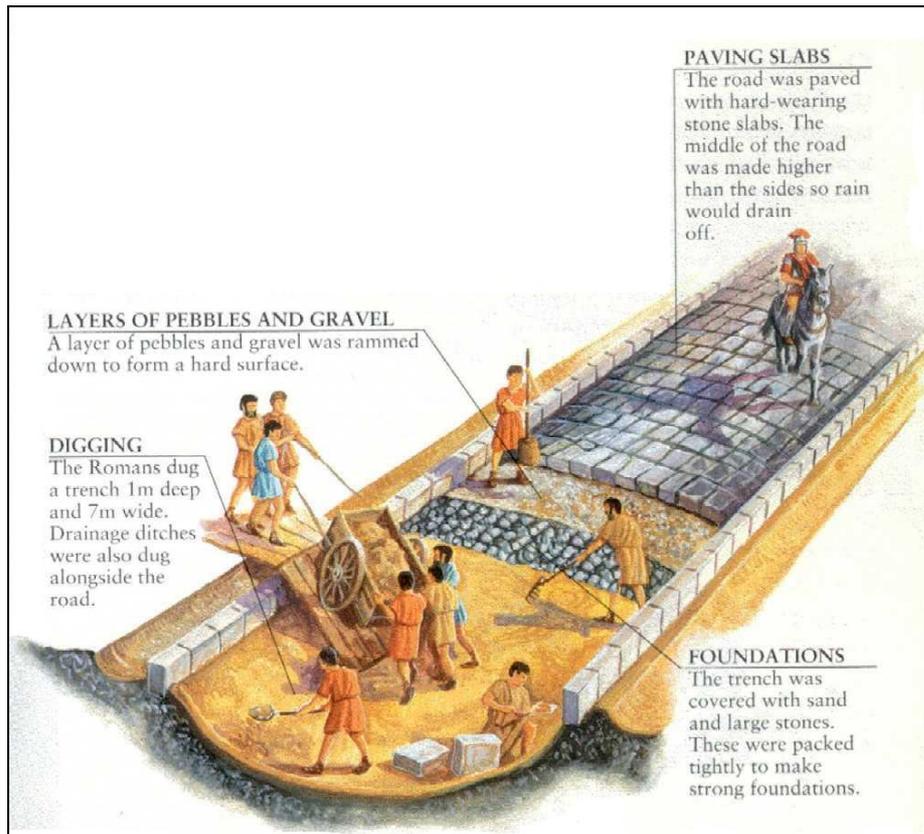


Figure 2.1 – Roman road construction [Haywood, 1994].

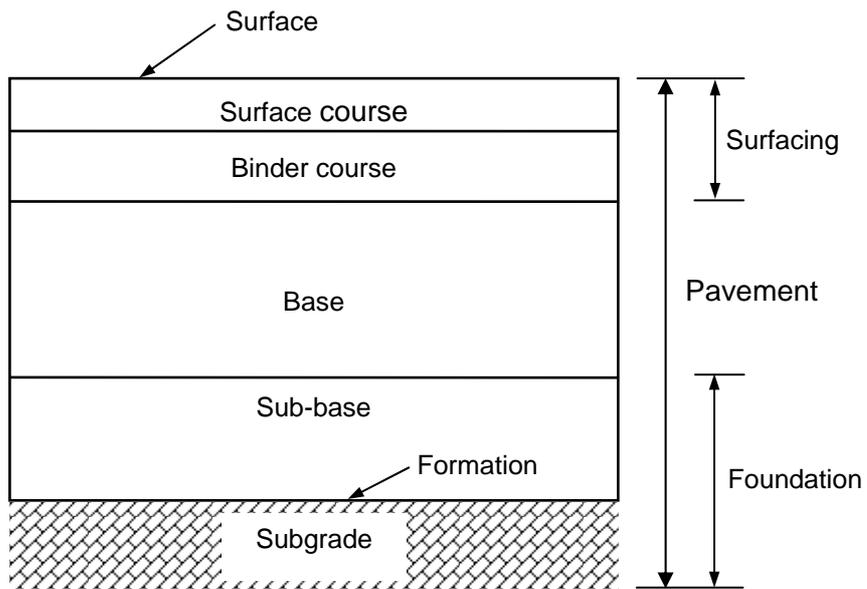


Figure 2.2 – Typical layers in a flexible pavement [Read and Whiteoak, 2003].

2.2 Introduction to Bituminous Materials

2.2.1 Bitumen

The most common asphalt binder is bitumen. The term 'bitumen' originated in Sanskrit where the words 'jatu' meaning pitch and 'jitu-krit' meaning pitch creating, referred to the pitch-producing properties of certain resinous trees. The later Latin terms of 'gwitu-men' and 'pixtu-men' (exuding pitch) became shortened into 'bitumen' when passing via French to English [Whitoak, 1990]. The origins of bitumen as an engineering material date from 3800 – 3000 B.C. when in the Eurphrates and Indus Valleys it was used as mortar for masonry and water proofing.

Bitumen is obtained from crude oil. The manufacture of bitumen involves distillation, blowing and blending. Atmospheric distillation is used to separate gas, gasoline, kerosene, gas oil and long residue (heaviest fraction consisting of a complex mixture of high molecular weight hydrocarbons). The long residue is then redistilled under vacuum at 350°C to 400°C to produce short residue, which is the feedstock used in the manufacturing of different grades of bitumen. In many cases, however, vacuum residues are processed by air ratification (blowing) to produce harder penetration grade bitumens which can then be blended to produce intermediate grades [Airey, 2000]. Additionally, the use of chemically modified binders (SBS, EVA etc) has been gaining popularity due to their improved performance [Brown et al., 1990].

2.2.2 Aggregates

Mineral aggregates may be divided into three main types as follows;

- Natural aggregate – Gravel and Sand,
- Processed aggregate – Natural aggregate crushed for size reduction,
- Synthetic aggregate – Slags, fired clays etc.

Aggregates are divided into three categories depending upon the size fraction. These are generally referred to as coarse aggregate, fine aggregate and filler. The terms

“coarse” and “fine” are not strictly defined but the material passing a 5 mm sieve and retained on the 0.075 mm sieve is normally called ‘fines’. ‘Filler’ is the material which passes 0.075 mm sieve [Airey, 2000].

The maximum aggregate size depends upon the type of mixture, the application for which it is to be used and the thickness of the pavement layer. Base materials, which are laid in thickness of 100 mm or more, may have a maximum aggregate size of 37.5 mm. Surface course mixtures may have a maximum aggregate size of 14 mm or 10 mm.

2.2.3 Mixtures

A bituminous mixture used for paving purpose is a mixture of mineral aggregates, bitumen and air. In certain case additives are used to enhance performance. The design of a bituminous mix involves the choice of aggregate type, grading, bitumen grade and bitumen content which will optimise the engineering properties in relation to the desired behaviour in service [Whiteoak, 1990].

As the range of possible mix compositions is almost infinite, only the mixtures directly related to this research are introduced.

Heavy Duty Macadam (HDM)

Continuously graded Dense Bitumen Macadam (DBM) is widely used in the United Kingdom as binder course and base materials on heavily trafficked roads. To cope with increase in traffic loading, a DBM with a 50 pen bitumen with higher binder content and increased filler contents has been developed [Leech, 1982]. This material, which is known as “Heavy Duty Macadam” (HDM) has been found to have increased resistance to permanent deformation and higher levels of stiffness modulus, as much as three times when compared with conventional dense macadams [Whiteoak, 1990]. It is reportedly a successful material for the construction and maintenance of major U.K. highways [Sewell, 1999].

High Modulus Base (HMB)

Pavement design procedures in France allow the use of a high stiffness base material known as Enrobé à Module Élevé (EME). EME is a bituminous material that uses

bitumen up to four times stiffer (15 – 50 Pen grade) than traditional binders (100 – 200 Pen grade). This was introduced in France in the early 1980's as a result of the energy concerns following the oil crisis in the mid 70's. Regulations to reduce oil product usage provided the drive to decrease the thickness of the bituminous layers. As a consequence, much stiffer materials were developed that enabled the base thickness to be reduced by up to 40%.

Recent research by the Transport Research Laboratory [Nunn and Smith, 1994] has focussed on the development and trials of equivalent UK materials. Results have shown that Heavy Duty Macadam modified with a 15 pen grade bitumen shows similar properties to EME indicating many potential advantages for conventional use, especially for heavily trafficked roads.

2.2.4 Stiffness of Bituminous Materials

Bitumens are visco-elastic materials and their deformation under stress is a function of both temperature and loading time. At high temperatures or long times of loading they behave as viscous fluids, whereas at very low temperatures or short times of loading they behave as elastic (brittle) solids. The intermediate range of temperature and loading times, more typical of conditions in service, results in visco-elastic behaviour [Whiteoak, 1990]. Hence, the simple concept of a Young's modulus having a single value for a particular material, does not apply.

Van der Poel [1954] suggested that the visco-elastic properties can be defined by a simple extension of the Young's modulus concept, provided that the two parameters (stress and strain) are assumed to have a linear relationship. He thus introduced the concept of 'stiffness modulus' as a fundamental parameter to describe the mechanical properties of bitumens. The stiffness modulus, S_b , is defined as the ratio of stress to strain for bitumen, in general, at a particular temperature and loading time.

$$\text{Stiffness } (S_b) = \frac{\text{stress}}{\text{strain}} \quad (2.1)$$

2.3 Durability of Bituminous Materials

The primary factors affecting the durability of bituminous paving mixtures (assuming they are constructed correctly) are age hardening and moisture damage. Ageing of the bituminous binder is manifested as an increase in its stiffness (or viscosity), which could lead an increase in mixture stiffness. Water damage is generally manifested as a loss of cohesion in the mixture and/or loss of adhesion between the bitumen and aggregate interface (stripping), which could lead to a decrease in mixture stiffness.

Ageing (hardening) is primarily associated with the loss of volatile components and oxidation of the bitumen during asphalt mixture construction (short-term ageing) and progressive oxidation of the in-place material in the field (long-term ageing). Other factors which also may contribute to ageing, such as steric hardening or UV radiation, are not considered in this review.

Moisture damage significantly influences the durability of bituminous mixtures. It is generally agreed that moisture can degrade the structural integrity of bitumen-aggregate mixtures through loss of cohesion or through failure of the adhesion between the bitumen and aggregate [Kennedy, 1985; Terrel and Al-Swailmi, 1994]. Reduction of cohesion results in a reduction of the strength and stiffness of the mixture and thus a reduction of the pavement's ability to support traffic-induced stresses and strains. Failure of the bond between the bitumen and aggregate (stripping) also results in a reduction in pavement support. Both mechanisms of moisture damage result in a weaker pavement layer and one which is prone to deform, and/or crack, under traffic loading. In addition, stripping can result in the loss of material and eventually the total deterioration of the asphalt mixture.

As the main objective of the research is to develop a testing protocol, the following sections describe a critical review of existing test methods, protocols and techniques for the assessment of ageing and moisture sensitivity of bituminous materials.

2.4 Ageing Tests

Tests related to ageing of bituminous materials can be broadly divided into two categories:

- Tests performed on bituminous binder; and
- Tests performed on bituminous (asphalt) mixtures.

Much of the research into the ageing of bitumen utilises the thin film oven technique to age the bitumen in an accelerated manner (e.g. thin film oven test, rolling thin film oven test, rolling microfilm oven test, tilt-oven durability test). Typically, these tests are used to simulate the relative hardening that occurs during the mixing and laying process (i.e. short-term ageing). To include long-term hardening in the field, thin film oven ageing is typically combined with pressure oxidative ageing [Airey, 2002].

2.5 Ageing Test for Bituminous Binder

Numerous attempts have been made by researchers over the last seventy years to correlate accelerated laboratory ageing of bitumen with field performance. Most of this research has used thin film oven tests to age the bitumen in an accelerated manner, with most of the thin film oven ageing methods relying on extended heating (oven volatilisation) procedures. The ageing tests are summarised in Table 2.1.

2.5.1 Extended Heating Procedures

Extended heating procedures tend to be used to simulate short-term ageing (hardening) of bitumen associated with asphalt mixture preparation activities. The most commonly used standardised tests, to control the short-term ageing of conventional, unmodified bitumen, are the thin film oven test (TFOT), the rolling thin film oven test (RTFOT) and the rotating flask test (RFT).

Thin Film Oven Test

The concept of the 'thin film oven test' as a means of accelerating the weathering process of bitumen was first introduced by Benson in 1937 [Lewis and Welborn, 1940]. In this early work, the film of bitumen was spread on a glass microscope

slide (25.4 mm × 76.2 mm) by means of a special gauge to give a film thickness of approximately 25.4 μm. The coated glass slide was placed in an oven (163°C) for 5 hours, and then visually observed under the microscope.

Table 2.1 – Bitumen ageing methods.

| Test Method | Temp | Duration | Sample Size | Film Thickness | Extra Features |
|--|-----------------|------------------|--------------------------|----------------|-----------------------------------|
| Thin film oven test (TFOT) – ASTM D1754, EN 12607-2 | 163°C | 5 hrs | 50 g | 3.2 mm | - |
| Modified thin film oven test (MTFOT) | 163°C | 24 hrs | - | 100 μm | - |
| Rolling thin film oven test (RTFOT) – AASHTO T240, ASTM D2872, EN12607-1 | 163°C | 75 min | 35 g | 1.25 mm | Air flow – 4000 ml/min |
| Extended rolling thin film oven test (ERTFOT) | 163°C | 8 hrs | 35 g | 1.25 mm | Air flow – 4000 ml/min |
| Nitrogen rolling thin film oven test (NRTFOT) | 163°C | 75 min | 35 g | 1.25 mm | N ₂ flow – 4000 ml/min |
| Rotating Flask Test (RFT) – DIN 52016, EN12607-3 | 165°C | 150 min | 100 g | - | Flask rotation – 20 rpm |
| Shell microfilm test | 107°C | 2 hrs | - | 5 μm | - |
| Modified Shell microfilm test (1963) | 99°C | 24 hrs | - | 20 μm | - |
| Modified Shell microfilm test (1961) | 107°C | 2 hrs | - | 15 μm | - |
| Rolling microfilm oven test (RMFOT) | 99°C | 24 hrs | 0.5 g | 20 μm | Benzene solvent |
| Modified RMFOT | 99°C | 48 hrs | 0.5 g | 20 μm | 1.04 mm φ opening |
| Tilt-oven durability test (TODT) | 113°C | 168 hrs | 35 g | 1.25 mm | - |
| Alternative TODT | 115°C | 100 hrs | 35 g | 1.25 mm | - |
| Thin film accelerated ageing test (TFAAT) | 130°C/ 113°C | 24 hrs 72 hrs | 4 g | 160 μm | 3 mm φ opening |
| Modified rolling thin film oven test (RTFOTM) | 163°C | 75 min | 35 g | 1.25 mm | Steel rods |
| Iowa durability test (IDT) | 65°C | 1000 hrs | TFOT residue – 50g | 3.2 mm | 2.07 MPa – pure oxygen |
| Pressure oxidation bomb (POB) | 65°C | 96 hrs | ERTFOT residue | 30 μm | 2.07 MPa – pure oxygen |
| Accelerated ageing test device / Rotating cylinder ageing test (RCAT) | 70°C ~ 110°C | 144 hrs | 500g | 2 mm | 4 to 5 l/hr – pure oxygen |
| Pressure ageing vessel (PAV) | 90°C ~ 110°C | 20 hrs | RTFOT/TFOT residue – 50g | 3.2 mm | 2.07 MPa – air |
| High pressure ageing test (HiPAT) | 85°C | 65 hrs | RTFOT residue – 50g | 3.2 mm | 2.07 MPa – air |

Since then a number of attempts have been made to develop a better test. Lewis and Welborn [1941] studied various methods and, after extensive testing of representative bitumens, proposed an oven heat test (TFOT). In this test, a 50 ml sample of bitumen was placed in a 140 mm diameter flat container resulting in a film thickness of 3.2 mm. The container was then heated in an oven for 5 hours at 163°C.

The TFOT was adopted by AASHTO in 1959 and ASTM in 1969 [Bell et al., 1994], as a mean of evaluating the hardening of bitumen during plant mixing. However, a major concern of the TFOT is the thick binder film. As the bitumen is not agitated or rotated during the test, there is a concern that ageing (primarily volatile loss) may be limited to the 'skin' of the bitumen sample.

This concern over the testing of bitumen in relatively thick films meant that there was a move, from the 1950s, to develop or modify ageing tests to age and test bitumen in microfilm thicknesses. One such example is the modified thin film oven test (MTFOT), used by Edler et al. [1985], where the binder film was reduced from 3.2 mm to 100 μm with an additional increased exposure time of 24 hours. This minor modification of the TFOT was done in order to increase the severity of the ageing process to include oxidative hardening of the binder as well as volatile loss.

Rolling Thin Film Oven Test

The RTFOT is probably the most significant modification of the TFOT involving the placing of bitumen in a glass jar (bottle) and rotating it in thinner films of bitumen than that of TFOT. The RTFOT therefore simulates far better the hardening which bitumen undergoes during asphalt mixing [Hveem et al., 1963; Shell Bitumen Review, 1973].

The RTFOT was developed by the California Division of Highways and involves rotating eight glass bottles each containing 35 g of bitumen in a vertically rotating shelf, while blowing hot air into each sample bottle at its lowest travel position [Hveem et al., 1963]. During the test, the bitumen flows continuously around the inner surface of each container in relatively thin films of 1.25 mm at a temperature of 163°C for 75 minutes. The vertical circular carriage rotates at a rate of 15 rpm and the air flow is set at a rate of 4000 ml/minute. The method ensures that all the bitumen is exposed to heat and air and the continuous movement ensures that no skin develops to protect the bitumen. The conditions in the test are not identical to those found in practice but experience has shown that the amount of hardening in the RTFOT correlates reasonably well with that observed in a conventional batch mixer [Whiteoak, 1990].

Several modifications have also been made to the RTFOT, although most of them have been relatively minor. For example Edler et al. [1985] used an extended time period of 8 hours rather than 75 minutes in their extended rolling thin film oven test (ERTFOT), while Kemp and Predoehl [1981] used 5 hours. A more recent modification is the development of a nitrogen rolling thin film oven test (NRTFOT) to determine more accurately the actual loss of volatiles, by minimising oxidative hardening, during the process [Parmeggiani, 2000]. The procedure is identical to the standard test except that nitrogen is blown over the exposed surface of the bitumen samples.

A similar application of the RTFOT with nitrogen gas is the rapid recovery test (RRT) used to obtain a quantity of 'recovered binder' from modified or unmodified cutback or emulsion binders [MCHW, 1998]. The procedure uses a temperature of 85°C with the RTFOT to evaporate water and/or the light solvent or highly volatile fraction of emulsions or cutback binders. Nitrogen gas is used instead of air to minimise oxidative hardening effects.

Rotating Flask Test (DIN 52016)

The RFT method consists of ageing a 100 g sample of bitumen in the flask of a rotary evaporator for a period of 150 minutes at a temperature of 165°C. As the flask is rotated at 20 rpm, the material forming the surface of the specimen is constantly replaced thus preventing the formation of a skin on the surface of the bitumen [Airey, 2002].

Shell Microfilm Test

The Shell microfilm test is another variation of the principal used with the TFOT. In this test a very thin, 5 μm , film of bitumen is aged for 2 hours on a glass plate at 107°C [Griffin et al., 1955]. The thinner film thickness was chosen to simulate the film thicknesses that exist in asphalt mixtures. The bitumen is evaluated on the basis of viscosity before and after testing to provide the 'Shell ageing index'. However, there is limited reported correlation between field performance and laboratory ageing using the Shell microfilm test [Leslie, 1979], except for the work done on the Zaca-Wigmore test roads [Zube and Skog, 1969]. Simpson et al. [1959] compared the

viscosity data for bitumen recovered from the two test roads with the Shell microfilm test and found a definite correlation between field and laboratory data.

The Shell microfilm test was modified slightly by Hveem et al. [1963] by increasing the film thickness to 20 μm and the exposure time to 24 hours with a slightly reduced temperature of 99°C. They also used the residue from RTFOT to prepare the glass plates (50 mm \times 50 mm \times 6 mm). These alterations demonstrated an indirect relationship between field and laboratory hardening. Additionally, slight variations were made by Traxler [1963] who increased the binder film thickness to 15 μm .

Rolling Microfilm Oven Test

The rolling microfilm oven test (RMFOT) is a modification of the RTFOT in order to obtain much thinner films of bitumen for ageing [Schmidt and Santucci, 1969]. The test consists of dissolving bitumen in benzene (solvent), coating the inside of the RTFOT bottles with this solution and then allowing the benzene to evaporate. The result of this process is the creation of a 20 μm film of bitumen which is then aged at 99°C for 24 hours.

The RMFOT was modified by Schmidt [1973] in order to reduce the amount of volatile loss during ageing. This was accomplished by placing a capillary in the opening of the RTFOT bottle and calibrating the capillary size to match the volatile loss from the bottle to that achieved during the ageing of asphalt mixture specimens at 60°C. A 1.04 mm diameter opening was selected and in addition the ageing time was increased from 24 to 48 hours. The modified RMFOT was found to have good correlation with field cracking of the Zaca-Wigmore pavements as well as with other field and laboratory aged asphalt mixtures. The notable disadvantage of the test is that only a very small amount of aged bitumen (0.5 g per bottle) could be produced for subsequent binder testing.

Tilt-Oven Durability Test

An additional modification of the RTFOT is the California tilt-oven durability test (TODT) where the oven is tilted 1.06° higher at the front to prevent bitumen

migrating from the bottles [Kemp and Predoehl, 1981]. In addition the TODT uses a lower temperature and longer time for ageing compared to the RTFOT, namely 168 hours at 113°C. This level of ageing approximates to that found for pavement mixtures after 2 years in hot desert climates. In addition, Kemp and Predoehl [1981] aged laboratory produced specimens in four distinct climates in the field and concluded that the test results from TODT could be used in a hot climate specification to control asphalt hardening. McHattie [1983] also performed a slightly modified TODT with test conditions of 100 hours at 115°C.

Thin Film Accelerated Ageing Test

A modification of the RMFOT is the thin film accelerated ageing test (TFAAT), developed by Petersen [1989], which has the advantage of producing an increased amount of aged binder as it uses a sample size of 4 g of binder compared to the 0.5 g of the RMFOT. Whereas extended heating tests, such as the TFOT and RTFOT, reflect only the ageing (mainly volatile loss) that occurs during hot-plant mixing, the TFAAT was developed to produce a representative level of volatilisation and oxidation to simulate the level of oxidative age hardening typically found for extended pavement ageing.

The TFAAT was developed to complement a column oxidation procedure developed by Davis and Petersen [1966] where a 15 μm thick bitumen film, coated on Teflon particles, was oxidised in a gas chromatographic column at 130°C for 24 hours by passing air through the column. As the TFAAT uses eight times more binder than the RMFOT, with subsequent increased binder films, the TFAAT either has to have longer ageing times or higher test temperatures to achieve the same degree of oxidative ageing as that found for the RMFOT. Petersen [1989] found that performing the test at 130°C for 24 hours produced the same degree of oxidative ageing found for the RMFOT as well as for 11 to 13 year old pavements. As with the RMFOT, the 31 mm diameter opening for the standard RTFOT bottle was reduced to 3 mm to restrict excessive volatile loss. The TFAAT can also be performed at the lower temperature of 113°C but for a longer period of 3 days compared to the one day test at 130°C.

Modified Rolling Thin Film Oven Test

One of the main problems with using the RTFOT for modified bitumens is that these binders, because of their high viscosity, will not roll inside the glass bottles during the test. In addition, some binders have a tendency to roll out of the bottles. To overcome these problems, Bahia et al. [1998] developed the Modified Rolling Thin Film Oven Test (MRTFOT).

The test is identical to the standard RTFOT except that a set of 127 mm long by 6.4 mm diameter steel rods are positioned inside the glass bottles during oven ageing. The principle is that the steel rods create shearing forces to spread the binder into thin films, thereby overcoming the problem of ageing high viscosity binders. Initial trials of the MRTFOT indicate that the rods do not have any significant effect on the ageing of conventional penetration grade bitumens [Bahia et al., 1998]. However, recent work at the Turner-Fairbanks research centre has indicated that using the metal rods in the MRTFOT does not solve the problem of roll-out of modified binder and further validation work is required before the technique can be accepted [Airey, 2002].

The rapid recovery test (RRT) uses a similar mechanism to MRTFOT to prevent the roll-out of emulsions or cutback binders but, instead of plain steel rods, the procedure uses 120 mm long by 12.2 mm diameter stainless steel or PTFE screws [MCHW, 1998]. The direction of the screw is such that the sample is drawn to the rear of the bottle during rotation in the RTFOT carousel. Oliver and Tredrea [1997] also used a roller with a screw thread to age PMBs in the RTFOT where, as the bottle rotated, the roller 'screwed' the binder towards the back wall of the bottle. Using their modified RTFOT with an exposure time of 9 hours and a temperature of 163°C, Oliver and Tredrea [1997] were able to produce similar changes in the rheological properties of polymer modified and unmodified bituminous binders to those found after 2.5 years exposure in a sprayed seal in a hot climate.

2.5.2 Oxidative (Air Blowing) Procedures

Although thin film oven tests can adequately measure the relative hardening characteristics of bitumens during the mixing process they generally fall short of

accurately predicting long-term field ageing. Attempts have been made to overcome this by combining thin film oven ageing with oxidative ageing.

Iowa Durability Test

The Iowa Durability Test (IDT) is one such test that combines thin film ageing with oxidative ageing [Lee, 1973]. The test consists of ageing binder residue from a standard TFOT in a pressure vessel at 2.07 MPa using pure oxygen at a temperature of 65°C for up to 1000 hours. As the residue binder from the TFOT is not transferred from its container, the film thickness during the pressure-oxidation treatment is still 3.2 mm. Lee [1973] found that ageing bitumen using the IDT produced a similar correlation to that found from binders aged in the field over a five year period. Based on this correlation and a considerable amount of field and laboratory data, Lee concluded that 46 hours of ageing with the IDT is equivalent to 5 years field ageing in Iowa conditions.

Pressure Oxidation Bomb

Edler et al. [1985] used a similar approach to that used by Lee, where residue from the ERTFOT was followed by oxidation under pressure using the pressure oxidation bomb (POB). The POB consists of a cylindrical pressure vessel fitted with a screw-on cover containing a safety blow-off cap, pressure gauge and stopcock. The vessel houses a metal support where twelve 40 mm by 40 mm glass plates coated with 30 μm bitumen films are positioned horizontally. The test consists of ageing the bitumen residue at a pressure of 2.07 MPa at 65°C for 96 hours.

Accelerated Ageing Test Device / Rotating Cylinder Ageing Test

Similar in concept to the RTFOT is the Accelerated Ageing Test Device, also known as the Rotating Cylinder Ageing Test (RCAT), developed at the Belgian Road Research Centre (BRRC) [Verhasselt, 2000]. Although standard tests such as the RTFOT and RFT can adequately simulate construction ageing, their high temperatures make them unsuitable for simulating field ageing. This has led to the development of the accelerated ageing device which has been based on a theoretical kinetic approach to ageing.

The device consists of a fairly large cylinder (tube), with an internal diameter of 124 mm and a length of 300 mm, which is capped at both ends but with a central aperture of diameter 43 mm at one end, where bitumen can be introduced (see Figure 2.3). After charging the cylinder with up to 500 g of bitumen, a stainless steel roller, 296 mm in length and 34 mm in diameter, is placed into the cylinder. The cylinder is then placed in a frame which rotates the cylinder at 1 rpm and flows oxygen through the aperture at a rate of 4 to 5 litres per hour (≈ 75 ml/min). Rotation of the roller within the cylinder distributes the bitumen into an even 2 mm thick film on the inner wall of the cylinder. Tests are conducted at temperatures between 70°C and 110°C. At discrete intervals, approximately 20 g to 25 g of bitumen is removed from the cylinder for subsequent testing. Due to the large initial quantity of bitumen, the procedure allows numerous evaluations to be made and progressive changes in the bitumen chemistry and physical properties to be investigated.

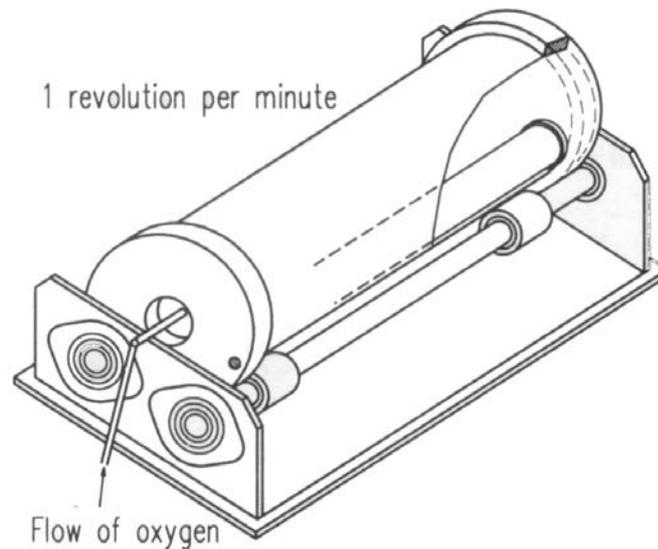


Figure 2.3 – Rotating Cylinder Ageing Test (RCAT).

Using the RCAT, Choquet [1993] found that ageing bitumen at 85°C for 144 hours reflects field ageing with regard to the formation of asphaltenes. He also noted that temperatures less than 100°C were essential in accelerated ageing tests in order to produce chemical and rheological changes similar to those found in the field. Verhasselt [1997] also found mutual agreement between in-service ageing in the

field and laboratory ageing using the RCAT for dense mixtures. However Francken et al. [1997] found that longer ageing times than 240 hours were required to simulate field ageing (12 years) of porous mixtures.

Pressure Ageing Vessel

The SHRP-A-002A research team developed a method using the pressure ageing vessel (PAV) to simulate the long-term, in-service oxidative ageing of bitumen in the field [Kennedy and Harrigan, 1990]. The method involves hardening of bitumen in the RTFOT followed by an additional oxidation of the residue in the PAV. Although much of the work done during the SHRP project was with the TFOT, such as the work done by Christensen and Anderson [1992], the RTFOT was eventually selected as the recommended procedure [Petersen et al., 1994]. The PAV procedure entails ageing 50 g of bitumen in a 140 mm diameter pan (to have 3.2 mm of binder film thickness) in the heated vessel (see Figure 2.4), pressurised with air to 2.07 MPa for 20 hours at temperatures between 90 and 110°C.

Migliori and Corte [1999] investigated the possibility of simulating RTFOT (short-term ageing) and RTFOT + PAV (long-term ageing) simply by means of PAV testing for unmodified penetration grade bitumens. They found that 5 hours of PAV ageing at 100°C and 2.07 MPa was equivalent to standard RTFOT ageing, and that 25 hours of PAV ageing at 100°C and 2.07 MPa was equivalent to standard RTFOT + PAV ageing.

High Pressure Ageing Test

The High Pressure Ageing Test (HiPAT) is a modification of the PAV procedure using a relatively lower temperature of 85°C and a longer duration of 65 hours [Hayton et al., 1999]. The reason for these modifications was the concern that the temperatures used in the PAV procedure were unrealistically high compared to expected pavement temperatures. In addition it was felt, particularly for modified binders, that the PAV procedure was liable to significantly alter the binders to an unrepresentative extent compared to that found in the field.

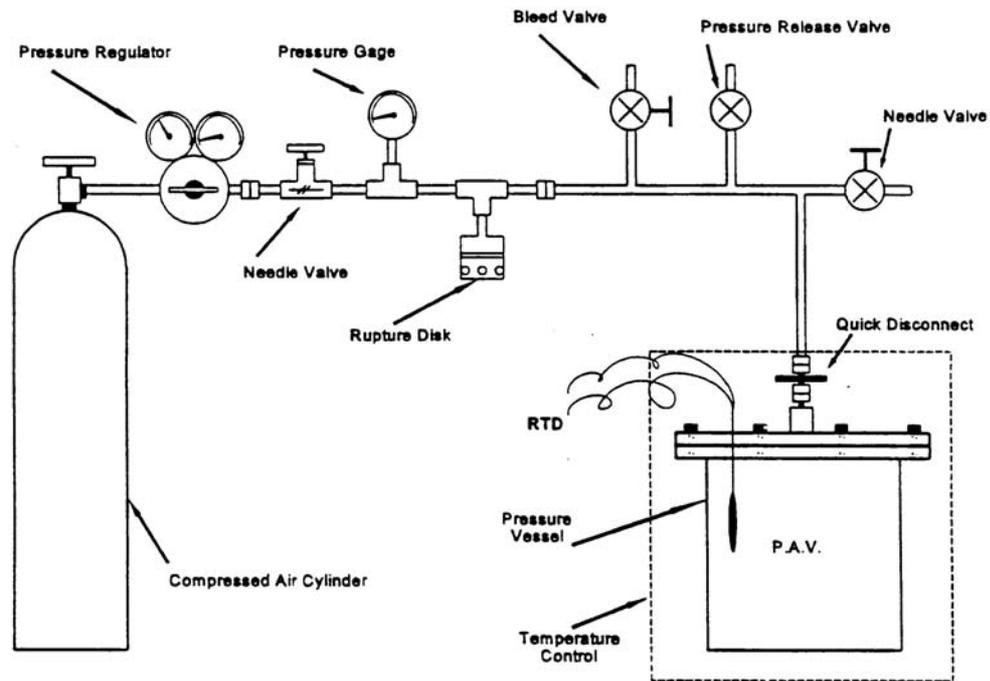


Figure 2.4 – Pressure Ageing Vessel (PAV).

Initial studies to predict long-term ageing in the field have suggested that the HiPAT process may be more severe than the natural ageing process for a dense asphalt mixture, such as Hot Rolled Asphalt (HRA), with a 10 year service life [Hayton et al., 1999]. However, the severity of this procedure can be useful for an extreme case study.

2.6 Ageing Tests for Bituminous Mixtures

In addition to artificially ageing binders, a number of methods also exist for artificially ageing the asphalt mixture. The basic procedure is to artificially age the mixture and then assess the effect of ageing on key material parameters (e.g. stiffness, viscosity, strength etc). Extended heating procedures typically expose the mixture to high temperatures for a specified period(s) of time before suitable testing (e.g. compressive testing, tests on recovered binder, etc). Oxidation tests typically utilise a combination of high temperature and pressure oxidation to age specimens.

Most of the initial studies on asphalt mixture ageing involved tests on the recovered binder as detailed by Hubbard and Gollomb [1937] and Shattuck [1940]. Understandably, the breakthroughs in the investigation of the properties of asphalt binder after plant mixing and service in pavements was the Abson recovery method developed in 1933 [Abson, 1933]. Since the development of the Abson recovery method, numerous studies have determined the properties of asphalt binder after plant mixing and pavement ageing [Leslie, 1979].

A large percentage of the initial laboratory ageing procedures used Ottawa sand as a standard ‘aggregate’ with the tests being done with ultraviolet light as well as extended exposure to heat and air [Lang and Thomas, 1939]. A list of asphalt mixture ageing tests is presented in Table 2.2.

Table 2.2 – Asphalt mixture ageing methods

| Test Method | Temp. | Duration | Sample Size | Extra Features |
|--|--------------|---------------------|---|------------------------|
| Production ageing [Von Quintas et al., 1991] | 135°C | 8, 16, 24, 36 hrs | Loose material | - |
| SHRP short-term oven Ageing (STOA) | 135°C | 4 hrs | Loose material | - |
| Bitutest protocol – short term ageing | 135°C | 2 hrs | Loose material | - |
| Ottawa sand mixtures (1952) | 163°C | Various periods | 50 mm x ϕ 50 mm Compacted specimens | - |
| Plancher method (1976) | 150°C | 5 hrs | 25 mm x ϕ 40 mm Compacted specimens | - |
| Ottawa sand mixtures (1981) | 60°C | 1200 hrs | - | - |
| Hugo and Kennedy [1985] | 100°C | 4 or 7 days | Compacted slabs | 80% relative humidity |
| Long-term ageing [Von Quintas et al., 1991] | 60°C + 107°C | 2 days + 5 days | Compacted specimens | - |
| SHRP long-term oven Ageing (LTOA) | 85°C | 5 days | Compacted specimens | - |
| Bitutest protocol – long term ageing | 85°C | 5 days | Compacted specimens | - |
| Kumar and Goetz [1977] | 60°C | 1, 2, 4, 6, 10 days | Compacted specimens | Air at 0.5 mm of water |
| Long-term ageing [Von Quintas et al., 1991] | 60°C | 5, 10 days | Compacted specimens | 0.7 MPa – air |
| Oregon mixtures (1986) | 60°C | 0, 1, 2, 3, 5 days | Compacted specimens | 0.7 MPa – air |
| SHRP low pressure oxidation (LPO) | 60°C or 85°C | 5 days | Compacted specimens | Oxygen – 1.9 l/min |
| Khalid and Walsh [2001] | 60°C | Up to 25 days | Compacted specimens | Air – 3 l/min |
| PAV on mixtures | 100°C | 72 hrs | Compacted specimens | 2.07 MPa – air |

2.6.1 Extended Heating Procedures

Pauls and Welborn [1952] exposed 50 mm by 50 mm cylindrical specimens of an Ottawa sand mixture to 163°C (TFOT and RTFOT ageing temperature) for various time periods. The compressive strength of the cylinders, as well as the consistency of the recovered binder, was compared to that of the original (unaged) material. Results from this study indicated that bitumen recovered from laboratory aged specimens or aged in the TFOT could be used to assess the short-term hardening properties of bitumens.

Plancher et al. [1976] used a similar oven ageing procedure to age 25 mm thick by 40 mm diameter specimens at 150°C for 5 hours. Before and after this accelerated ageing process, the resilient modulus of the samples was measured at 25°C and the ratio was used as the ageing index.

Hugo and Kennedy [1985] oven aged laboratory compacted slabs at 100°C for 4 or 7 days under either dry atmosphere or 80% relative humidity conditions. After completion of the ageing process, bitumen was recovered from 10 mm slices off the top of the slabs. The viscosity of the bitumen before and after the ageing process was measured and the ratio was used as the ageing index. In addition, the weight loss of the samples after the ageing process was used to indicate volatile loss.

Most of the methods used for laboratory ageing of asphalt mixtures involve the ageing of compacted asphalt mixture specimens. However, Von Quintas et al. [1991] investigated the use of force draft oven ageing to simulate short-term 'production' hardening on loose mixture samples. In this method, loose asphalt material was heated at 135°C in a force draft oven for periods of 8, 16, 24 and 36 hours. Although this method showed similar levels of ageing to those found in the field, there was considerable scatter in the laboratory data [Bell, 1989].

The SHRP short-term oven ageing (STOA) procedure was developed under the SHRP-A-003A project, based on the work done by Von Quintas et al. [1991]. The procedure requires loose mixtures, prior to compaction, to be aged in a forced draft oven for 4 hours at 135°C. The process was found to represent the ageing occurring

during mixing and placing and also represents pavements of age less than 2 years [Bell et al., 1994; Monismith et al., 1994].

Scholz [1995] developed a similar short-term ageing procedure to simulate the amount of hardening which occurs during the construction process for continuously graded (DBM) and gap graded (HRA) mixtures. The procedure is similar to the SHRP STOA procedure except that the conditioning temperature is either 135°C or the desired compaction temperature, whichever is higher, and the conditioning period is 2 hours for DBM. However it is also suggested that no conditioning period is required for HRA mixtures [Brown and Scholz, 2000].

Von Quintas et al. [1991] also investigated long-term ageing using a force draft oven where compacted asphalt mixture specimens were aged for 2 days at 60°C followed by an additional 5 days at 107°C. However, Bell [1989] commented that the elevated temperature level used in the test may cause specimen disruption (structural loosening) in some cases, particularly for high penetration grade asphalt mixtures (softer mixtures).

The SHRP long-term oven ageing (LTOA) procedure was also developed under the SHRP-A-003A project. After STOA, the loose material is compacted and placed in a forced draft oven at 85°C for 5 days [Harrigan et al., 1994]. The parameters used for LTOA are meant to represent 7 to 10 years of service. Further field validation [Bell et al., 1994; Monismith et al., 1994] also suggested the following correlation between the protocol and field ageing.

- 2 days at 85°C – 2 to 6 years for both Dry-Freeze and Wet-No-Freeze;
- 4 days at 85°C – 7 years for Dry-Freeze and 15 years for Wet-No-Freeze;
- 8 days at 85°C – over 9 years for Dry-Freeze and over 18 years for Wet-No-Freeze climate.

In association with his short-term procedure, Scholz [1995] developed a long-term oven ageing procedure for the compacted asphalt mixture specimens. The procedure is identical to the SHRP LTOA procedure consisting of forced draft oven ageing of

compacted specimens (using short-term aged mixture) at 85°C for 5 days [Brown and Scholz, 2000].

2.6.2 Oxidative (Air Blowing) Procedures

In addition to oven ageing of loose material and compacted specimens, Von Quintas et al. [1991] also used a pressure oxidation treatment. The procedure consisted of conditioning compacted specimens at 60°C at a pressure of 0.7 MPa for 5 or 10 days.

Kim et al. [1986] used a similar pressure oxidation treatment on compacted specimens of Oregon mixtures. Samples were subjected to oxygen at 60°C and 0.7 MPa for 0, 1, 2, 3 and 5 days. The effects of ageing were evaluated by indirect tensile stiffness and indirect tensile fatigue. Although the stiffness results generally increased with ageing, some mixtures showed an initial decrease in stiffness in the early part of the ageing procedure. This was attributed to a loss of cohesion in the samples (structural loosening) at the temperature of 60°C used in the ageing test. A similar phenomenon was observed by Von Quintas et al. [1991] and therefore a certain level of confinement to the specimen may be applied to keep its structural integrity as intact as possible during the process.

Another long-term ageing procedure that was developed under the SHRP-A-003A programme was a Low Pressure Oxidisation (LPO) procedure, carried out on compacted specimens after they had been short-term aged. The procedure consists of passing oxygen through a confined triaxial specimen at 1.9 l/min at either 60°C or 85°C for a period of 5 days [Airey, 2002].

Khalid and Walsh [2001] developed a LPO test for accelerated ageing of porous asphalt. The system consists of feeding compressed air, at a flow rate of 3 l/min, through a series of heat exchange coils placed in an oven, and then through a number of porous asphalt samples. A test temperature of 60°C was used and a rubber membrane was fitted over the samples to ensure that air flowed through the samples instead of around their periphery (see Figure 2.5). The technique has been shown to

recreate the ageing effect produced by the SHRP LTOA procedure, although due to its lower temperature, longer ageing times are required [Khalid and Walsh, 2000].

Korsgaard et al. [1996] used the PAV to age gyratory compacted dense asphalt mixture specimens. The binder of the aged specimens was then recovered and properties were compared to the standard PAV aged binder. They found an ageing condition of 72 hours at 2.07 MPa and 100°C was comparable to the standard PAV binder ageing process, but conceded that 60 hours may be more appropriate for more porous mixtures.

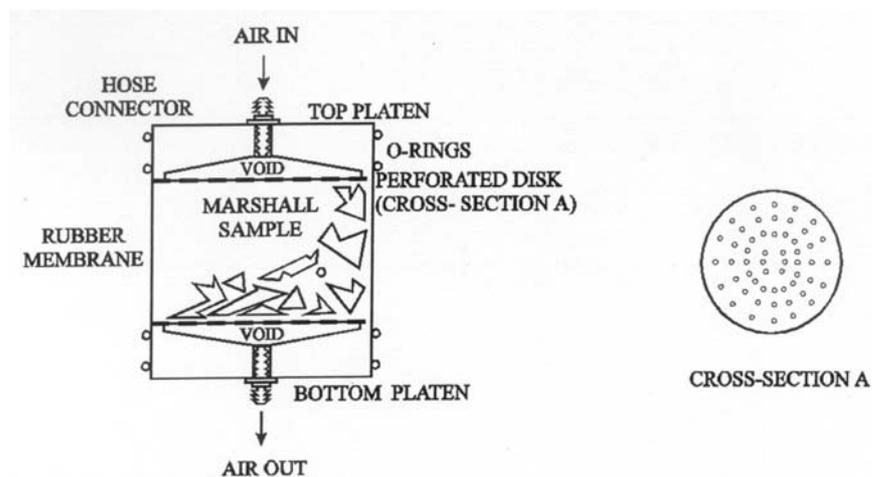


Figure 2.5 – LPO technique for porous asphalt [Khalid and Walsh, 2001]

2.7 Moisture Sensitivity Tests

The development of tests to determine the moisture sensitivity of asphalt mixtures began in the 1930s [Terrel and Shute, 1989]. Since then numerous tests have been developed in an attempt to identify the susceptibility of asphalt mixtures to moisture damage. These moisture sensitivity tests can generally be divided into two categories:

- Tests conducted on loose coated aggregate; and
- Tests conducted on compacted mixtures.

Test methods in the first category generally involve immersing the uncompacted loose mixture in water (or a chemical solution) either at room temperature or an elevated temperature for a specific period of time, and assessing the separation of the bitumen binder from the aggregate (stripping) by visual inspection.

Tests on compacted mixtures generally use either samples prepared in the laboratory or cored from in-service pavements. Typically, the samples are conditioned in water to simulate in-service conditions and assessment is made by a ratio of conditioned to unconditioned mechanical properties of the material (i.e. strength or stiffness). Immersion trafficking tests which incorporate traffic loading during a moisture conditioning process are not considered in this review.

2.7.1 Tests on Loose Mixtures

Several methods have been developed to assess the amount of bitumen loss which occurs as a result of uncompacted, coated aggregate being immersed in water. However, for the majority of these tests little information is available to correlate test data with field performance. The various methods differ in the type of specimen used, the way in which the specimen is immersed in water and the manner in which stripping (adhesion loss) is assessed. A list of stripping tests for loose aggregates is presented in Table 2.3.

Static Immersion Tests

The static immersion test [AASHTO T182, ASTM D1664] involves coating 100 g of aggregate with bitumen, immersing it for 16 to 18 hours in 400 ml of distilled water (pH 6 to 7), then visually estimating the total visible area of the coated aggregate as above or below 95%. The visual assessment is made while the mixture is still immersed in the water. Although the method may indicate mixtures showing some degree of moisture sensitivity, it is doubtful that the long-term potential of stripping is considered [Terrel and Shute, 1989]. The static immersion test was also discontinued as an ASTM standard in 1993 [Airey, 2002].

Another example of a static immersion test is the total water immersion test ('twit' test) [Whiteoak, 1990]. The test involves coating 14 mm single size aggregate with a

known quantity of bitumen and then soaking the coated aggregate in distilled water at 25°C for 48 hours. The percentage of bitumen stripped off the aggregate is then visually assessed. However, as with the ASTM method, indication of stripping in the laboratory does not always relate to poor field performance and vice versa.

Table 2.3 – Stripping tests for loose aggregate.

| Test Method | Water Volume | Duration | Aggregate Size | Sample Size | Extra Features |
|---|----------------------------|----------------|--|------------------------|-----------------------------------|
| Static immersion test – AASHTO T182, ASTM D1664 | 400 ml distilled water | 16 to 18 hrs | Single size | 100 g | - |
| Total water immersion test - 'twit' | Distilled water | 48 hrs | 14 mm aggregate | - | 25°C |
| Rolling bottle method | 250 ml deionised water | 75 min | 6.3 mm to 8 mm with 0.1 mm binder film | 200 particles | Glass rod, flask rotated @ 40 rpm |
| Boiling water test - ASTM D3625 | 500 ml distilled water | 10 min | Single size or graded | 200 – 300 g | Boiling water |
| Ancona stripping test | 200 ml distilled water | 45 min | 6 mm to 10 mm with 3 g of bitumen | 60 g | Boiling water |
| Boiling water stripping test | 600 ml demineralised water | 10 min | 10 mm to 14 mm with 1.8% binder | 200 g | Boiling water, Chemical attack |
| Ultrasound method | Water | - | Test piece – 20 mm x 80 mm | 0.2 g bitumen – 0.12mm | Ultrasound |
| Net adsorption test – SHRP | 2 ml of water | 6 hrs 8 hrs | Passing 4.75 mm | 50 g | 140 ml – bitumen-toluene sol |
| Net adsorption test – Modified | 2 ml of water | 6 hrs 8 hrs | Passing 4.75 mm, Specific grading | 50 g | 140 ml – bitumen-toluene sol |

Dynamic Immersion Tests

Dynamic immersion tests are similar to static immersion tests except that the loose mixture is mechanically agitated by shaking or kneading. A visual assessment is then made to estimate the degree of stripping. However, the reproducibility of the test can be very poor [Whiteoak, 1990].

Rolling Bottle Method

The rolling bottle (flask) method (CEN prEN 12697-11) is used to determine the adhesion between single size aggregate and bitumen. In the test 200 spherical aggregate particles (6.3 mm to 8 mm) are coated with a 0.1 mm thick binder film. The coated aggregate is then placed in a 250 ml flask containing deionised cold

water and a glass rod and rotated at 40 rpm for three days. The amount of retained bitumen is then visually determined [Airey, 2002].

Boiling Water Test (ASTM D3625)

This test is a result of the assimilation of different boiling tests used by several US state agencies [Terrel and Shute, 1989], such as the Texas boiling test [Kennedy et al., 1983]. The boiling water test involves placing a 200 to 300 g sample of coated aggregate (single size aggregate or aggregate graded to design specifications) in boiling water (500 ml of distilled water) for 10 minutes. The mixture is stirred three times with a glass rod whilst it is being boiled. After boiling, the mixture is dried and the amount of bitumen loss is determined by visual assessment. The test is very subjective and known to provide inconsistent results in terms of identifying moisture sensitive mixtures. In addition the test only reflects the loss of adhesion and does not address loss of cohesion.

Ancona Stripping Test

The Ancona Stripping Test (AST) is used to evaluate the stripping potential of a bitumen-aggregate system [Bocci and Colagrande, 1993]. The procedure entails placing a sample of bituminous gravel (60 g of gravel, passing 10 mm sieve and retained on 6 mm sieve, mixed with 3 g of bitumen) in a 600 ml beaker with 200 ml of distilled water. The 600 ml beaker is then placed in a 2000 ml beaker containing 600 ml of boiling water for 45 minutes. At the end of this period, the beaker is removed and cooled to ambient temperature. The bituminous gravel is removed from the beaker and a visual assessment made of the percentage stripping.

Boiling Water Stripping Test

The BRRC have combined the boiling water test with a more objective means of assessing the amount of stripping using a stripping ratio calibration curve, thereby eliminating the low precision associated with visual assessment [Choquet and Verhasselt, 1993]. The procedure involves coating 1.5 kg of aggregate (10 to 14 mm fraction) with 1.8% by mass of bitumen [BRRC, 1991]. Mixtures of uncoated (bare) aggregate and the coated aggregate are then produced and subjected to chemical attack to produce a calibration curve of acid consumption against percentage uncoated aggregate. 200 g of the coated aggregate is then boiled in 600 ml of

demineralised water for 10 minutes, allowed to dry and subjected to chemical attack to determine, based on the calibration curve, the amount of stripping.

Ultrasonic Method

Vuorinen and Valtonen [1999] developed an ultrasonic method to measure the resistance to stripping of coated aggregates. In the test a polished stone test piece (20 mm × 80 mm, with a thickness of 10 mm) is coated with 0.2 g of bitumen to give a binder film thickness of 0.12 mm. The coated test piece is then subjected to ultrasound under water where microscopically small bubbles of negative pressure strip the bitumen mechanically from the stone. The degree of stripping is determined either by weighing the stripped test piece or by visual assessment.

Net Adsorption Test

The Net Adsorption Test [SHRP Designation M-001; Harrigan et al., 1994] was developed by Curtis et al. [1993] and used as a screening procedure for selecting bitumens and aggregates, as well as determining the effectiveness of antistripping additives, as part of the Superpave Mixture Design Method [Kennedy et al., 1994]. This is accomplished by measuring the amount of bitumen dissolved in toluene that is adsorbed onto the aggregate surface followed by the amount which is desorbed (removed) by the addition of water to the system. The amount of bitumen which remains on the aggregate after aqueous desorption is termed the Net Adsorption. The adsorption value over 70% is considered as a good aggregates-binder bond performance [Cominsky, 1994].

Walsh et al. [1996] later proposed a modification to the Net Adsorption Test. In the modified net adsorption test, the aggregate is prepared to a specific grading rather than simply passing the 4.75 mm sieve. In addition an initial adsorption value is calculated as well as the net adsorption in order to provide a more discriminating assessment of affinity and resistance to stripping of the binder-aggregate system [Woodside et al., 1994].

2.7.2 Tests on Compacted Mixtures

Numerous tests on compacted asphalt mixtures, either prepared in the laboratory or cored from existing pavements, have been developed in an attempt to assess the moisture susceptibility of asphalt mixtures. These methods are summarised in the following sections and listed in Table 2.4.

Table 2.4 – Moisture damage tests for compacted asphalt mixtures.

| Test Method | Thermal cycling | Performance Tests |
|--|---|--|
| Freeze-thaw pedestal test (FTPT) | -12°C for 12 hours and 49°C for 12 hours | Cracking of specimen over a fulcrum |
| Immersion compression test – AASHTO T165, ASTM D1075 | 49°C for 4 days or 60°C for 24 hours, 23°C for 4 hours | Compressive strength |
| Marshall stability test – AASHTO T245 | Vacuum treatment under water @ 0°C to 1°C, 60°C for 48 hours | Marshall stability |
| Duriez test – NFP 98-251-1 | 18°C for 7 days | Unconfined compression @ 18°C and 1 mm/s |
| Lottman procedure | Distilled water @ partial vacuum of 600 mm Hg for 30 minutes, atmospheric pressure for 30 minutes, -18°C to -12°C for 15 hours, 60°C for 24 hours | Indirect tensile strength and stiffness |
| Tunnicliff and Root procedure | Distilled water @ partial vacuum of 508 mm Hg until 55% to 80% saturation, 60°C for 24 hours | Indirect stiffness |
| Modified Lottman procedure – AASHTO T283 | Distilled water @ partial vacuum of 508 mm Hg until 60% to 80% saturation, -18°C to -12°C for 15 hours, 60°C for 24 hours | Indirect tensile strength and stiffness |
| Bitutest protocol | Partial vacuum of 510 mm Hg @ 20°C for 30 minutes, saturation at 60°C for 6 hours, 5°C for 16 hours | NAT ITSM testing @ 20°C |

The tests typically involve the measurement of a change in a mechanical property of a compacted asphalt mixture after a certain procedure of moisture conditioning. The generally measured mechanical properties are indirect tensile stiffness and/or strength.

Texas Freeze-Thaw Pedestal Test

The Texas Freeze-Thaw Pedestal Test (FTPT) attempts to simulate viscosity changes in asphalt mixtures which have been in service for five years [Kennedy et al., 1982]. In the test, small specimens (41 mm in diameter by 19 mm in height) are fabricated from a single size aggregate (passing the 0.85 mm sieve, and retained on the 0.5 mm sieve). The variation or effect of physical properties such as aggregate gradation,

density, aggregate interlock are minimised through the use of a single size aggregate so that the test primarily evaluates the strength of bonding and binder cohesion.

After fabrication, the specimen is cured at 23°C for 3 days then placed on a pedestal which acts as a fulcrum. The arrangement is placed in a water bottle and subjected to thermal cycling, consisting of freezing at -12°C for 12 hours and thawing at 49°C for 12 hours (total 24 hours), until the specimen is cracked. Kennedy et al. [1982] proposed that the range of values between 10 and 20 thermal cycles to cracking would be the borderline between stripping and non-stripping mixtures. The test seems to be able to identify some moisture susceptible mixtures while being insensitive to others.

Immersion Compression Test

The Immersion Compression Test [AASHTO T165, ASTM D1075] is widely used throughout the United States to evaluate the loss of strength of compacted asphalt mixtures. In the test the index of retained strength (IRS) is obtained by comparing the compressive strength of unconditioned specimens (air cured at 23°C for 4 hours) to that of conditioned duplicate specimens which have been immersed in water for 4 days at 49°C (or 60°C for 24 hours) and then conditioned in water at 23°C for 4 hours. The Asphalt Institute recommends that mixtures be rejected if they have an IRS less than or equal to 75% [Terrel and Shute, 1989].

Marshall Stability Test

The Marshall Stability Test [AASHTO T245] is widely used to evaluate the relative performance of asphalt mixtures. In the test, the stability of unconditioned specimens is compared with the stability of duplicate specimens which have been subjected to some form of moisture conditioning. Terrel and Shute [1989] note that the conditioning procedure varies amongst organisations and is usually an adaptation from one of the existing procedures.

The Shell method uses at least eight specimens manufactured using a prescribed aggregate type, aggregate gradation, bitumen content and void content [Whiteoak, 1990]. Four of these specimens are tested according to the standard Marshall

stability. The remaining four specimens are vacuum treated under water at a temperature between 0 and 1°C, stored in a water bath at 60°C for 48 hours and tested for Marshall stability. The retained Marshall stability is then determined as the ratio of the conditioned/unconditioned specimens.

Duriez Test (NFP 98-251-1)

The Duriez test has been used for over 40 years in France to assess moisture sensitivity of asphalt mixtures [Corte and Serfass, 2000]. The procedure is performed on 80 mm or 120 mm diameter cylindrical specimens, statically compacted under a pressure of 12 MPa. The moisture conditioning consists of submerging specimens under water at 18°C for 7 days. The sensitivity to moisture is then assessed as the ratio of the unconfined compressive strength of the conditioned to unconditioned specimens at a temperature of 18°C and a loading rate of 1 mm/s.

Lottman Procedure

The Lottman procedure [Lottman, 1982] is commonly used to predict moisture damage in dense-graded bituminous mixtures. In this procedure samples are subjected to vacuum saturation alone or vacuum saturation followed by freeze-plus-warm-water soak, more commonly referred to as freeze-thaw. The test specimens, 100 mm in diameter and 63 mm in height, are then tested to produce conditioned to unconditioned ratios of indirect tensile strength and stiffness. The short-term analysis, vacuum saturated to dry ratio, is intended to reflect a field performance up to 4 years, while the long-term performance, vacuum saturated plus freeze-thaw to dry ratio estimates the field performance from 3 to 12 years.

The vacuum saturation part of the procedure consists of submerging the specimens in distilled water in a partial vacuum of 600 mmHg (80 kPa) for 30 minutes. The samples are then left saturated at atmospheric pressure for a further 30 minutes, conditioned in water at the test temperature for 3 hours and then tested to obtain conditioned and unconditioned ratios of indirect tensile strength and stiffness. The freeze-thaw procedure consists of tightly wrapping the vacuum saturated specimens in plastic wrap, placing them in heavy-duty plastic bags, each containing approximately 3 ml of distilled water, and freezing them at -18 to -12°C for

15 hours. The plastic wrap is then removed and the samples are heated to 60°C in a distilled water bath for 24 hours, conditioned in water at the test temperature for 3 hours and tested.

Tunnickliff and Root Procedure

The Tunnickliff and Root procedure [Tunnickliff and Root, 1984] is similar to the Lottman procedure [Lottman, 1982]. However the procedure uses a slightly different vacuum saturation and thermal treatment methodology, and excludes freezing of the saturated specimens. The procedure controls the degree of saturation to ensure that enough moisture is present to initiate moisture damage and to avoid unrelated damage, such as stresses induced from over saturation. Conditioning involves submerging the specimens in distilled water and incrementally applying a partial vacuum of 508 mmHg (68 kPa) in 5 minute increments until a degree of saturation between 55 to 80% is achieved. The specimens are then heated in a distilled water bath at 60°C for 24 hours, conditioned in water at the test temperature of 25°C for 1 hour and tested.

Modified Lottman Procedure (AASHTO T283)

The AASHTO T283 (or modified Lottman) procedure combines features of both the Lottman [1982] and Tunnickliff and Root [1984] procedures. The Lottman procedure attempts to achieve a 100% saturation in its specimens, while the Tunnickliff and Root procedure attempts to control the saturation level between 55 and 80%. Due to concerns on over saturation, where the negative pressure applied in the vacuuming process may cause extra non-moisture-related damage in specimens, the modified Lottman procedure uses a decreased saturation level of between 60 and 80% [Scherocman et al., 1986]. As the saturation level achieved by partial vacuum is primarily responsive to the magnitude of the vacuum and relatively independent of the length of time, this reduced saturation was achieved by reducing the partial vacuum from 600 mmHg (80 kPa) to 508 mmHg (68 kPa).

LINK Bitutest Moisture Sensitivity Protocol

Scholz [1995] produced a test method for measuring moisture sensitivity of compacted bituminous mixtures that involves determining the ratio of conditioned to

unconditioned indirect stiffness modulus values as measured with the Nottingham Asphalt Tester (NAT). The conditioning consists of saturation under a partial vacuum of 510 mmHg (68 kPa) at 20°C for 30 minutes, followed by immersion in water at 60°C for 6 hours, and immersion in water at 5°C for 16 hours. The samples are finally conditioned in water at 20°C for 2 hours prior to the stiffness measurement.

A very severe form of winter conditioning has been produced by Hobeda [2000] for extreme Nordic conditions. The procedure consists of submerging test specimens under vacuum saturation, firstly, in a concentrated NaCl solution for 48 hours and then in a distilled water environment for a further 48 hours. The specimens are then subjected to seven freeze-thaw cycles (-20°C and 20°C for 12 hours each) before indirect tensile stiffness testing at 10°C.

2.8 Summary and Conclusions

An extensive review of existing test methods, protocols and techniques for the assessment of ageing and moisture sensitivity of bituminous materials, has been undertaken. The testing methods can be broadly grouped into the following categories; 1) Binder ageing, 2) Mixture ageing (loose mixture or compacted specimen) and 3) Moisture sensitivity (loose mixture or compacted specimen).

The binder ageing can be divided into short and long term ageing. The ‘short-term’ describes the ageing process (mainly volatile loss) that occurs during the asphalt mixture construction process (heating up, mixing, delivery and compaction) and the ‘long-term’ describes the progressive oxidation process of the in-place material in the field during its service life.

The most commonly used binder ageing tests are the high temperature TFOT and RTFOT. It is noted that bitumen aged in the TFOT and RTFOT experiences higher volatile loss during testing compared to that experienced during low temperature field ageing of pavement mixtures. Also the level of oxidative ageing in the tests is considerably lower than that found during field ageing. Therefore these tests are

only suitable to simulate for short-term ageing, but not ideal to estimate the long-term ageing of bitumen in pavements.

Based on the inability of these high temperature oven ageing tests to predict 'long-term' field ageing, tests such as the Shell Microfilm Test, RMFOT, TFAAT and others were introduced with reduced temperatures and increased ageing times. Tests such as POB and PAV utilise a pressurised condition to the ageing process to give a more accelerated oxidation process under moderate temperatures and relatively short test periods. From the questionnaire survey undertaken (see Appendix B), the most commonly used tests for short-term ageing and long-term ageing simulation appear to be the RTFOT and PAV protocols respectively.

Eckmann [1999] stated that RTFOT and similar test methods are probably adequate for short-term ageing as there is good evidence that they simulate short-term ageing reasonably well. Nevertheless he admitted that these test methods may need to overcome some operational difficulties when testing PMBs, such as the use of the MRTFOT as recommended by Bahia et al. [1998]. In terms of long-term ageing, Eckmann proposed to look into the kinetic aspect of ageing, such as the RCAT, although he generally concluded that no one test seems to be satisfactory in all cases.

Regardless of the short-term or long-term aspect, the elevated temperature is apparently the essential and critical acceleration factor for ageing process. However, for the long-term aspect, there are concerns over this 'unrealistically' elevated temperature as it could significantly alter the binders to an unrepresentative extent to that found in the field, particularly for modified binders [Hayton et al., 1999]. Thus the introduction of a pressurised condition, such as the PAV protocol, seems to be a breakthrough approach as it gives one more factor to control the acceleration of the ageing process (oxidation) together with the temperature and test duration. The HiPAT procedure seems to be a reasonable and recommendable protocol for the long-term ageing simulation as it uses an even lower temperature than the PAV.

Most ageing protocols for the asphalt mixture also use an elevated temperature as a dominant factor to age the binder (in the mixture) in an accelerated manner to shorten the test period. It is interesting to note that the elevated temperatures applied

on the compacted mixture are considerably lower than those for the loose mixture. This is to avoid/minimise unnecessary structural disruption (due to cohesion loss of binder) caused by the high temperature. The 'highest' temperature applied on compacted specimens was found to be 100°C used by Korsgaard et al. [1996] for their PAV trial. However, this was to recover binder from the specimens, rather than performing any mechanical test on the compacted specimen. Knowing that even at 60°C there were concerns over the high temperature effect [Kim et al., 1986; Von Quintas et al., 1991 and Bell, 1989], 85°C appears to be the maximum applicable temperature for compacted specimens.

Moisture susceptibility tests generally have a conditioning and an evaluation phase. The conditioning processes associated with most test methods are to simulate the deterioration of the asphalt mixture in the field exposure conditions, in an accelerated manner to be tested in a reasonably short period of time. Normally the acceleration is achieved by immersing the material at an elevated temperature. It is noted that the elevated temperature applied on the loose mixture is commonly the boiling temperature of 100°C. However, 60°C appears to be the highest temperature used on compacted specimens. As with the ageing tests, this is to avoid (or minimise) any unnecessary structural disruption caused by the high temperature, rather than moisture damage. Additionally the samples can be subjected to extra conditioning factors which do not necessarily simulate field conditions but possibly more accelerate the moisture damage, such as ultrasonic methods [Vuorinen and Valtonen, 1999].

The two general methods of evaluating 'conditioned' specimens are either a visual evaluation or the subjection of the specimen to a physical test. Visual evaluation is used extensively in moisture susceptibility procedures especially for the coated aggregate tests (uncompacted 'loose' mixture). However, visual assessment tends to be rather subjective and moreover, the assessment is performed on the material 'before' its compaction process, which is one of the most decisive factors for the 'field performance' of the material.

Most moisture sensitivity test procedures on the compacted mixture measure the loss of strength or stiffness of an asphalt mixture due to moisture damage. The most

common way of quantifying the damage caused by the conditioning process is to directly compare the 'conditioned' mechanical properties to those of 'unconditioned'. The mechanical properties can be evaluated using various mechanical tests which can be divided into destructive (compression, stability, indirect tensile strength and fatigue) and non-destructive (indirect tensile stiffness and resilient modulus) approaches. An apparent advantage of using a non-destructive test is that the very same specimen can be used for a direct comparison of unconditioned and conditioned properties. Therefore a relatively small number of specimens is required to define a trend.

Saturation is a common link to all the conditioning procedures and is the key to the laboratory simulation of moisture damage in the test specimens. Saturation is obtained either by immersion of the specimen in static or boiling water and/or by applying a certain degree of vacuum. The principal reason for the vacuum is to draw the water into the specimen to shorten the time required for the specimen to reach the desired saturation level.

The correlation of ageing simulated in the laboratory test and that of the field material has been relatively well established, compared to the correlation of moisture sensitivity tests to field performance. Since ageing is only focussed on the binder property, the correlation can be directly established by comparing the chemical component and/or physical properties of the binder aged in the laboratory to those of the binder recovered from field material of a known period of service life and in a known climate condition. However, moisture sensitivity is focussed on mixture properties which are a much more complicated aspect. In field material after a certain period of service life, the measured material properties result from combined effects of various damage factors, such as binder ageing, together with adhesion loss due to moisture damage, while the mixtures (including aggregates) are gradually degraded by traffic load and/or other environmental factors. Thus, it is practically difficult to directly correlate the material property change (strength, stiffness etc) from a moisture sensitivity test to those observed in field material, in terms of service life.

It is interesting to note that existing durability protocols are designed to investigate one aspect only (i.e. ageing test for ageing sensitivity, moisture sensitivity test for moisture damage). However, particularly in the long-term service condition, it is more likely that the pavement is undergoing both aspects of ageing and moisture damage together (i.e. materials are damaged by water infiltration while the binder in the material is being aged).

Chapter 3. Preliminary Trials

3.1 Ageing & Moisture Sensitivity Test Review

From the literature review (see Chapter 2) and the questionnaire survey undertaken (see Appendix B), considering both practicability and the availability of testing equipment in the laboratory, a couple of different ageing regimes and a moisture sensitivity regime were selected as the first step of the test programme.

The main purpose of these preliminary trials was to find a suitable test method which could reproduce in the laboratory the decrease in stiffness modulus that had been measured from the HMB trial site (see Section 1.1). For the purpose of combining ageing and moisture sensitivity process, this preliminary investigation basically performed the ageing process first, followed by the water sensitivity process. This was considered to be a combined durability protocol at this stage.

A 28 mm Dense Bitumen Macadam (DBM) base material [BS 4987: Part 1, 2001] using a typical acidic aggregate (referred to as Acidic) and a 15 Penetration grade of binder (referred to as 15B, 4% by mass) was used to replicate the material used in the HMB trial site. A target air void content of 8% was identified (this was the typical air void content measured from the site) based on 'Rice' maximum density of the mixture [BSEN 12697: Part 5, 2002] and 'sealed' bulk density of core specimens (see Chapter 5 for more details of the specimen production). The mixture was compacted using a laboratory roller compactor and cylindrical specimens were cored from the slab for standard Indirect Tensile Stiffness Modulus (ITSM) testing [BS DD 213, 1993]. The Stiffness Modulus of the material at 20°C was the main performance parameter used throughout this study.

The stiffness modulus was measured before and after each ageing regime. Following each ageing regime a moisture sensitivity cycle was performed on the same specimens and the stiffness modulus was again measured. The LINK moisture sensitivity protocol [Scholz, 1995] was selected for the standard moisture sensitivity test at this stage. Each cycle consisted of conditioning the specimen in water, at

60°C for 6 hours, 5°C for 16 hours and finally 20°C for 2 hours. Typically, each test comprised up to four moisture conditioning cycles. The following sub-sections describe the selected regimes in more detail.

Regime #1

Regime #1 was used as the control. Specimens were stored at 5°C in a cold dry room in an attempt to prevent any ageing or moisture damage.

Regime #2

Regime #2 used the standard LINK ageing protocol where the compacted specimen was heated in a dry oven at 85°C for 120 hours [Scholz, 1995].

Regime #3

Regime #3 was similar to regime #2 but included a large water bath placed in the oven to create a relatively humid environment for the ageing process in an attempt to combine the effects of ageing and moisture damage.

Regime #4

Regime #4 used a Low Pressure Oxidation (LPO) configuration where hot air at 85°C was forced through the specimen using a vacuum pump. The test temperature was selected to be 85°C to give the same temperature condition as in the other regimes. This was achieved by placing the specimen unit (see Figure 3.1) into a dry oven at 85°C. The flow rate was 3l/min, based on a configuration developed by Khalid and Walsh [2001], and the test duration was 120 hours (identical to Regimes #2 and #3). The LPO configuration was used to ensure continued oxidation throughout the entire specimen during the test duration.

Regime #5

Regime #5 used the standard Pressure Ageing Vessel (PAV) for binder ageing to age the compacted mixture specimens. The HiPAT protocol [Hayton et al., 1999] was used, where a temperature of 85°C and a pressure of 2.1 MPa were applied for 65 hours. The HiPAT protocol was chosen as it uses a relatively lower temperature, which appears to be more applicable to the compacted specimen from the literature review (see Section 2.5.2), than the standard PAV protocol. A purpose built tray was used to place two specimens into the vessel.

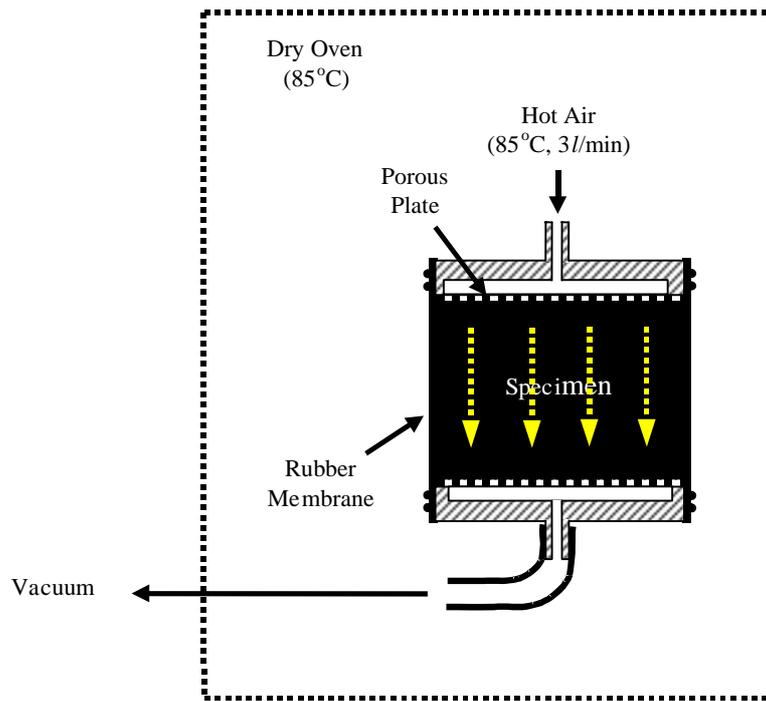


Figure 3.1 – Low pressure oxidation configuration (Regime #4).

Regime #6

Regime #6 was a unique approach. Basically it is same to regime #5 but with the modification that the specimen was partially saturated with water using a vacuum pump (a partial vacuum of 68 kPa was applied for 30 minutes). The purpose of this additional saturation process was to draw a certain amount of water into the air voids in the specimen, hoping to create a certain moisture damage condition together with the ageing process. The partially saturated specimen was placed into the PAV where the same HiPAT ageing protocol as Regime #5 was applied. The humidity inside the vessel was considered approximately constant during the treatment as considerable water condensation was found when the vessel was opened (due to a tightly concealed environment), after the protocol was completed.

3.2 Results and Analysis

Figure 3.2 shows the stiffness ratio plotted as a function of the number of treatment cycles for each regime. The stiffness ratio is defined as the ratio of the stiffness modulus measured after the treatment to the stiffness modulus measured after ageing process (Point B in x-axis). Figure 3.3 shows the same results but this time the

stiffness ratio is defined as the ratio of the stiffness modulus measured after the treatment to the initial stiffness modulus measured before ageing process (Point A in x-axis). Treatments 1 to 4 refer to the number of moisture conditioning cycles. It can be seen from Figure 3.2 that, as expected, the control (unaged) specimens (Regime #1) showed virtually no change in stiffness modulus due to ageing (A to B). It is though interesting to observe that, from Figure 3.2, the control specimens showed a marginal increase of stiffness modulus after the first moisture conditioning cycle (Point 1 in x-axis). This is thought to be partly attributed to the extra support given by the ‘saturated’ air voids (in the specimen) resulting in a marginally increased stiffness of the specimen. An additional trial to observe the stiffness change caused by the vacuum process was performed (see Table 3.1) confirming the case. Three of 15B Acidic specimens and three of 15B Basic specimens (see Chapter 5 for the specimen description) were ITSM tested under standard condition (20°C, dry) then the LINK vacuum saturation procedure (a partial vacuum of 68 kPa for 30 minutes, at 20°C) was applied. The saturated specimens were then ITSM tested again under the same condition (20°C, saturated). However this can also be partly attributed to a partial age hardening of the specimen due to the elevated temperature of 60°C used in the moisture conditioning process [Scholz, 1995].

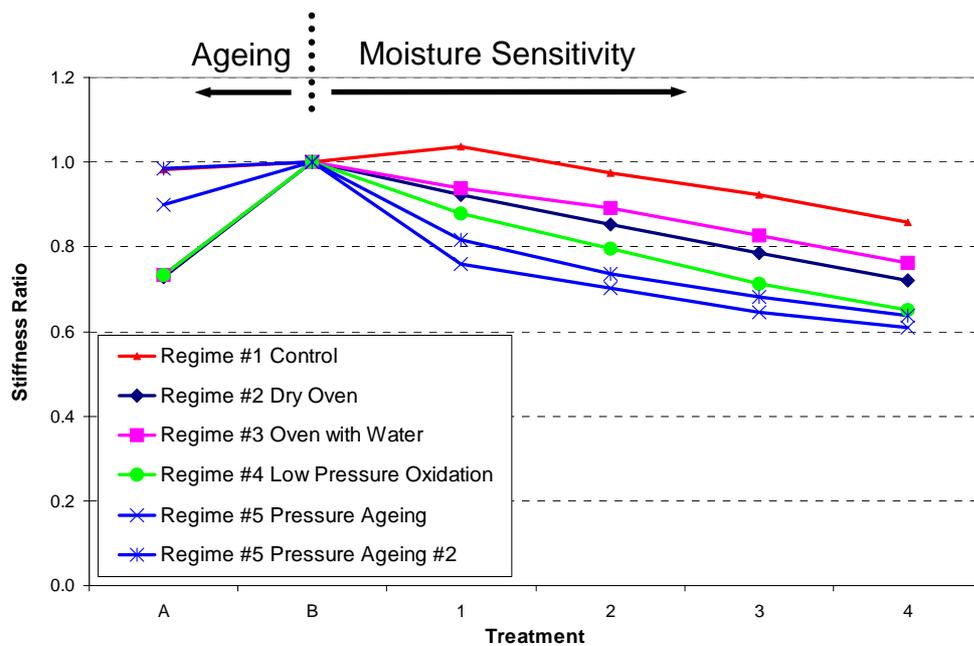


Figure 3.2 – Results of ageing & moisture sensitivity combined tests (normalised to after ageing conditions, Point B).

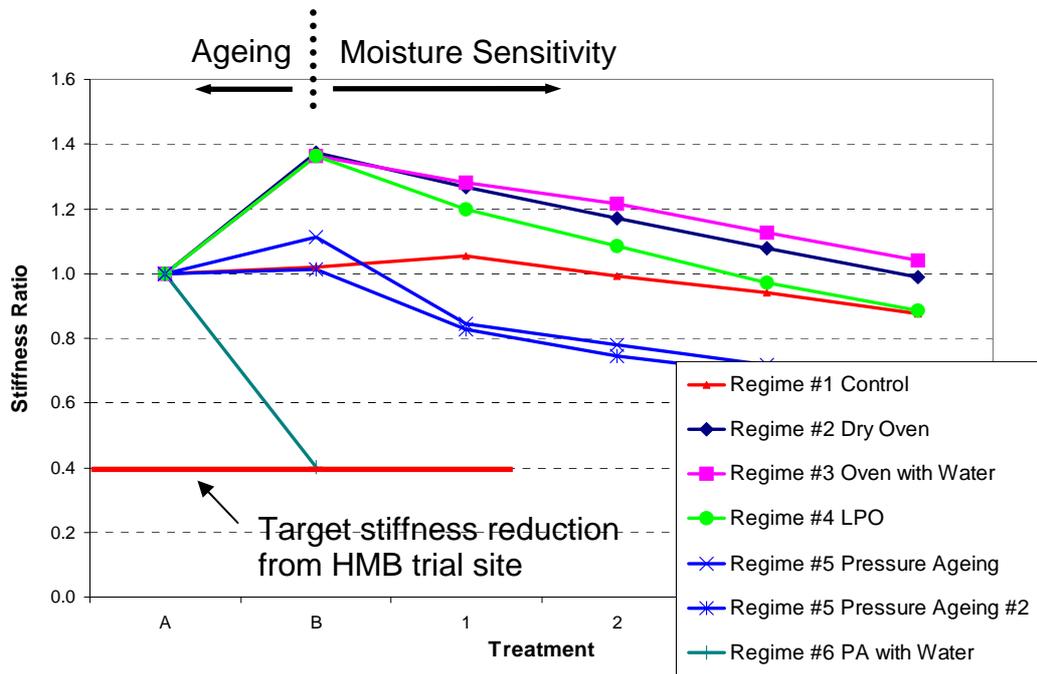


Figure 3.3 – Results of ageing & moisture sensitivity combined tests (normalised to initial conditions, Point A).

Table 3.1 – Comparison of stiffness modulus before and after the vacuum process.

| Specimen | No. | Saturation (%) | Initial (dry) Stiffness (MPa) | Saturated Stiffness (MPa) | Ratio |
|------------|-------------|----------------|-------------------------------|---------------------------|-------------|
| 15B Acidic | 1 | 59.8 | 10483 | 11195 | 1.07 |
| | 2 | 49.4 | 8973 | 10146 | 1.13 |
| | 3 | 48.5 | 10882 | 11817 | 1.09 |
| | Mean | | | | 1.09 |
| 15B Basic | 1 | 60.3 | 10186 | 11085 | 1.09 |
| | 2 | 54.3 | 10570 | 11679 | 1.10 |
| | 3 | 58.5 | 10018 | 11773 | 1.18 |
| | Mean | | | | 1.12 |

It can be seen from Figures 3.2 and 3.3 that the oven aged specimens with and without moisture (Regimes #2 and #3) showed very similar ageing sensitivity with up to a 37% increase in stiffness modulus after ageing. Regime #2 then showed a 28% reduction in stiffness modulus whilst regime #3 showed a 24% reduction in stiffness modulus due to moisture damage. It is clear that the additional humidity of Regime #3 did not create a more severe ageing/moisture damage environment (compared to Regime #2).

It can also be seen from Figures 3.2 and 3.3 that Regime #4 (LPO) showed a very similar ageing sensitivity (36% increase in stiffness modulus) compared to the oven ageing (Regimes #2 and #3). It could be assumed that, since the material has a high air void content ($\approx 8\%$), the hot air could freely flow inside the specimen without being forced.

Figures 3.2 and 3.3 show that the HiPAT aged specimens (Regime #5, 2 repetitions) showed the least ageing and the most moisture sensitivity. The stiffness modulus increased by only 5% after ageing and then decreased by 40% due to moisture damage.

As shown in Figure 3.3, the saturated HiPAT aged specimens (Regime #6) showed the most severe behaviour even before undergoing the moisture conditioning process. After the ageing process, the stiffness modulus dropped to approximately 40% of its initial value (i.e. a 60% reduction).

The HiPAT aged regimes without/with moisture (Regimes #5 and #6) appear to make specimens more susceptible to the following moisture conditioning (Regime #5) or incorporated saturated water (Regime #6). It is assumed that the high pressure inside the vessel has induced a certain type of internal damage (e.g. micro-cracking), which has enabled water to infiltrate more easily to the specimen. A more detailed investigation of the high pressure effect on compacted specimens is presented in Chapter 7.

3.3 Recovered Binder Assessment

Binder was recovered from the specimens after they had been subjected to the two pressure ageing protocols (Regimes #5 and #6) to assess the influence of the dry (Regime #5) and partially saturated pressure ageing procedures (Regime #6) on the binder properties. The binders were recovered using the BS protocol [BSEN 12697-4, 2000] in sufficient quantities to allow Dynamic Shear Rheometer (DSR) testing to be undertaken. The ‘recovered 15B’ DSR results were compared to those from the ‘15B binder’ DSR test series (see Appendix C for more details). Figures 3.4 and 3.5 show the complex modulus and phase angle master curves of the recovered 15B binders from the processed specimen and aged 15B binders at a reference temperature of 25°C.

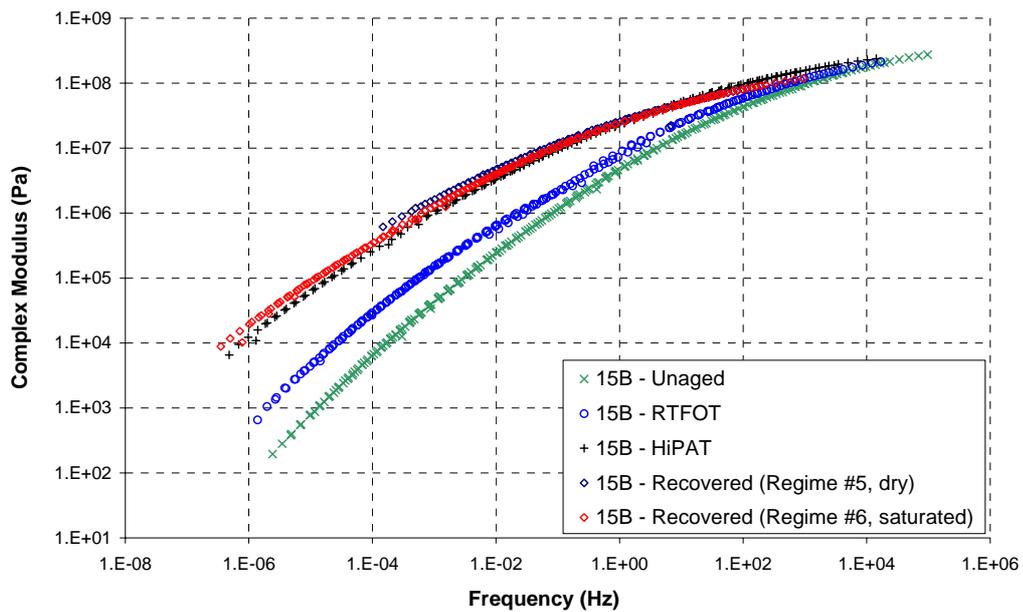


Figure 3.4 – Complex modulus master curves of aged and recovered 15B binders.

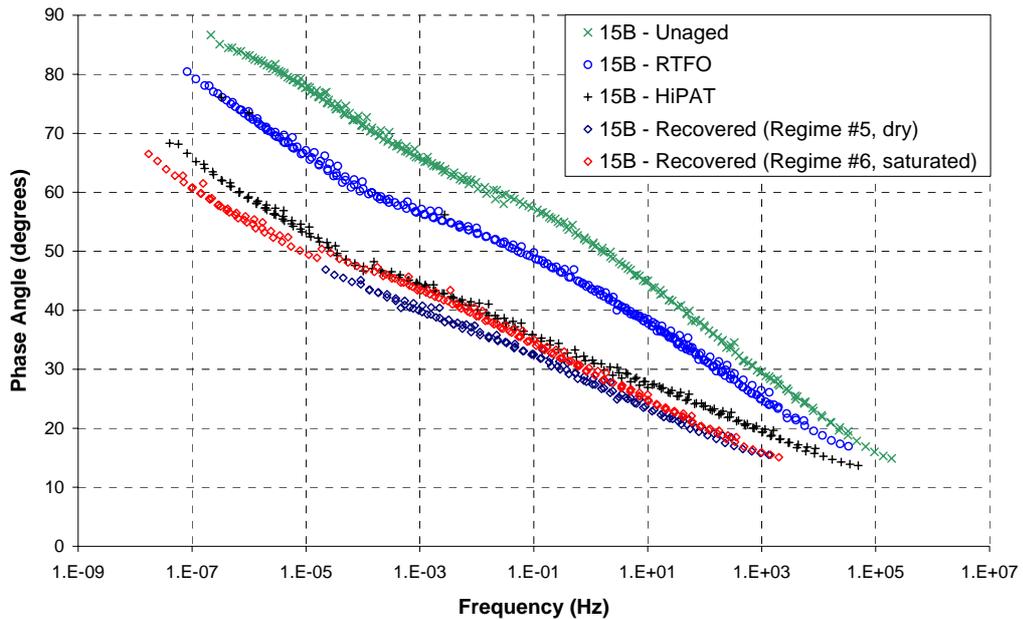


Figure 3.5 – Phase angle master curves of aged and recovered 15B binder.

These figures clearly show that the binders recovered from specimens tested using Regimes #5 and #6 have reasonably similar rheological characteristics to those of the HiPAT aged binder. It can therefore be concluded that the loss of mixture stiffness modulus observed in Regime #6 is due to moisture damage rather than any alteration in the rheological characteristics of the binders. Also, regardless of the presence of water in the pressure vessel, the binder in the mixture is likely to be aged according to the given HiPAT ageing condition. Further investigation and discussions of the recovered binder assessment are presented in Chapter 9 (see Section 9.4.2).

3.4 Summary and Conclusions

Among the various regimes tried in this chapter, to find a suitable durability test method which fulfils the given objectives of this study, the HiPAT ageing using saturated specimens (Regime #6) showed the most distinctive and interesting behaviour. Firstly it was the only process which was severe enough to produce the target stiffness reduction (60%) within reasonably shorter period of time (65 hours). Secondly it was the only process which had ageing and moisture sensitivity schemes in ‘one process’.

The investigation (Section 3.3) on the recovered binder extracted from the specimens which had been through the Regimes #5 and #6 clearly demonstrated a comparable level of binder ageing to the standard HiPAT aged binder. Any loss of stiffness modulus or strength for the moisture aged asphalt mixtures can therefore be attributed to a loss of the cohesive and/or adhesive properties of the mixture rather than any unexpected “softening” of the binder.

Knowing that the binder in the mixture will be ‘long-term’ aged according to the given HiPAT ageing conditions (air temperature of 85°C and pressure of 2.1 MPa), while the mixture is ‘moisture damaged’ by saturated water in the pressure vessel (water temperature of 85°C), Regime #6 approach showed a strong possibility to be a suitable durability test method which fulfils the given objectives of this study (see Section 1.2). Thus the following chapters will describe the investigation and development of the PAV with water ageing protocols (referred to as the Saturated Ageing Tensile Stiffness, **SATS**, test).

Chapter 4. Development of SATS Protocol

4.1 Testing Method (Preliminary SATS)

From the initial trial of the SATS protocol as described in Chapter 3 (Regime #6), it was soon found that the standard PAV equipment for binder ageing was not suitable for the purpose. The first problem encountered was the size of the pressure vessel (ϕ 200 mm \times 230 mm, internal dimensions) as it could contain only two specimens per test. Moreover, the sophisticated pressure control system of the vessel showed a sign of malfunction due to the presence of water inside the vessel. Thus, a larger size (ϕ 150 mm \times 435 mm, internal dimensions) of pressure vessel (referred to as **SATS PAV**, see Figure 4.1 (a)) with a manual pressure control system, which could contain up to six specimens at a time using a purposely built portable racking system (see Figure 4.1 (b)), was purchased and commissioned. The new pressure vessel was used to artificially age and moisture condition 100 mm diameter by 65 mm thick cylindrical bituminous mixture specimens.

The specimens were cored from roller compacted slabs and trimmed to the required thickness of 65 mm (see Chapter 5 for more details). The initial stiffness modulus of the specimens was measured using the ITSM test at 20°C, after which the specimens were partially saturated with water using a vacuum pump under a negative pressure of 68 kPa for 30 minutes. The partially saturated specimens were then placed into the pressure vessel and subjected to the HiPAT protocol (temperature of 85°C and pressure of 2.1 MPa for 65 hours). At the end of the conditioning period, the specimens were allowed to cool and were then re-tested using the ITSM test at 20°C. Finally, the ratio of conditioned stiffness modulus to initial stiffness modulus (referred to as ‘retained stiffness’) was determined for each of the specimens. This is referred to as the preliminary SATS protocol.

4.2 Variability Investigation

A series of 28 mm DBM base mixture specimens [BS 4987: Part 1, 2001] using a 15 Penetration grade binder (referred to as 15B, 4% binder content) were produced with

either Acidic (referred to as 15B Acidic) or Basic aggregate (referred to as 15B Basic). The target air void content was 8% (see Chapter 5 for more details).

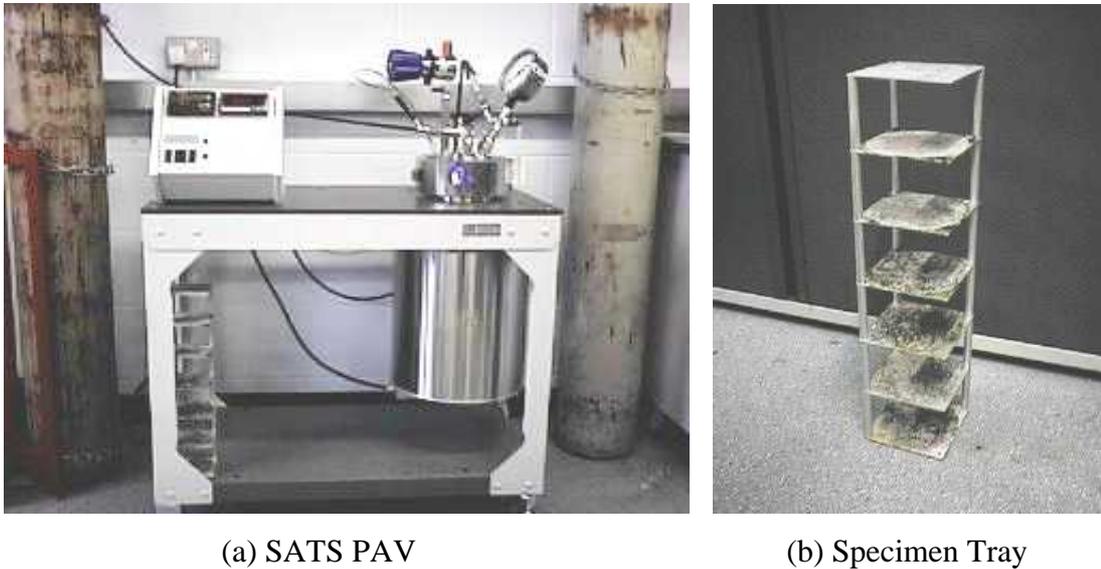


Figure 4.1 – The SATS apparatus.

To determine test variability, nominally identical materials were tested under the same nominal conditions. Figure 4.2 shows the results of repeated tests (six repetitions) of 15B Acidic and a test of 15B Basic. The numbers on the x – axis refer to the position of each specimen from top to bottom in the specimen tray (see Figure 4.3).

From Figure 4.2, it is clear that the level of stiffness reduction of the specimens (15B Acidic) observed from the Regime #6 trial in Chapter 3 (around 0.4 of retained stiffness), is generally limited to the specimens from the upper positions (position 1, 2 or 3) of the pressure vessel. Moreover, it is clear that the trend is for the retained stiffness to decrease for specimens that are higher up in the pressure vessel. Although the 15B Basic shows generally higher retained stiffness distribution (0.6 ~ 0.8), the trend is similar to that of 15B Acidic.

Results from test repetitions clearly indicated that there were different testing conditions for the different specimen locations. Thus, a more careful investigation

was performed to investigate the test conditions within the vessel and the key factors controlling the moisture damage.

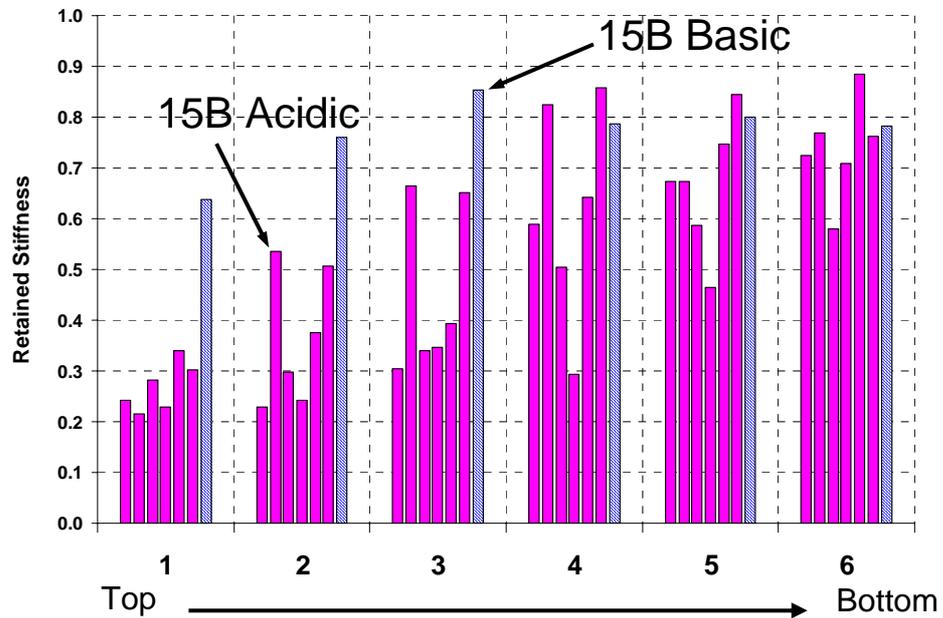


Figure 4.2 – Results from repeated preliminary SATS test on 15B Acidic and Basic specimens.

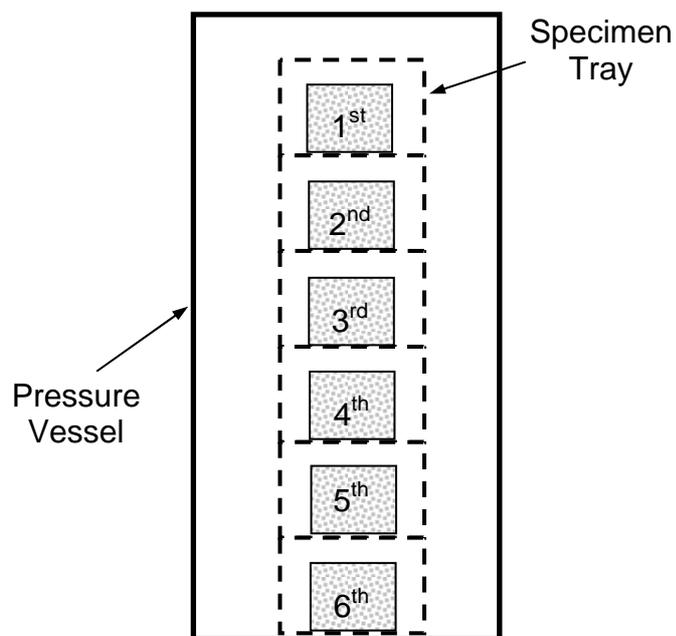


Figure 4.3 – Schematic diagram of preliminary SATS setup.

4.3 Detailed Investigation of the SATS Setup

4.3.1 Temperature

Figure 4.4 shows a schematic diagram illustrating the SATS PAV heating elements. A set of dummy specimens with thermocouples inserted in them was placed in the vessel and the target temperature (85°C) was controlled. Atmospheric pressure was used for this investigation (no additionally applied pressure).

It can be seen from Figure 4.4 that the temperature difference between specimens located at the top and bottom of the vessel was approximately 13°C. The reason was thought to be due to the combination of heat loss through the vessel cover and the fact that the main heating element is located at the base of the vessel. Also it should be noted that there is no internal forced air circulation (the source apparatus is actually intended to house a liquid medium).

To investigate the effect of this temperature variation on ageing of the bituminous material, six identical ‘dry’ specimens (15B Acidic) were tested in the vessel using the HiPAT protocols (referred to as ‘Dry HiPAT’). The results are shown in Figure 4.5. It can be seen from this figure that the retained stiffness modulus vary from approximately 1.00 (Positions 2 and 3) to 1.15 (location 5) which are very similar values observed from the Regime #5 (after HiPAT ageing) in Chapter 3.

The standard PAV used in preliminary trials in Chapter 3 has about a half height of the SATS PAV and has a proper insulation around the vessel (including the top cover). Therefore it is expected to have a much smaller temperature variation (only two specimens per test). As the specimen ageing under 13°C difference showed very similar values to those from the standard PAV, without a distinctive trend to the position of the specimen (as shown in Figure 4.2), it is presumed that the temperature variation in the SATS PAV has a very minor effect on the results observed in Section 4.2.

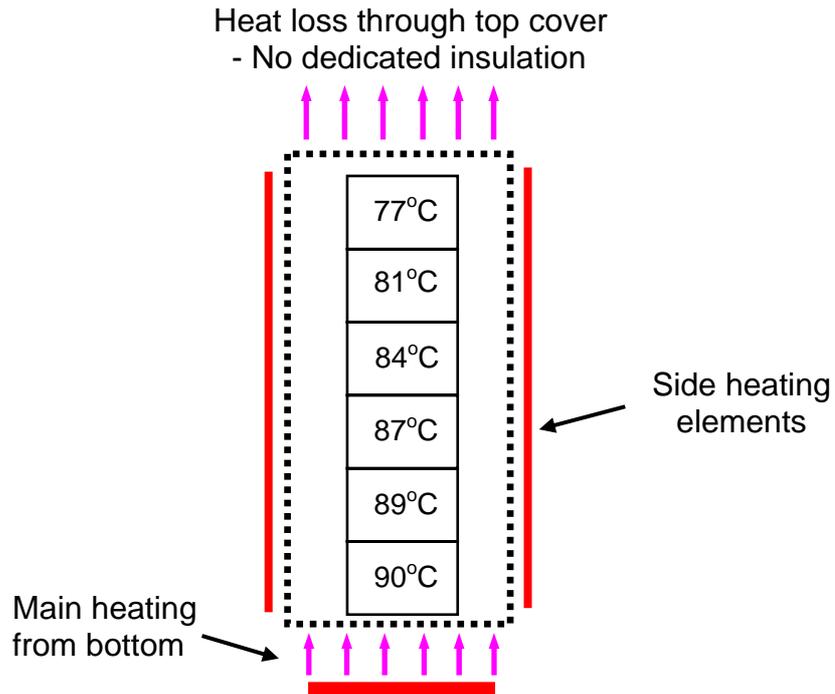


Figure 4.4 – Schematic diagram of the SATS PAV and measured temperature at each specimen position.

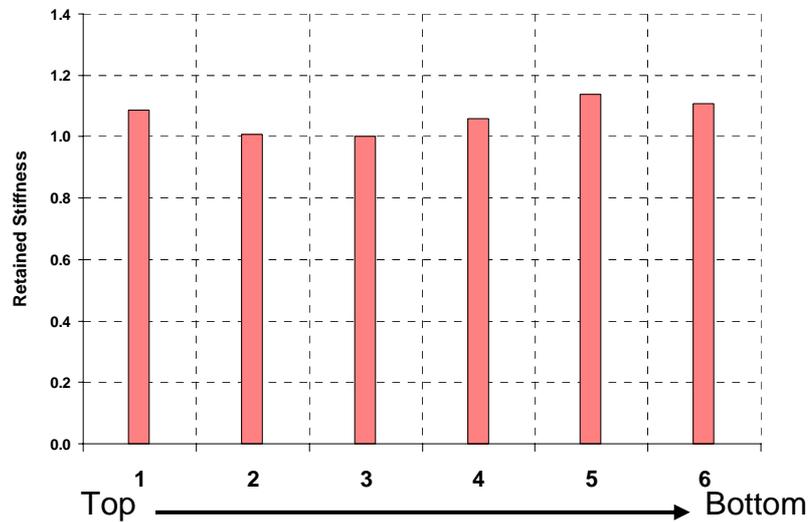


Figure 4.5 – Dry HiPAT test results using the SATS PAV (15B Acidic).

Simple modifications that involved insulating the top cover (see Figure 4.6 (b)) and utilising the side heating source only were found to significantly reduce the temperature variation, up to 4°C (see Table 4.1).

Table 4.1 – Modification of the SATS PAV to minimise temperature variation.

| Target temperature of 85°C | Max Temp. (°C) | Min Temp. (°C) | Variation (°C) |
|--|----------------|----------------|----------------|
| Normal setup | 90 | 77 | 13 |
| Insulation on top cover | 86 | 80 | 6 |
| Side heating source only (+ insulation on top cover) | 86 | 82 | 4 |



(a) Normal setup



(b) Top cover insulation

Figure 4.6 – Insulation on top cover of SATS PAV

4.3.2 Saturation

Once the specimens are vacuum saturated and placed in the vessel, as described in Section 4.1, it is likely that a certain portion of the saturated water will evaporate or drain from each specimen and circulate inside the vessel. Consequently, due to the temperature difference between the inside of the vessel ($\approx 85^{\circ}\text{C}$) and the outside atmosphere ($\approx 20^{\circ}\text{C}$), and also knowing that the side wall of the vessel is surround by heating elements (see Figure 4.4), evaporated moisture will be ‘condensed’ mostly

on the bottom face of the top cover. Figure 4.7 shows a schematic diagram of the likely water circulation in the vessel during the testing process.

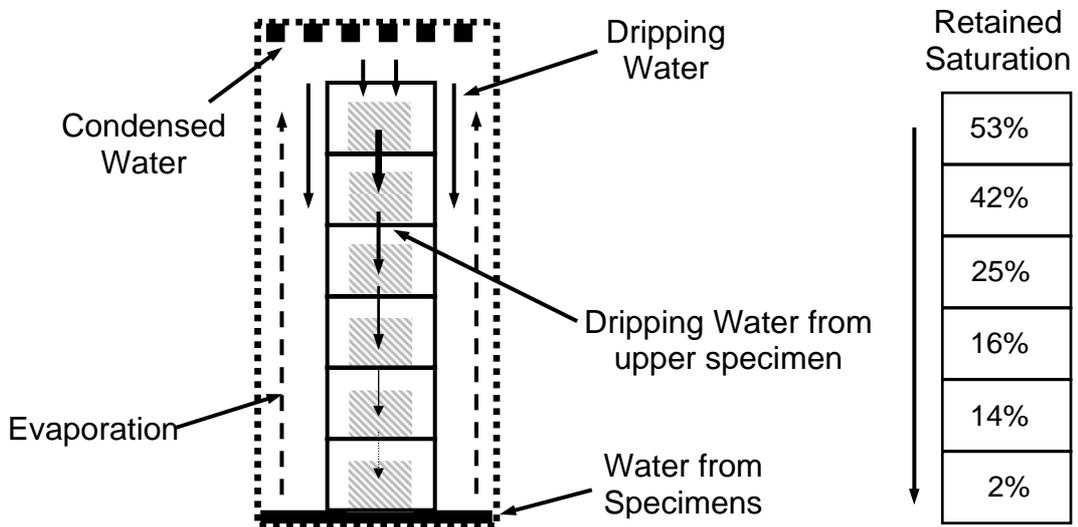


Figure 4.7 – Schematic diagram of water circulation in the preliminary SATS setup, and retained saturations (measured after the protocol).

From Figure 4.7 it is likely that a significant amount of the condensed water will directly drop on to the top specimen, while the rest will drop directly on to the bottom of the vessel. However, the rest of the specimens will also absorb a certain amount of water, since it is easily assumed that excess water from the upper specimen will eventually permeate through to the lower specimens.

A measurement of the saturation of each specimen, after the protocol had been completed (referred to as the 'retained saturation'), has been carried out and the results are also shown in Figure 4.7. It is clear that the saturation gradually decreases from the top to the bottom specimen.

The retained stiffness values of individual specimens were compared to the retained saturation. The results are shown in Figure 4.8 and it is clear that the specimens with a higher retained saturation show a lower retained stiffness (i.e. higher damage).

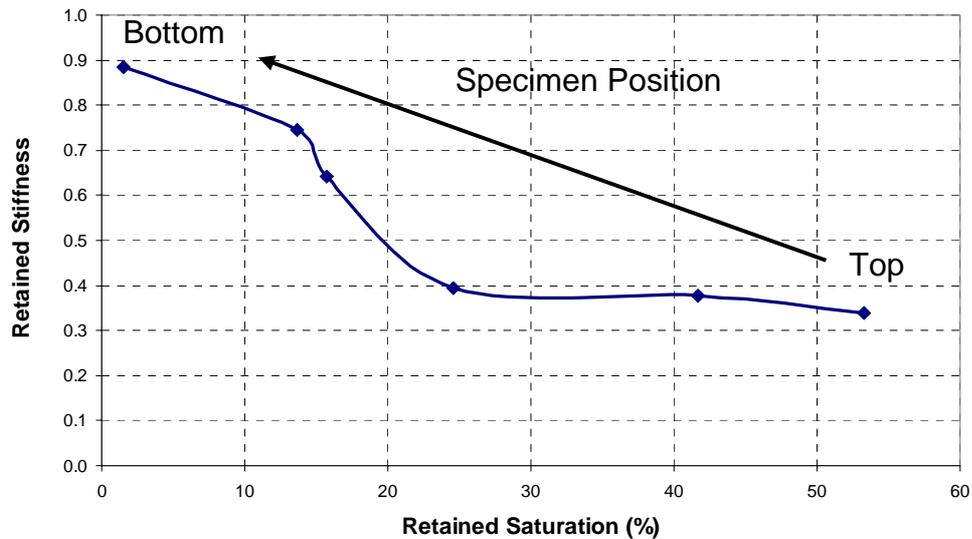


Figure 4.8 – Retained stiffness versus retained saturation plot from SATS.

The variability investigation using nominally identical sets of specimens revealed that the test conditions (temperature and saturation) vary dependent on the specimen position within the vessel (see Section 4.2). The temperature variation between the top and bottom specimens was investigated and found to be around 13°C. However, the temperature difference is considered to have a very minor role, showing little difference on the retained stiffness results (see Section 4.3.1). It is also found that the temperature variation could be controlled up to 4°C, through certain modifications. The variation in the retained saturation is thought to have a major effect on the retained stiffness, and consequently is assumed to be the major factor which caused the observed stiffness variation in Section 4.2. Plotting the results in terms of retained stiffness versus retained saturation clearly demonstrated a correlation between both factors.

Therefore, it was decided that a range of modifications would be investigated with the principal objective to better control the variability in retained saturation between specimens. Results from identical specimens (15B Acidic) from the modifications are presented and reviewed in the following sections.

4.4 Top Cover on Specimen Tray

The objective of this modification was to prevent or minimise any condensed water dripping directly onto the top specimen. A circular, inverted dish shaped ‘air permeable’ membrane was introduced on top of the specimen tray. This is to ‘divert’ the dripping water from condensation and also to prevent any ‘unnecessary condensation’ beneath the membrane. Figure 4.9 shows a schematic diagram of the likely water circulation when the top cover was introduced.

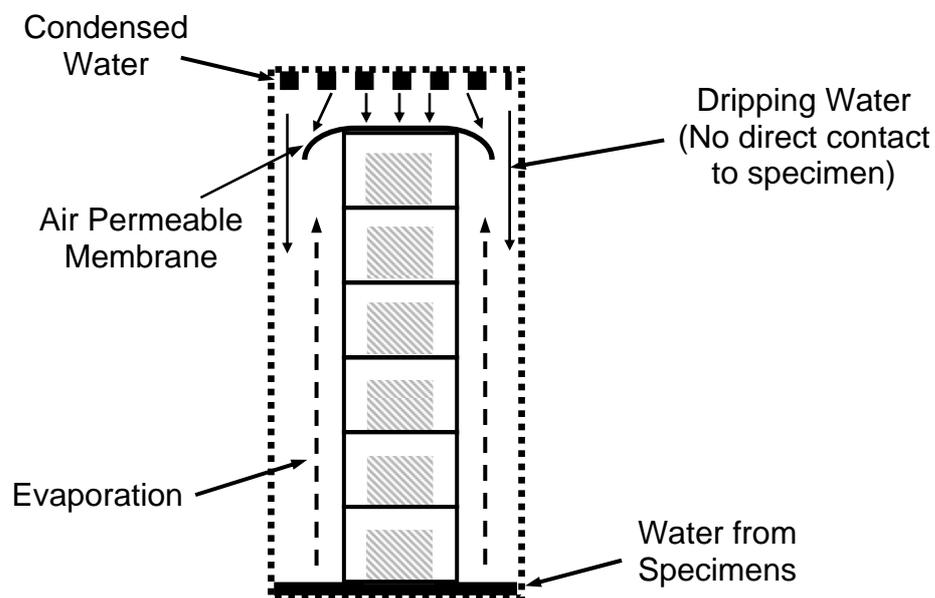


Figure 4.9 – Schematic diagram of water circulation when top cover applied.

The results were compared to the preliminary results (see Section 4.2) and are presented in Figure 4.10. It can be seen from this figure that the modification resulted in more consistent (less variable) results from top to bottom specimen. However, it can also be seen that the retained stiffness values are higher than those from the preliminary SATS test, indicating a much reduced damage.

It is clear that the inserted membrane successfully diverted the dripping water, as expected, preventing major water contact to the specimens. Assuming that the specimens would have much less saturation (especially the upper specimens), the results indicated that the condensed, dripping water is the major factor of saturation

variation observed in Section 4.3.2. It also indicated that the retained saturation variation is the key factor for the retained stiffness variation observed in Section 4.2.

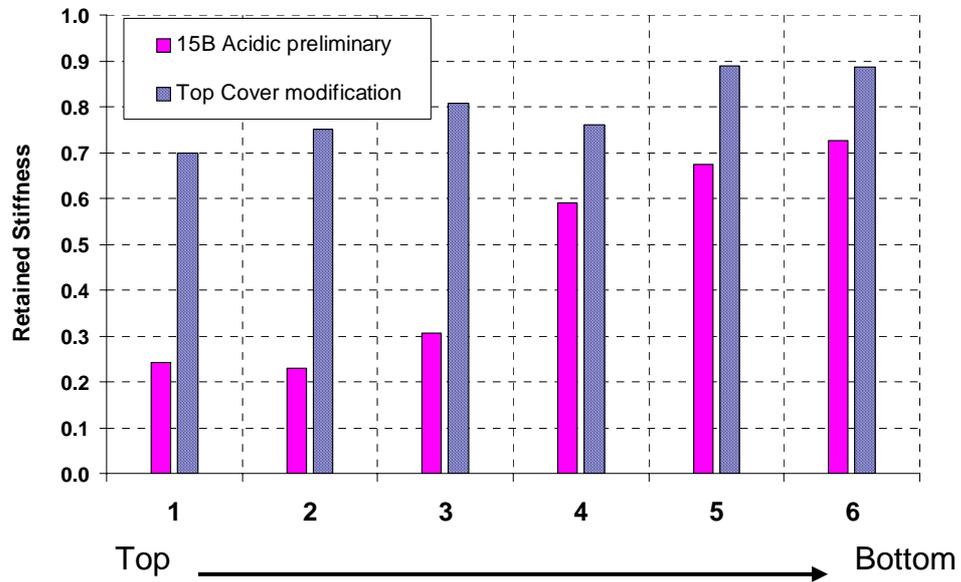


Figure 4.10 – Results from the SATS test when the top cover is introduced.

4.5 Individual Specimen Wrapping

The objective of this modification was to ensure that the quantity of water inside each specimen remained similar during the test. Each specimen was wrapped with several layers of plastic film, immediately after the vacuum saturation procedure. The wrapped specimens were placed in the vessel in the normal way (see Figure 4.11) and the same protocol was applied. This time the saturation was measured again after the test protocol (referred to as the 'retained saturation').

Figure 4.11 shows the testing setup and the retained saturation. It can also be seen from this figure that most of the retained saturations are low compared to the unmodified results (see Figure 4.7). It is clear that the wrapping film could not retain the initially absorbed water (around 50 % saturation). It is assumed that the high pressure made small punctures in the film, and thus the punctured film could not prevent water evaporation from the specimens, while preventing condensed water from being re-absorbed. Figure 4.12 shows the retained stiffness versus retained

saturation for this modification compared to the results from the preliminary SATS (from Figure 4.7). It is clear from this figure that the retained saturation range is much reduced. However, it is interesting to note that a very similar pattern of the retained saturation and the retained stiffness relationship can be found.

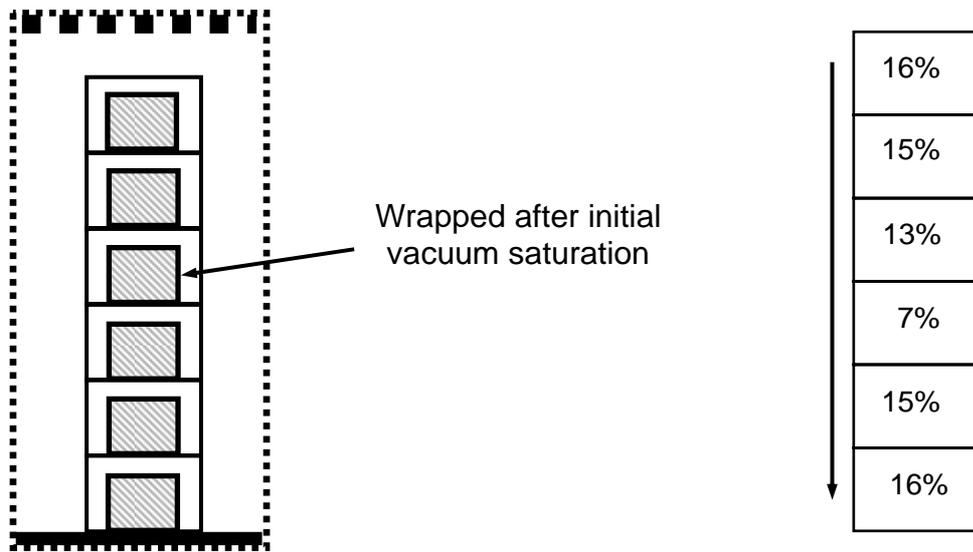


Figure 4.11 – Setup for the individually wrapped specimens, and the retained saturations.

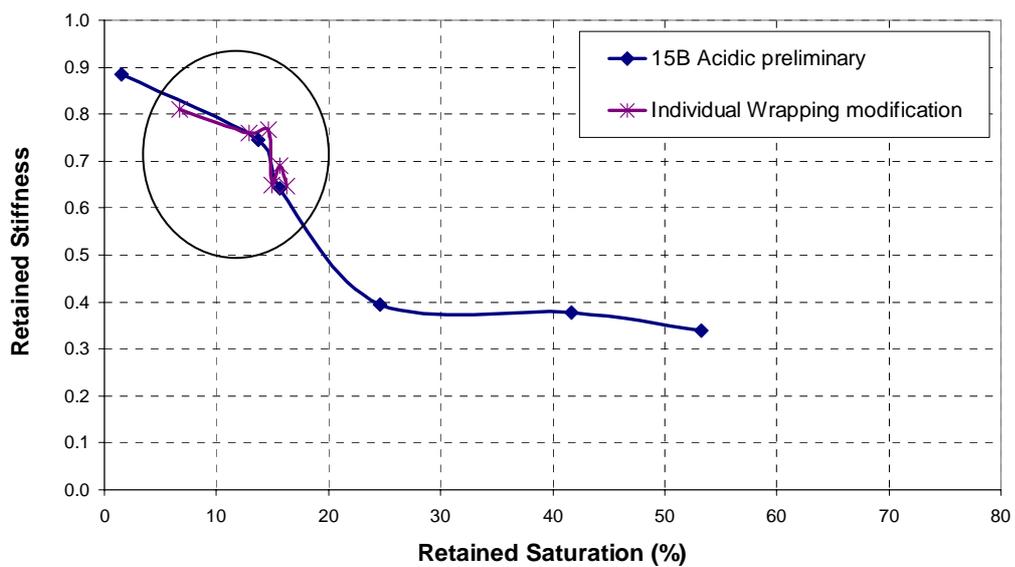


Figure 4.12 – Results from the individual wrapping trial.

4.6 Individual Container for Each Specimen

As with the previous modification, the objective was to ensure that the quantity of water inside each specimen remained similar during the test. In this set of tests open containers (see Figure 4.13) were used instead of wrapping film.

Initially, the water from each specimen will be retained in each container but eventually will evaporate and condense at the top the vessel. From Figure 4.14, it is clear that a certain amount of dripping water will gather in the top container while the rest will drop directly to the bottom of the vessel. It is also likely that a certain amount of water could reach the lower containers following the outer surface of the upper containers, due to the surface tension of water, resulting in an irregular saturation distribution as seen in Figure 4.14.



Figure 4.13 – Specimen in the open container.

Results from this modification are shown in Figure 4.15. It can be seen from this figure that the results show a wider range and more irregular distribution of retained saturation. Again a similar relationship between retained saturation and retained stiffness was found.

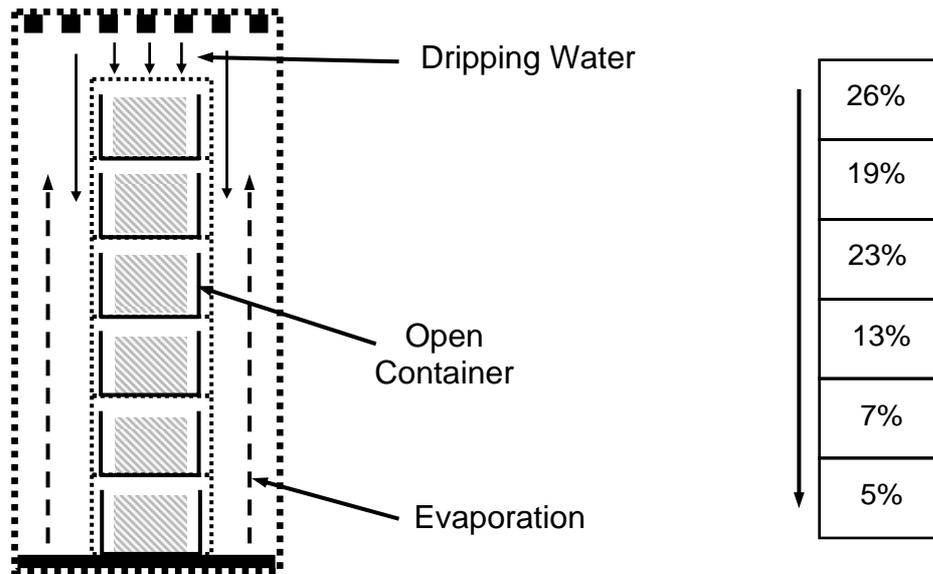


Figure 4.14 – Schematic diagram of water circulation with the open containers, and retained saturation.

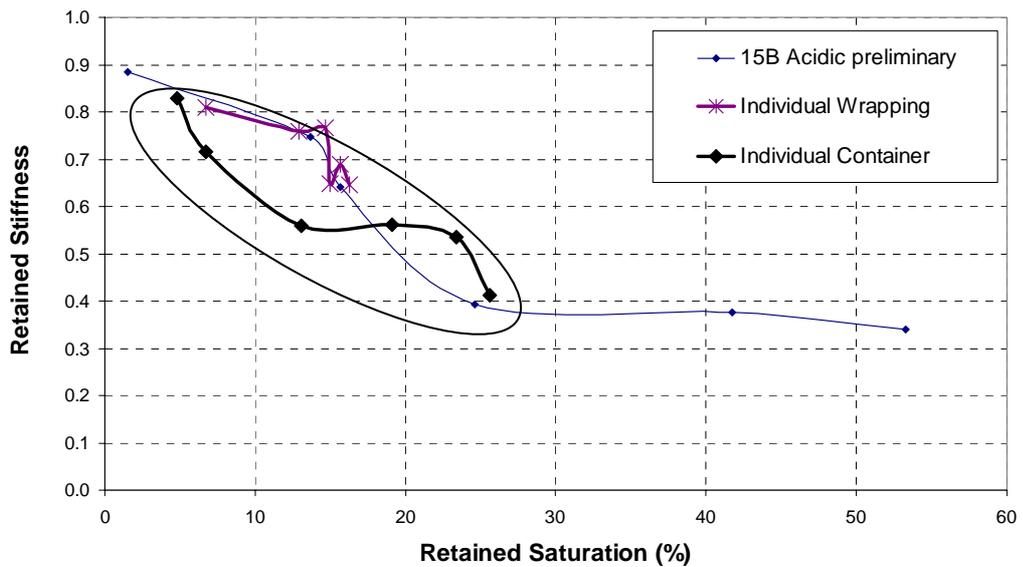


Figure 4.15 – Results from the individual container trial.

4.7 Middle Collar in Tray

The objective of this modification was to increase the saturation levels of the lower specimens (positions 4, 5 and 6). A water collecting collar, comprising an air permeable membrane as described in Section 4.4, was inserted in the middle of the specimen tray as shown in Figure 4.16. It was anticipated that the collar would

collect the dripping water of around the specimen, otherwise directly to the bottom the vessel, to supply it to the specimens in the lower half of the vessel.

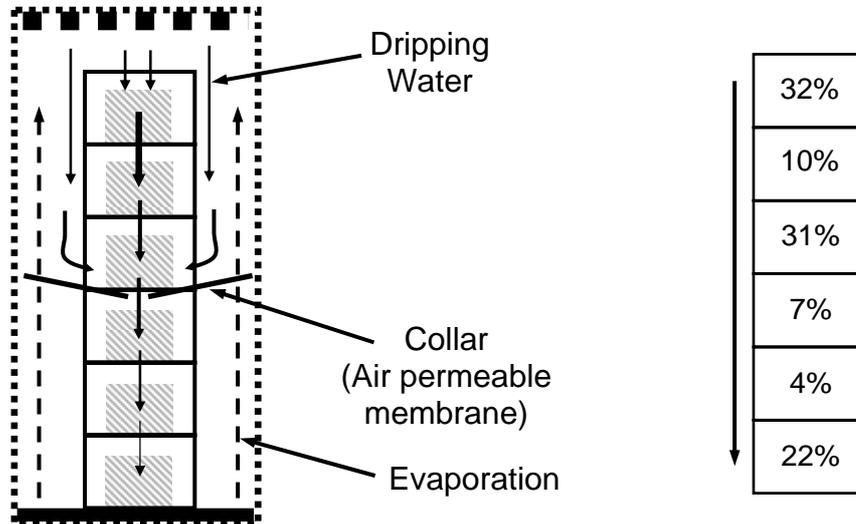


Figure 4.16 – Schematic diagram of water circulation with the middle collar introduced.

It can be seen from Figure 4.16 that the distribution of retained saturation is quite irregular. The reason for this is not clear but is likely to be due to the design/manufacture of the collar. The retained stiffnesses are shown in Figure 4.17. It can be seen from this figure that, although there is an irregular distribution of retained saturation, the correlation between the retained stiffnesses and retained saturation is similar to those observed before.

4.8 Semi Immersion SATS

To investigate damage caused by even higher retained saturation levels, additional water was added to the vessel to immerse the bottom two specimens (see Figure 4.18).

Five specimens were used to simplify the water level control (otherwise one specimen would be partially immersed). The results indicated that, overall, the specimens showed higher retained saturations, with the immersed specimens

showing the highest saturation and the lowest retained stiffness (most damaged), as expected. The results are presented in Figure 4.19 and, although the data points are clearly shifted to right side of the graph (to the higher retained saturation range), show a similar trend for the retained saturation versus retained stiffness relationship compared to those found from the previous modifications.

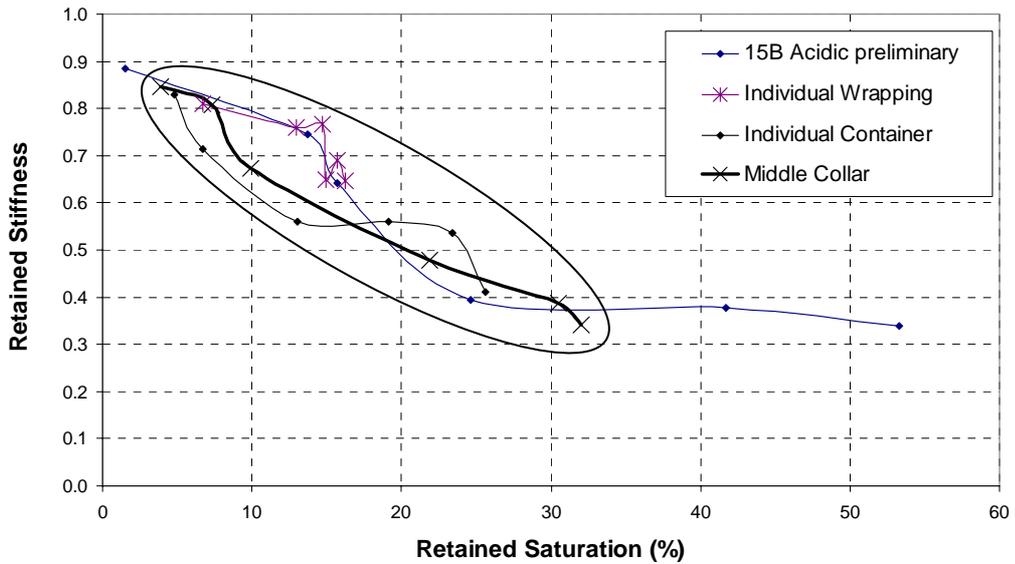


Figure 4.17 – Results from the middle collar trial.

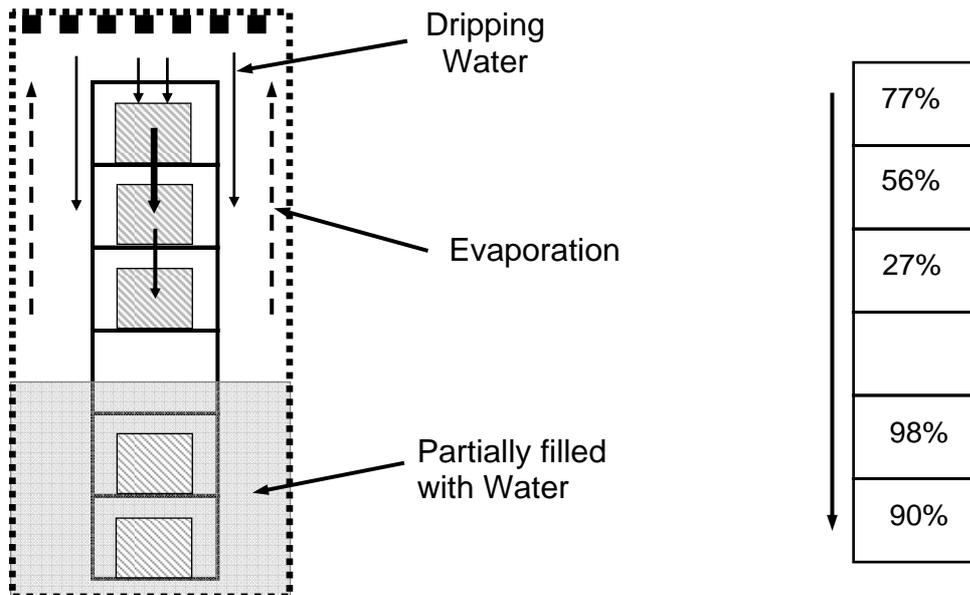


Figure 4.18 – Schematic diagram of the semi immersion SATS setup.

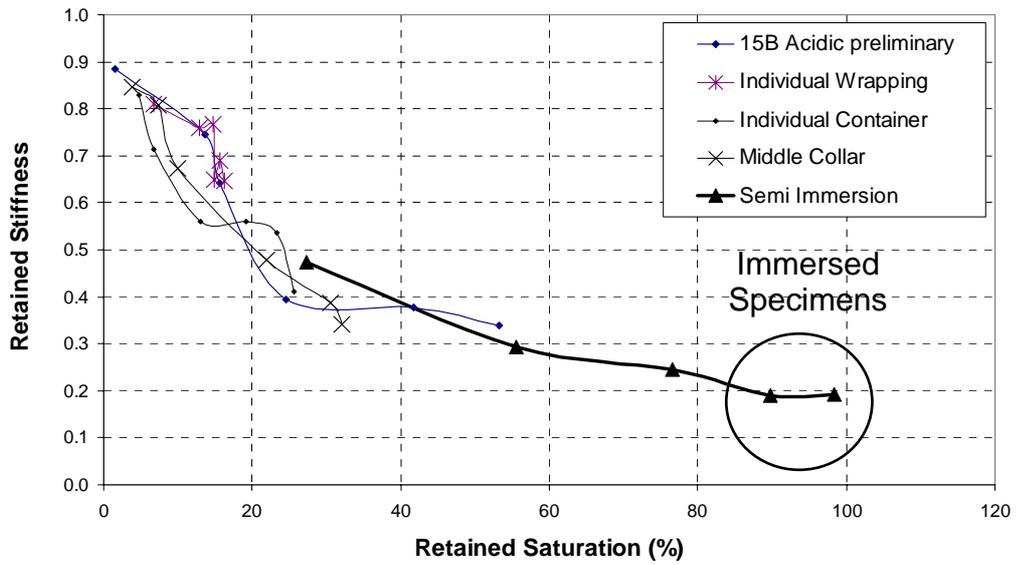


Figure 4.19 – Results from the semi immersion SATS.

4.9 Semi Immersion SATS #2

This is similar to the previous modification, but only the bottom specimen was immersed as shown in Figure 4.20. The objective of this modification was to produce a wider range of retained saturations.

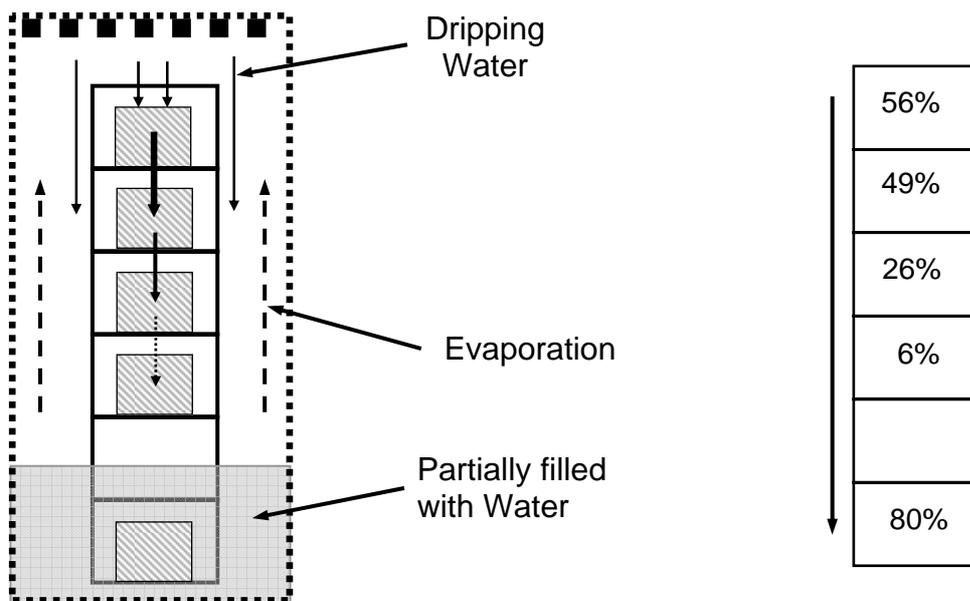


Figure 4.20 – Schematic diagram of the semi immersion SATS setup #2.

Figure 4.21 clearly presents a similar trend of retained saturation versus retained stiffness relation as seen before, with a wider range of retained saturations (from a very dry to complete immersion) compared to those from the previous setup (see Section 4.8).

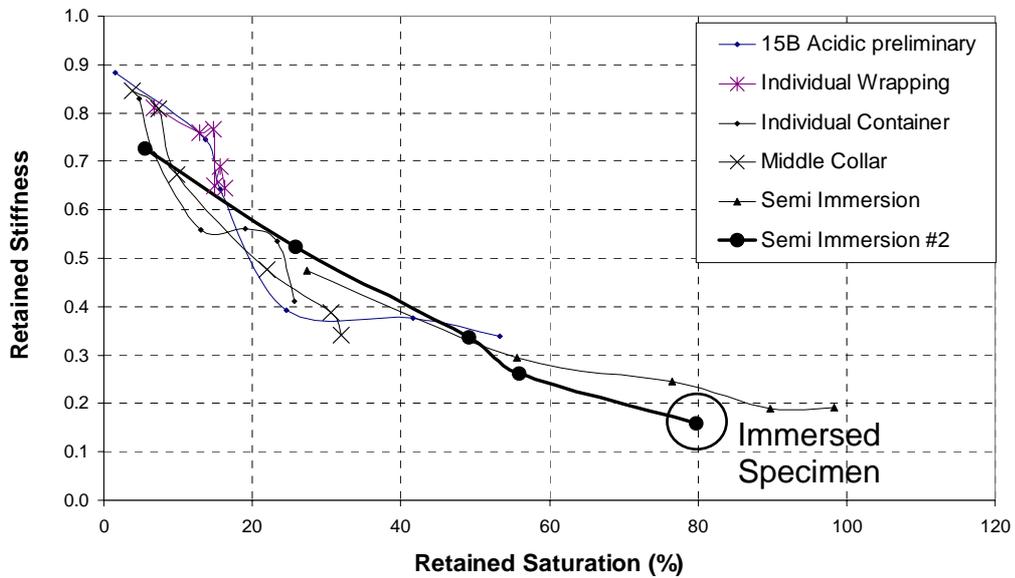


Figure 4.21 – Results from the semi immersion SATS #2.

4.10 Summary and Conclusions

A preliminary version of moisture and ageing combined test procedure (based on HiPAT protocol) has been developed to rapidly assess the durability of typical HMB materials. The procedure (referred to as ‘preliminary SATS’) was performed on laboratory manufactured 100 mm diameter cores. The variability investigation using nominally identical sets of specimens revealed that the test conditions vary dependent on the specimen position within the vessel and the key factor is believed to be the retained saturation.

Various modifications were tried to optimise the SATS protocol. A cover on top of the specimen tray prevented the condensed water directly dripping on to the top specimen. This clearly reduced the retained saturation variation but also ‘protected’ the specimens from moisture damage. Trials to confine the initial saturation water in each specimen using wrapping film or open containers were not successful. The

wrapping film could not hold the high pressure, and the retained water in the individual containers eventually evaporated. A trial to insert a water collecting collar in the middle of the specimen tray was also not successful, and it appeared that a much more complicated design and accurate manufacture of the collar shape would be required to achieve success.

An alternative approach was tried involving adding more water to the vessel. The bottom specimen was completely immersed and no other modification applied. The results showed a more distinctive retained saturation variation from the completely immersed specimen (nearly 100% retained saturation) to a virtually dry specimen immediately above the water.

It is interesting to note that the results from all the various modifications generally fitted in a single retained saturation/retained stiffness correlation curve. The results therefore suggested that the correlation of retained saturation and retained stiffness, plotted in a single curve, could be an ideal parameter to indicate the ageing and moisture related durability of a bituminous mixture.

It is concluded that the retained saturation is the key factor to control the damage created by the SATS protocol. It is also found that a certain level of retained saturation variation depending on the specimen location is inevitable and therefore need to be accepted as a test parameter.

Considering the practicability and repeatability of the protocol, the partially immersed SATS trial (see Section 4.9) appeared to be the most suitable approach, as it produces from a very dry specimen to a completely immersed specimen with gradually saturated specimens between them. Therefore, the partially immersed SATS as described in Section 4.9 was chosen as the main test protocol for this study, and referred to as 'standard SATS' (or SATS) protocol for the rest part of this thesis.

Chapter 5. SATS Durability Test

5.1 Materials

The specification for 28 mm DBM base material [BS 4987: Part 1, 2001] was used to produce a series of mixtures to replicate the material used in the HMB trial site described in Chapter 1, and to investigate the influence of various mixture components on the test results, and its variability. The following sections describe the details of materials, testing protocols and analysis of the results.

5.1.1 Binders

Three 15 Penetration grade binders (referred to as 15B, 15D and 15E) and a 50 Penetration grade binder (50C) were used (4% Binder Content). The details of the various characteristics of these binders are presented in Appendix C.

5.1.2 Aggregates

A typical acidic aggregate (referred to as 'Acidic') and a typical alkaline aggregate (referred to as 'Basic') were used. The Acidic aggregate source was that the same as that used in the HMB trial site described in Chapter 1. Figure 5.1 shows the gradations of both aggregates compared against the 28mm DBM base specification [BS 4987: Part 1, 2001].

5.2 Sample Manufacture

5.2.1 Maximum Density

Rice density test was performed to determine the maximum densities of the Acidic and Basic mixtures [BSEN 12697: Part 5, 2002]. Values of 2.493 (Mg/m³) and 2.479 (Mg/m³) were obtained for the Acidic and Basic mixtures respectively.

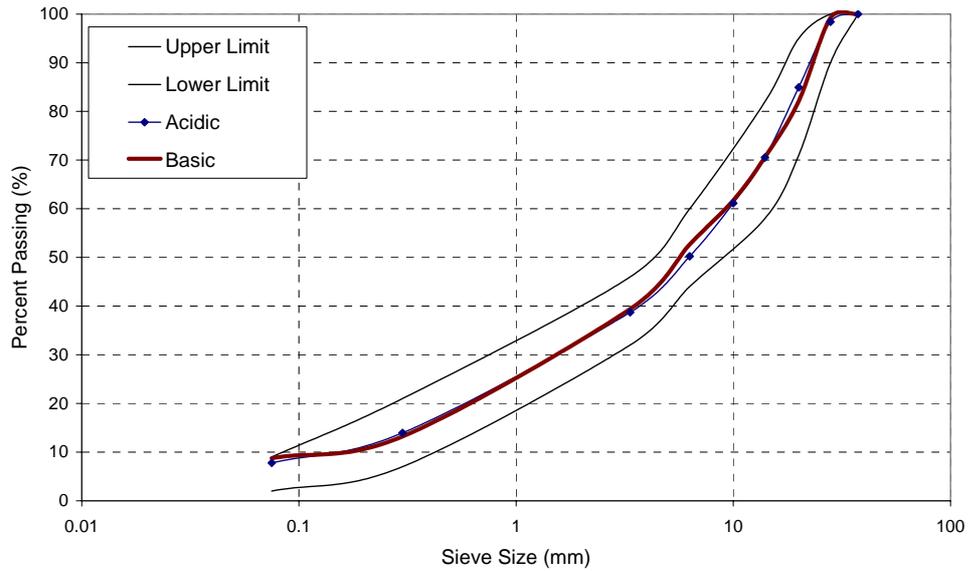


Figure 5.1 – Gradations of Acidic and Basic aggregates (28 mm DBM).

5.2.2 Specimen preparation

The mixtures were compacted using a roller compactor (see Figure 5.2) to produce a slab (305 mm × 305 mm × 100 mm). In this study no deliberate attempts were made, such as an extra oven storage process prior to compaction, to short-term age the loose mixture. The target air void content of 8% was controlled (this was the typical air void content measured from the HMB trial site), based on the determined maximum density. Four cylindrical specimens were cored (ϕ 100 mm) from the compacted slab and trimmed to the required thickness of 60 mm. The sealed bulk density and air void content of each specimen were calculated using Equations 5.1 and 5.2, and then the specimens were stored in a cold room (5°C) for future use. The air voids results for individual specimens are presented in Appendix E.

$$\rho = \frac{M_{au} \times \rho_w}{M_{ac} - M_{wc} - \left(\frac{M_{ac} - M_{au}}{S_{gf}} \right)} \quad (5.1)$$

where;

ρ = Bulk density of the specimen

M_{au} = Mass of the specimen in air uncoated (kg)

- M_{ac} = Mass of the specimen in air coated with aluminium foil (kg)
 M_{wc} = Mass of the specimen in water coated with aluminium foil (kg)
 S_{gf} = Density of aluminium foil (1650 kg/m³)
 ρ_w = Density of Water

$$V_v = \left(1 - \frac{\rho}{\rho_{\max}} \right) \times 100 \quad (5.2)$$

where;

- V_v = Air voids of the specimen
 ρ_{\max} = Maximum density of the specimen
 ρ = Bulk density of the specimen



Figure 5.2 – Roller slab compactor.

5.3 SATS Test Protocol (Standard SATS)

The initial stiffness modulus of the specimens at 20°C was measured using the ITSM test under standard conditions [BS DD 213, 1993], after which the specimens were partially saturated with water using a vacuum pump under a negative pressure (suction) of 68 kPa for 30 minutes. The partially saturated specimens were then placed into the pressure vessel, which contained sufficient water to fully immerse the bottom specimen (see Figure 5.3), and were subjected to the HiPAT conditioning protocol (a temperature of 85°C and a pressure of 2.1 MPa for 65 hours). At the end of the conditioning period, the saturation was again measured (referred to as the 'retained saturation'); the specimens were then allowed to cool and conditioned for the ITSM test at 20°C (conditioned stiffness). Finally, the ratio of conditioned stiffness modulus to initial stiffness modulus (referred to as the 'retained stiffness') was determined for each of the specimens. A more detailed SATS protocol is presented in Appendix D. The retained stiffness from each specimen was plotted against the retained saturation of the same specimen, which, as explained in Chapter 4 (see Section 4.10), is used as the durability parameter of the mixture for this research.

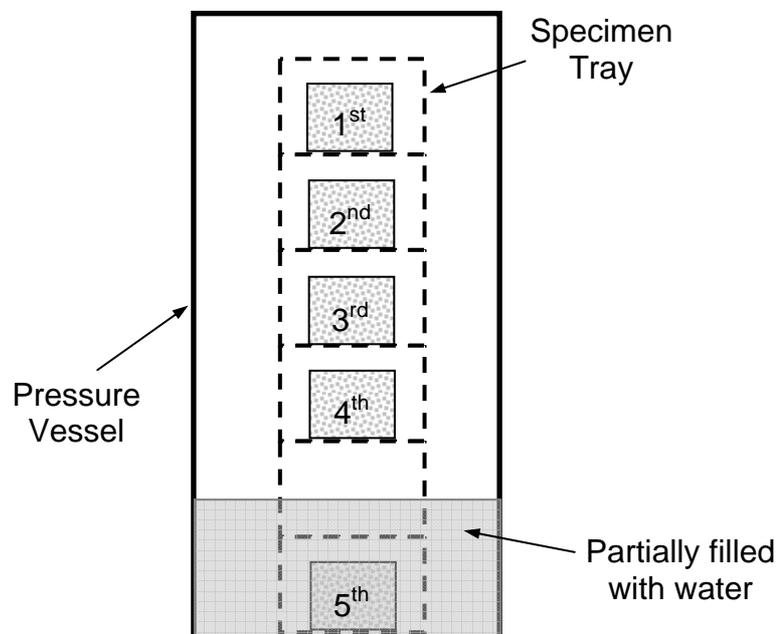


Figure 5.3 – Schematic diagram of SATS setup.

5.4 SATS Test Series

Five specimens were used for one SATS test. Retained stiffness and retained saturation after the SATS protocol were measured for each test specimen and plotted on a graph. Figure 5.4 shows typical results from specimens from the 15B binder with Acidic aggregate (15B Acidic). Normally, two repetitions were carried out for each mixture to produce 10 data points per graph.

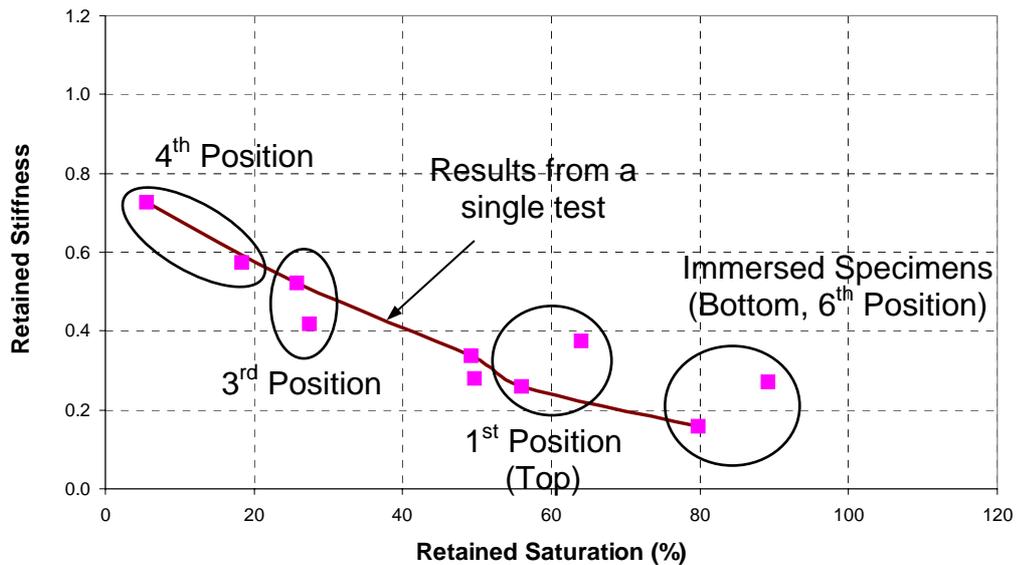


Figure 5.4 – Typical results of a test set from the SATS protocol.

The first series of tests was on the Acidic aggregate mixtures with four binders (15B, 15D, 15E and 50C), and the results are presented in Figure 5.5. The mixtures were nominally identical except for the binders. The second series of tests was on nominally identical Basic aggregate mixtures with the same range of binders. The results are presented in Figure 5.6 in the same manner and compared to those of the Acidic mixture series (from Figure 5.5).

It can be seen from Figure 5.5 that, for the Acidic specimens, the general trend is for the retained stiffness to decrease as the retained saturation increases. At high retained saturations the retained stiffness is as low as 0.2, indicating that significant damage was induced in the specimen by the conditioning procedure. Most specimens from the 15 Penetration grade binder materials commonly showed a

retained saturation range between 10 and 90%, and a retained stiffness range between 0.6 and 0.2.

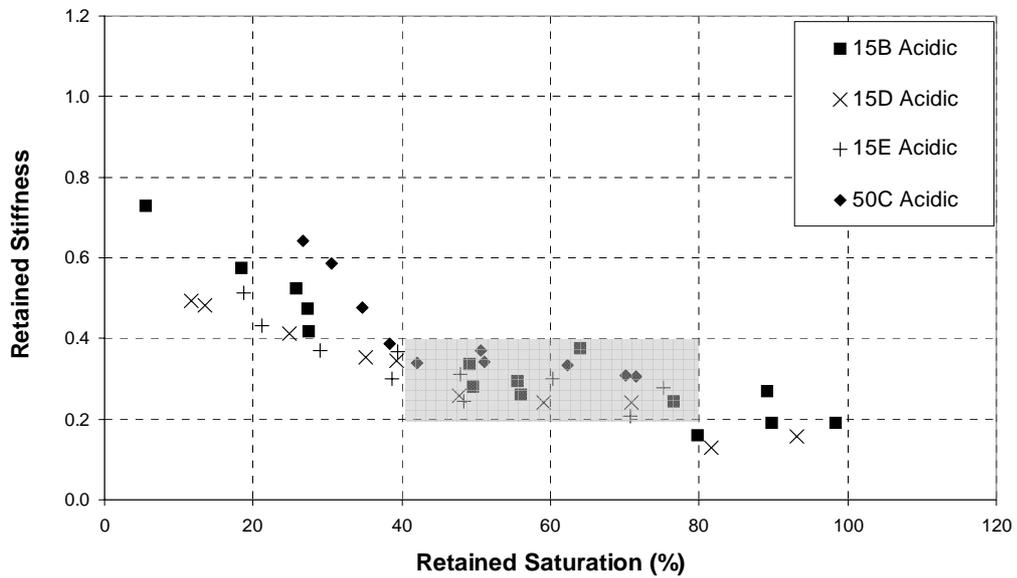


Figure 5.5 – Results from the Acidic aggregate mixture SATS series.

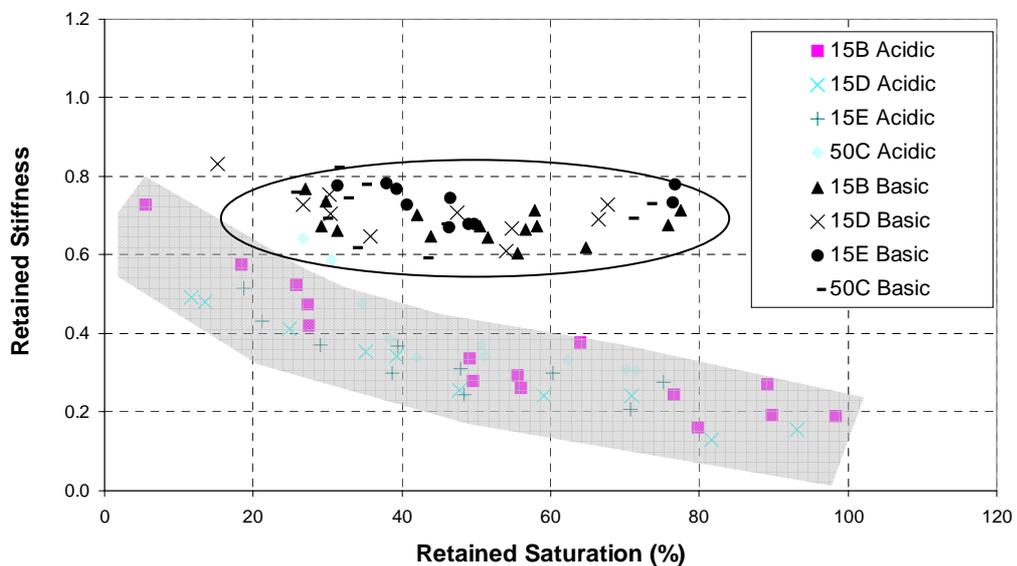


Figure 5.6 – Results from the Basic aggregate mixture SATS series.

From Figure 5.6, it is clear that the retained saturation range was much reduced (25 ~ 75%) compared to that of the Acidic mixtures, indicating that the specimens typically absorbed less amount of water than the Acidic mixtures during the process. Results for the Basic specimens show a smaller reduction in retained stiffness that

levels off at approximately 0.7 as the retained saturation is increased. For a comparison, the retained stiffness of 15B Acidic and 15B Basic at a same retained saturation of 40% are approximately 0.4 and 0.7 respectively. This indicates that significantly less damage occurs in the Basic mixture compared to the Acidic mixture. The relative performance differences between the binders were found to be insignificant compared to the effect of the aggregates.

5.5 Test Variability Investigation

To investigate variability of the SATS test, a total of six repetitions were performed on the Acidic and Basic mixtures with 15B binder. Two sets of results of both mixtures were from the main test series described in the previous section, and four more repetitions were additionally performed. Results are presented in Figure 5.7.

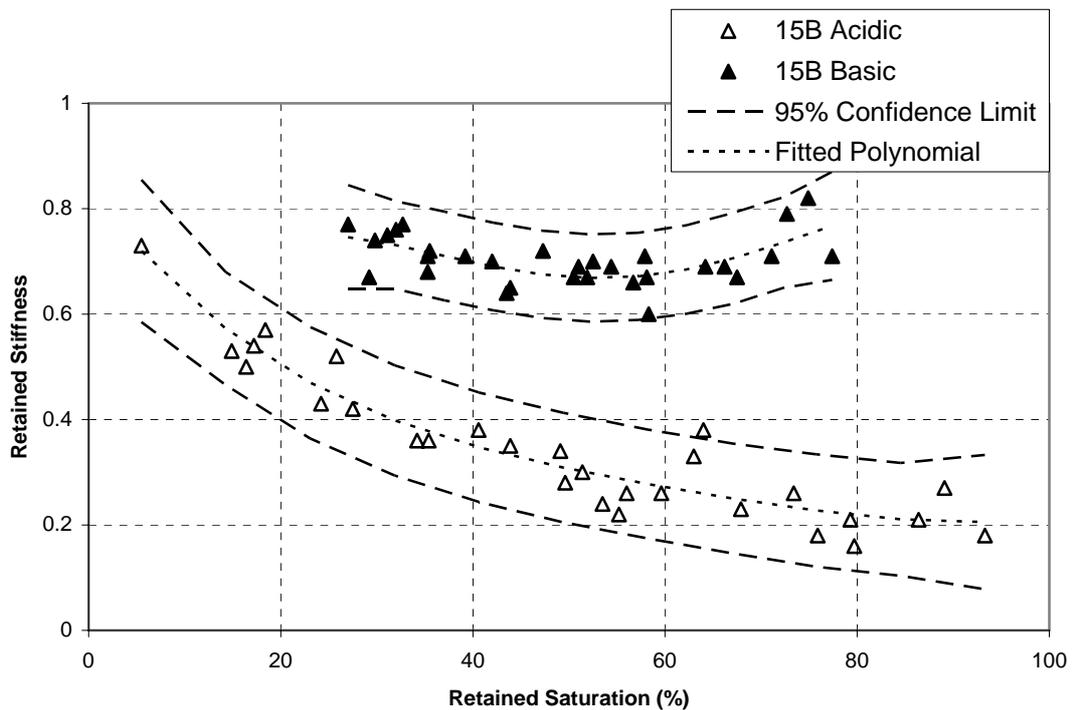


Figure 5.7 – Test variability (15B Acidic and 15B Basic).

5.5.1 Retained Stiffness

It is not straightforward to assess the variability in the results shown in Figure 5.7 since neither the retained stiffnesses nor the retained saturations are controlled. The easiest approach is to fit (in a least squares sense) a non-linear regression model to the data and determine confidence limits for the fitted curve. The Matlab Curve Fitting Toolbox [Anon, 2002] was used for this purpose and a confidence level of 95% was chosen. A number of non-linear curves were tried and it was found that a satisfactory fit to the data was achieved using a 4th order polynomial (dashed lines in Figure 5.7). The fitted constants and the R^2 value for the fit are given in Table 5.1. The R^2 for the 15B Acidic is significantly higher (0.90) than that for the 15B Basic (0.42) probably because of a greater range in the retained saturation (x – axis).

Table 5.1 – Fitted constants.

| | $y = C_1x^4 + C_2x^3 + C_3x^2 + C_4x + C_5$ | | | | | |
|------------|---|----------------------|----------------------|----------------------|----------------------|----------------------|
| | C₁ | C₂ | C₃ | C₄ | C₅ | R² |
| 15B Basic | -8.83e-8 | 1.98e-5 | -1.46e-3 | 4.02e-2 | 3.78e-1 | 0.42 |
| 15B Acidic | 1.84e-8 | -4.66e-6 | 4.65e-4 | -2.46e-2 | 8.42e-1 | 0.90 |

Figure 5.8 shows the SATS main test series results (from Figure 5.6) fitted in the 95% confidence limits calculated from the above six repetitions of 15B Acidic and 15B Basic (from Figure 5.7). It can be seen that, since most of the results fall inside the confidence limits determined for mixtures containing 15B binder only, it was not possible to statistically distinguish the performance of the materials containing different binders used in this research.

5.5.2 Retained Saturation

Figure 5.9 shows a graph of retained saturation versus specimen position in the test vessel for the above repetitions (average values from the six repeats are shown by the solid lines). Means and coefficient of variations (CoV) are summarised in Table 5.2.

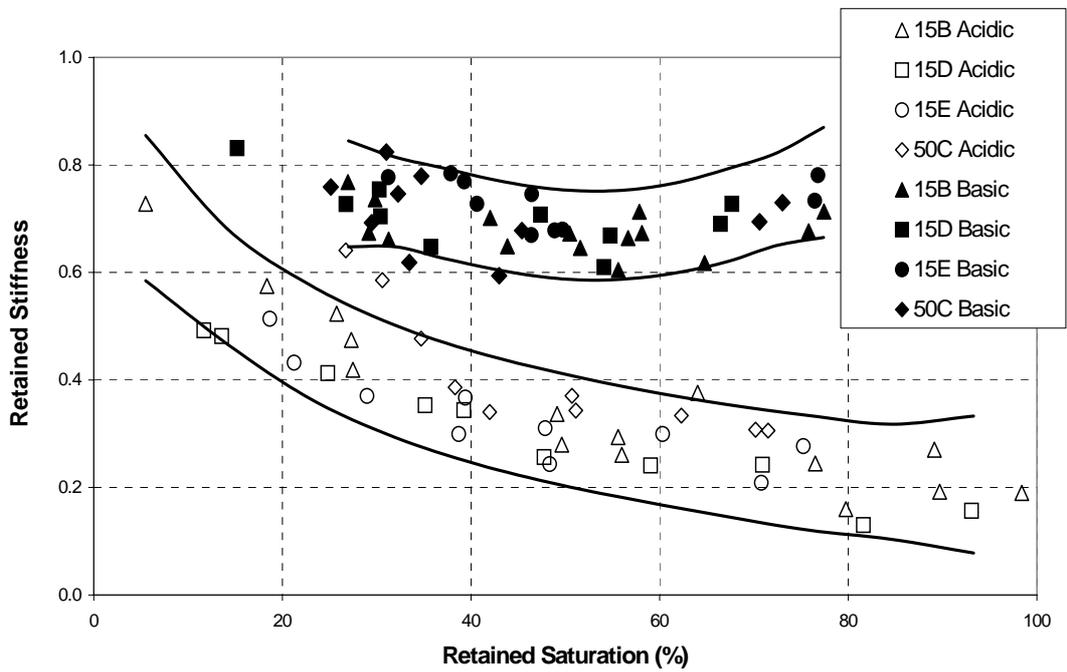


Figure 5.8 – Results from the main test series and the 95% confidence limits

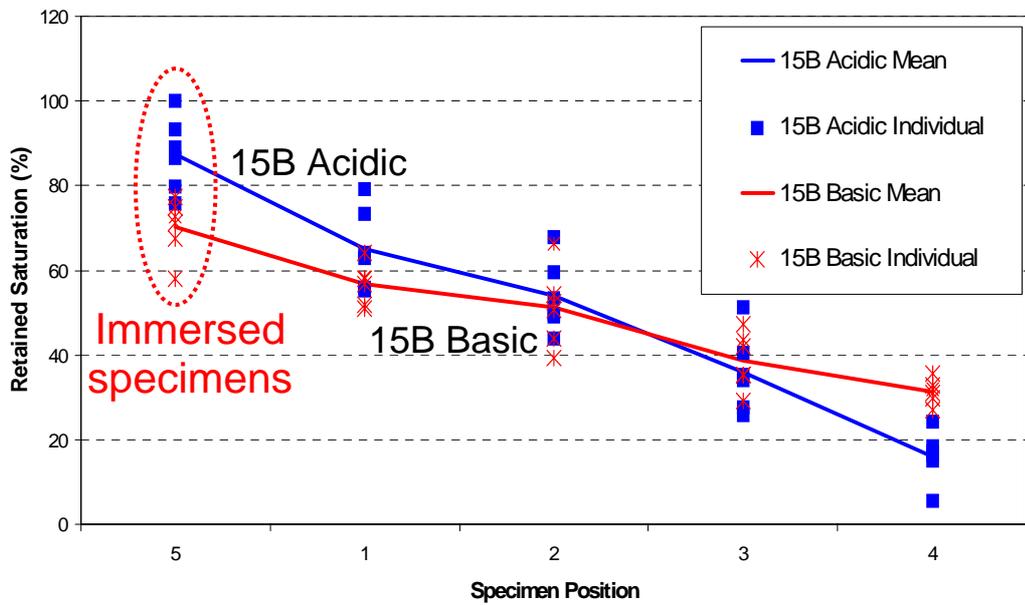


Figure 5.9 – Retained saturation at each specimen position.

It can be seen from this figure that, apart from the immersed specimen (5th position from Figure 5.3), the general trend for both mixtures is for the retained saturation to decrease for specimens that are located further down inside the pressure vessel. It can also be seen from this figure that the retained saturation for the specimens above the water level ranges from 5 to 80%. To investigate the reason for this variation it is necessary to investigate the conditions inside the pressure vessel.

Table 5.2 – Mean and CoV of retained saturation.

| Specimen Position | 15B Acidic | | 15B Basic | |
|-------------------|------------|---------|-----------|---------|
| | Mean (%) | CoV (%) | Mean (%) | CoV (%) |
| 1 | 65.2 | 14.7 | 56.7 | 8.5 |
| 2 | 53.9 | 15.6 | 51.1 | 18.2 |
| 3 | 35.8 | 26.1 | 38.8 | 17.2 |
| 4 | 16.1 | 37.9 | 31.4 | 9.1 |
| 5 | 87.4 | 10.1 | 70.3 | 9.9 |

The nominal temperature of the air in the pressure vessel is 85°C. Assuming that there is enough water in the vessel to fully saturate the air, the partial pressure of the vapour (p_g) at this temperature and the specific volume of the vapour (v_g) can be determined from steam tables [Smith et al., 1996] to be 58 kPa and 2.828 m³/kg respectively. Since the vessel is pressurised to a pressure (p) of 2.1 MPa, the partial pressure of the dry air (p_a) is 2.042 MPa ($p - p_g$). The specific humidity (ω) of the air can be calculated from the partial pressures using Equation 5.3 [Eastop and McConkey, 1986]:

$$\omega = \frac{M_g}{M_a} \times \frac{p_g}{p_a} \quad (5.3)$$

where $M_g = 18$, $M_a = 28.96$ are the molecular weights of the water vapour and the dry air respectively [Eastop and McConkey, 1986].

The weight of pressurised dry air (m_a) in the vessel can be calculated using Equation 5.4, which was derived from the ideal gas equation of state [Eastop and McConkey, 1986]:

$$m_a = \frac{P_a V_a M_a}{R_o T} \quad (5.4)$$

where P_a = pressure of dry air, V_a = volume of dry air, M_a = molecular weight of dry air, R_o = ideal gas constant ($8.314 \text{ m}^3 \cdot \text{Pa} \cdot \text{mol}^{-1} \text{K}^{-1}$) and T = air temperature (K), respectively.

As the approximate volume of air in the vessel (volume excluding water in Figure 5.3) is $5.5 \times 10^{-3} \text{ m}^3$ ($\phi 0.15 \text{ m}$, $h = 0.31 \text{ m}$), from Equation 5.4, the weight of pressurised dry air (m_a) in the vessel is calculated to be 109.2 g. The specific humidity inside the chamber is 0.018 (from Equation 5.3) which means that the air inside the pressure vessel contains 0.018 kg of water vapour per kg of dry air. Thus the mass of water vapour in the vessel is calculated to be approximately 1.97 g. Consequently, it can be seen that although the air inside the pressure vessel is fully saturated, the quantity of water in the air is negligible and cannot be accounted for the retained saturations measured from the specimens.

Observations from the test showed that when the pressure vessel was opened after the procedure, there was a significant amount of condensation on the underside of the lid. Further investigations (see Section 4.3.1) revealed that the reason for this was that the temperature on the underside of the top lid was lower than other area. Consequently, during the test it is likely that there is a continuous cycle of moisture condensing on the underside of the top lid and “dripping” onto the top specimen. There is then a cascade effect where progressively smaller amounts of water “drip” onto the specimens below resulting in a decrease in retained saturation level for specimens that are located deeper inside the pressure vessel.

5.6 Summary and Conclusions

The developed SATS (standard SATS) protocol was applied to a series of mixtures including a mixture (15B binder with Acidic aggregates) nominally identical to that used in the HMB trial site. For Acidic aggregate mixture series, at a retained saturation of 40% the retained stiffness modulus was approximately 0.4, which compares favourably with the 60% reduction in stiffness modulus observed from the initial HMB trial site at the TRL (for nominally identical mixtures).

The retained stiffnesses of four Acidic mixtures manufactured with different binders showed a clear and gradual relationship against the retained saturations (i.e. highly saturated specimens showed lower retained stiffness). The immersed specimens commonly showed the lowest retained stiffness with the highest retained saturation, while the top specimen (1st position) showed the lowest retained stiffness and the highest retained saturation amongst the 'non-immersed' specimens (1st ~ 4th positions). The specimens from the 4th position commonly showed the highest retained stiffness (least damaged) and were in a virtually dry condition.

The retained saturation of four Basic mixtures manufactured with different binders showed a similar trend in the retained saturation distribution, with higher retained saturations from the upper specimens and the highest values from the immersed specimens, although relatively lower than those from the Acidic mixtures. However, the retained stiffness remains at an approximate constant value of 0.7 over a wide range of retained saturations (between 15 and 80%) indicating a less moisture sensitivity.

The most distinctive behaviour was found from the immersed specimens which generally showed a better performance (higher retained stiffness) than specimens from the 1st or 2nd positions, even though the retained saturations were always higher. Thus the most damaged Basic specimens were commonly found from the top specimens for this set of tests.

An investigation of test variability showed a reasonably good repeatability with the mixtures selected. No statistically clear difference was found from nominally

identical mixtures that contained binders from different sources, since most of the data points fall inside the confidence limits determined from variability investigation using 15B Acidic mixtures (see Section 5.5.1).

The retained saturation variation at each specimen position was investigated and generally showed a similar and acceptable range of variation. The Acidic mixture showed both the lowest and highest retained saturations and this is likely to be due to differences in the internal air voids structure between the two mixtures. Further discussions to this aspect are presented in Chapter 7 (see Section 7.6).

Difference in retained saturations (measured after the test) depending on the position of the specimen in the pressure vessel is considered to be beneficial since the sensitivity of a material to the variable amount of moisture could be observed from the procedure.

It has been generally concluded that, for the mixture range investigated and described in this chapter, the developed protocol (standard SATS) successfully discriminates the durability of mixtures with respect to combined ageing and moisture damage, on the basis of a measured reduction in stiffness similar to that observed in the original HMB trial site material.

Chapter 6. Mixture Variability

6.1 Introduction

The developed SATS (standard SATS) test was applied on a wider range of mixture conditions. The first series of tests was on the nominally identical Acidic and Basic mixtures as in Section 5.4, but with certain additives (i.e. a Hydrated Lime Filler and an Anti-Stripping Agent). The second series of tests was to investigate the mixture volumetric variability. Using the same materials used in Chapter 5 a series of specimens was manufactured to have a lower air void content ($\approx 4\%$), while keeping the same binder content (4%), to produce a 'more fully compacted' mixture. Also another series of specimens was manufactured with a higher binder content (5%), while keeping the air voids in a similar range ($\approx 8\%$), to produce a 'richer' mixture. Finally, a set of 14 mm EME (EME 2) specimens ($AV = 4\%$ and $BC = 5.75\%$) was manufactured using the 15E binder and the Basic aggregate used in Chapter 5.

6.2 Materials

The identical types and range of materials (binders and aggregates) were used. The Basic aggregate used for the 14 mm EME trial (with 15E binder) was also the same source but the grading was controlled according to its specification [Nunn and Smith, 1994].

6.2.1 Hydrated Lime Filler

A hydrated lime filler (HLF), chemically known as calcium hydroxide ($\text{Ca}(\text{OH})_2$) [Parker, 1946], was used as a primary additive to both aggregates. The hydrated lime filler was added to the selected mixtures (2% of aggregate by weight) as a replacement for the natural rock filler.

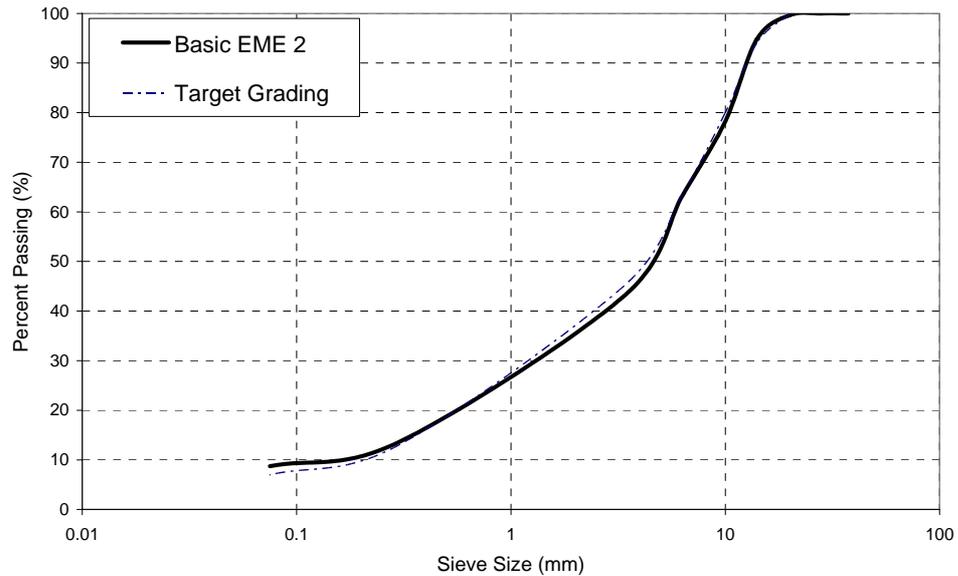


Figure 6.1 – Gradations of Basic aggregate for 14 mm EME (EME 2).

6.2.2 Anti-Stripping Agent

An anti-stripping agent was used. The agent was added to selected mixtures (0.5% of binder content by weight) as an additive to the binder.

6.3 SATS Test Series (Additive Investigation)

The SATS protocol was applied as described in Chapter 5 (also see Appendix D). A series of specimens was manufactured, using the identical materials and specifications to those used in Chapter 5, but with Hydrated Lime Filler (HLF) or Anti stripping agent added as described in Section 6.2. The results of Acidic mixtures with HLF are presented in Figure 6.2 and compared to those of the Acidic and Basic mixture series from Chapter 5.

It can be seen from this figure that the Acidic with HLF mixtures typically showed superior performance compared to the results of the Acidic control mixtures (around 160% increase in retained stiffness) as well as the results of the Basic control mixtures (around 20% increase in retained stiffness). The retained stiffness was in the range between 0.7 and 0.9 and the retained saturation was between 20 and 60%. Again the relative performance of the binders was found to be insignificant.

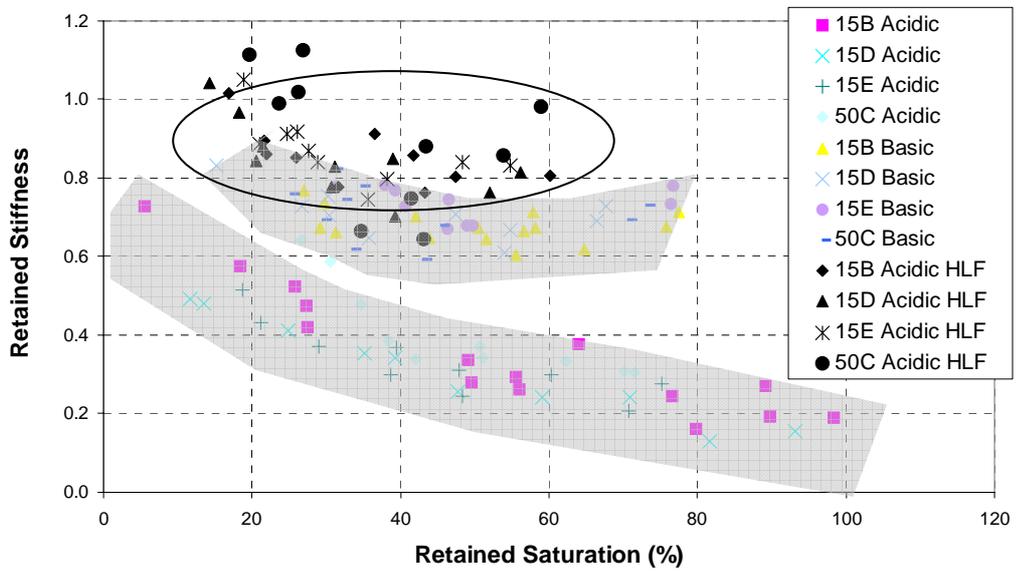


Figure 6.2 – Results from the Acidic mixtures with HLF mixture series.

The results of 15B Basic with HLF are presented in Figure 6.3 and, as no significant influence of binders had been found in the previous work, no further mixtures with different binders were tested.

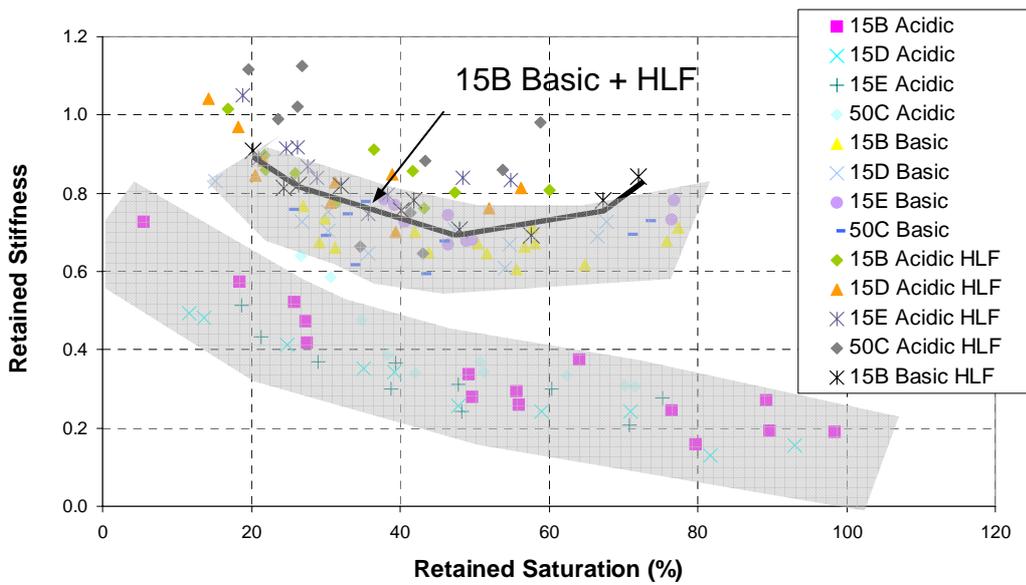


Figure 6.3 – Results from the 15B Basic with HLF mixture.

From Figure 6.3, the 15B Basic with HLF mixture shows slightly better performance, though this is thought to be insignificant. It is assumed that, as the Basic aggregate is already an alkaline aggregate, the hydrated lime filler could not further improve the interaction between the aggregate and binder.

The results of 15B Acidic with the anti-stripping agent are presented in Figure 6.4 and compared to those of the Acidic and Basic control mixtures. It can be seen from Figure 6.4 that the agent marginally improved the performance of the Acidic mixture, but the effect was much less significant than that of the hydrated lime filler (see Figure 6.2).

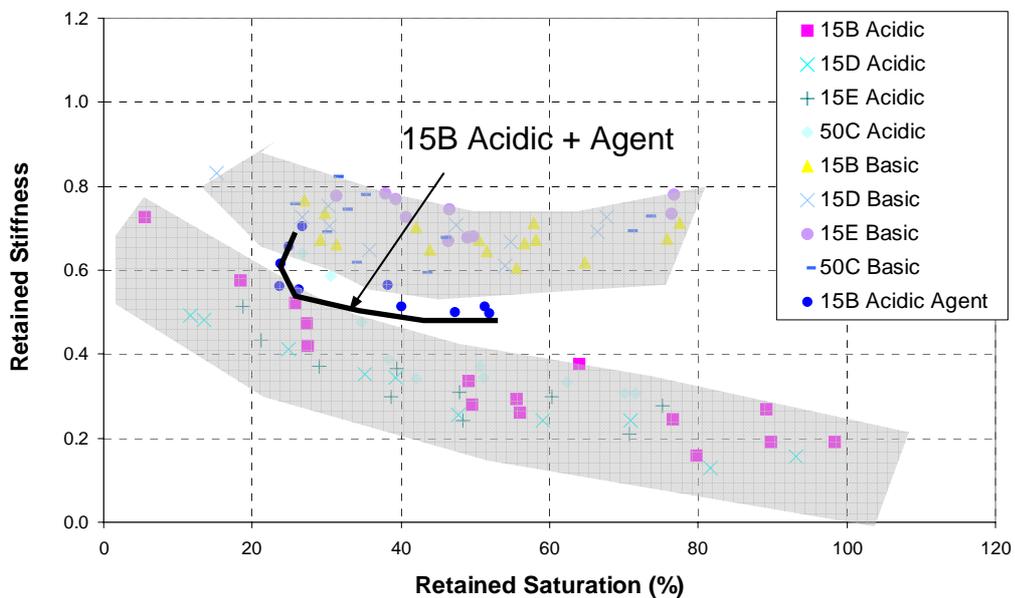


Figure 6.4 – Results of the 15B Acidic mixture with anti-stripping agent.

6.4 SATS Test Series (Mixture Variability)

Using the identical binder (15B) and aggregates (Acidic and Basic) used in Chapter 5, a series of specimens was manufactured to have a lower air void content ($\approx 4\%$), while keeping the same binder content (4%), to produce a ‘more fully compacted’ mixture. Also another series of specimens was manufactured with a higher binder content (5%), while keeping the air voids in a similar range ($\approx 8\%$), to produce a ‘richer’ mixture.

The SATS test results of 15B Acidic are presented in Figure 6.5 and compared to those of the control mixture (AV = 8% and BC = 4%) from the main test series. Results of 15B Basic mixtures are presented in Figure 6.6.

From Figure 6.5, the 15B Acidic mixture with low air voids (AV = 4% and BC = 4%) show virtually no change in its retained stiffness indicating similar moisture sensitivity to the control mixtures (AV = 8% and BC = 4%). However, the mixture with high binder content (AV = 8% and BC = 5%) showed an increase in the retained stiffness (approximately 200% increase in retained stiffness at 40% of retained saturation) indicating an improved moisture sensitivity. The 15B Basic mixtures with low air voids (AV = 4% and BC = 4%) and with high binder content (AV = 8% and BC = 5%), from Figure 6.6, commonly show a relatively poor performance than the control mixture (AV = 8% and BC = 4%) showing slightly reduced retained stiffness range (0.5 ~ 0.7).

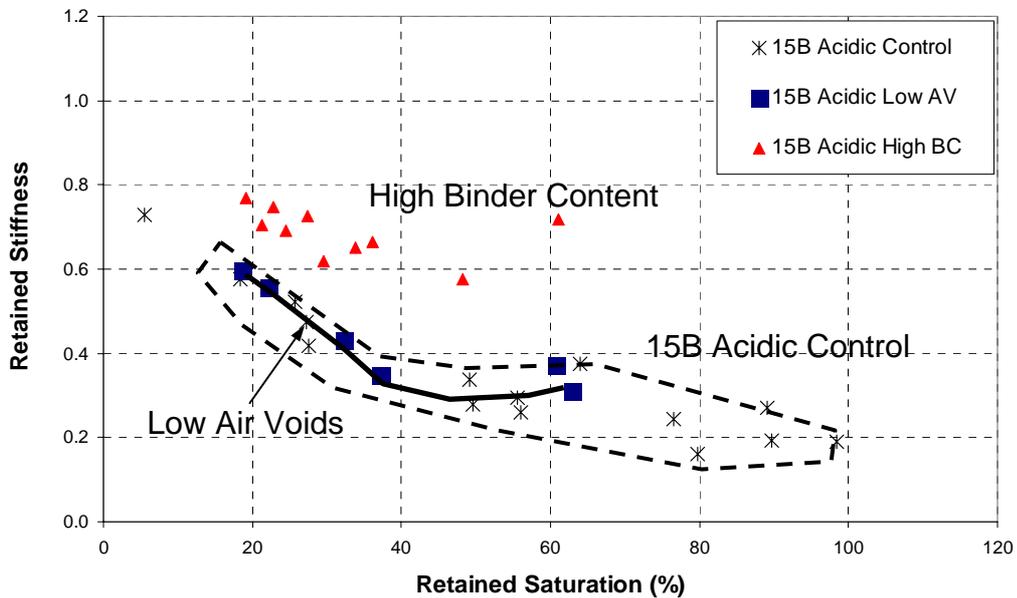


Figure 6.5 – Mixture variability investigation (15B Acidic).

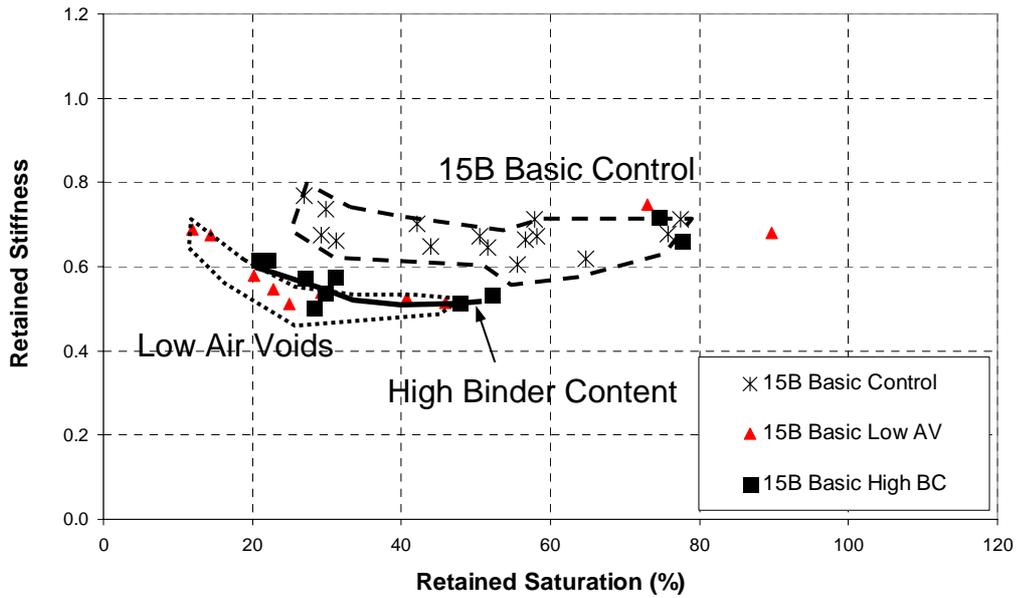


Figure 6.6 – Mixture variability investigation (15B Basic).

6.4.1 14 mm EME (EME 2)

A set of 14 mm EME (EME 2) specimens (AV = 4% and BC = 5.75%) was manufactured using the 15E binder and Basic aggregate (see Section 6.2). The SATS results are presented in Figure 6.7

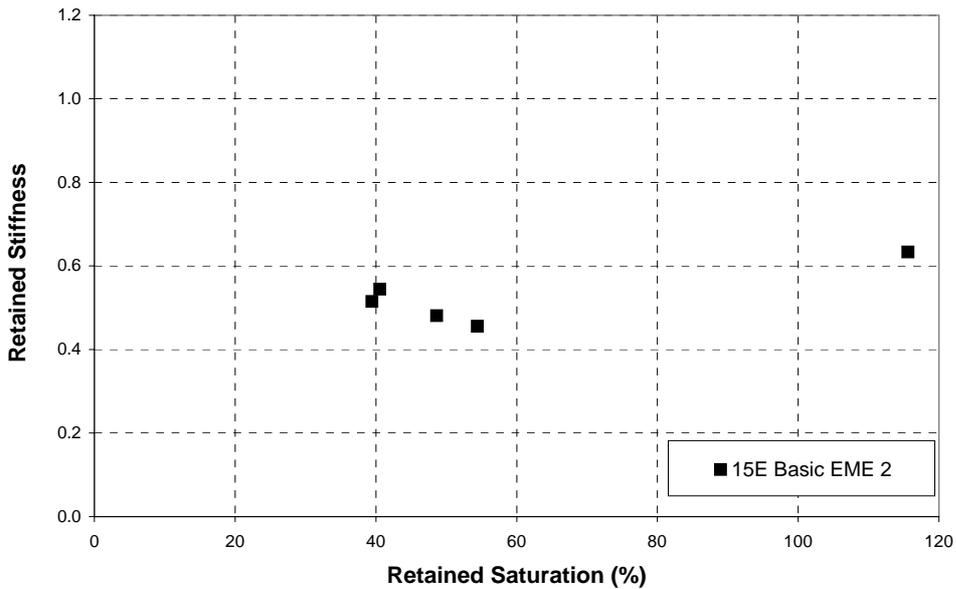


Figure 6.7 – SATS test results of 15E Basic EME 2.

Since the EME material has generally richer and well compacted properties (AV = 4% and BC = 5.75%), it was expected to have a better performance than other materials. However, from Figure 6.7, it is clear that the EME material did not perform better than the 15B Basic control material showing relatively low retained stiffness range (0.4 ~ 0.6). The most notable aspect observed from the EME mixture was the change of specimen shape. The entire specimen showed an expansion, especially in the diametral direction (e.g. initial diameter = 98 mm, diameter after test = 101 mm), as shown in Figure 6.8. Also the immersed specimen showed a retained saturation of 116%. Because the retained saturation is calculated based on the initial volumetric property, a retained saturation of higher than 100% indicates a change in the volumetric property during the process.

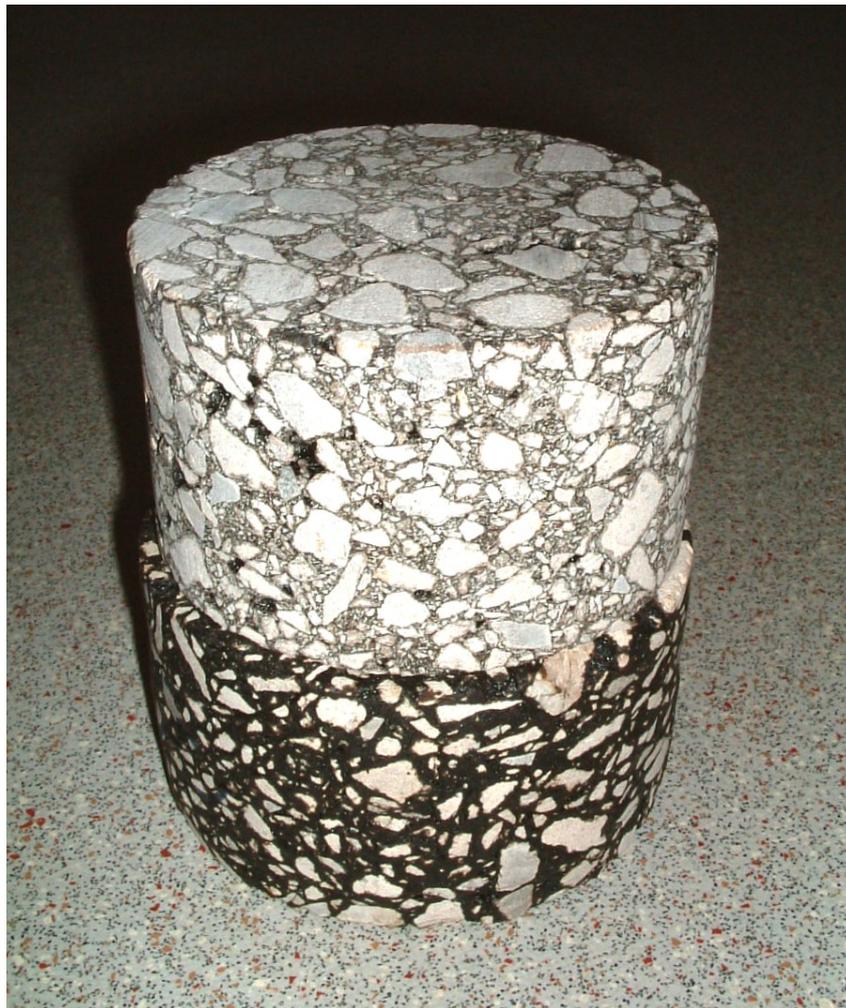


Figure 6.8 – EME specimens before and after the SATS test.

6.5 Summary and Conclusions

The SATS test was applied on a wider range of mixture conditions. Hydrated Lime Filler (HLF) has generally been considered to improve the bonding interaction between acidic aggregates and bituminous binders. The results from four Acidic mixtures with HLF showed superior performances. The retained saturation range (up to 60%) was also much reduced compared with both that of the Acidic control mixture (up to 90%) and that of the Basic control mixture (up to 75%). It was notable that some of the specimens with very low retained saturations (less than 25%) showed even higher retained stiffnesses than 1.0 (≈ 1.10). As with the Basic mixtures, the immersed specimens commonly showed better performance than that of the 'non-immersed' specimens, despite generally higher retained saturations.

The already good performance of the Basic control mixture was not significantly improved by the adding of HLF. Only one binder (15B) was investigated for this trial.

The effect of anti-stripping agent was investigated and showed an improved performance, although it was found to be much less significant than that of hydrated lime filler. However, it should be noted that the dosage of anti-stripping agent (0.5% by weight of binder) was considerably low compared to the dosage of the hydrated lime filler (2% by weight of aggregate), and a greater effect may be expected with a higher dosage. Only one binder (15B) was used for this trial.

The investigation on mixture variability demonstrated an unexpected result where the well compacted (lower air voids of 4%) or the richer mixture (higher binder content of 5%) showed worse performance than the control mixture, especially for the Basic mixtures. 15E EME Basic material also showed worse performance than expected. Moreover, it clearly showed a volumetric expansion having a retained saturation of higher than 100%.

An assumption was made that the specimen is damage/affected by another factor together with the ageing and moisture factors in the vessel, and the richer and/or well compacted mixture is considered to be more susceptible to the factor. The following

chapter presents a series of investigation to understand material behaviour and effect of controlling factors of the SATS protocol.

Chapter 7. SATS Parameter Investigation

7.1 Review of SATS Protocol

The SATS protocol was developed based on the specific aim to develop a long-term durability test protocol which can combine the binder ageing together with the moisture sensitivity test. The SATS main test series, as described in Chapter 5, and the recovered binder investigation (see Section 3.3) indicate that the SATS protocol successfully fulfils the main objectives of this study. More specifically, the SATS protocol could reproduce the given target stiffness modulus reduction (i.e. around 60% reduction, retained stiffness of 0.4) within a practically reasonable time scale (65 hours). Also, within the very same process, the SATS protocol could age the binder in the mixture comparable to the HiPAT binder ageing protocol which is considered to be 10 or more years of long-term ageing process [Hayton et al., 1999].

However, the SATS test was mainly focused on the High Modulus Base material with a relatively low binder content ($\approx 4\%$) and a relatively high air void content ($\approx 8\%$). Extended application of SATS to various types of mixtures (see Chapter 6) indicated that richer and/or well compacted mixtures were damage/affected by another factor, not only by ageing and moisture damage, showing some unexpected results. Therefore the following sections described a further investigation and modification on the key factors (e.g. pressure, temperature and test duration etc) of SATS protocol.

7.2 Test Setup

7.2.1 Temperature variation

The temperature inside of the pressure vessel was measured with partially filled water as seen in Figure 5.3 and compared to those from the early trial (from Section 4.3.1) in Table 7.1. Figure 7.1 shows the measured temperature at each specimen position.

Table 7.1 – Temperature variations of each SATS setup.

| Target temperature of 85°C | Max Temp. (°C) | Min Temp. (°C) | Variation (°C) |
|---|----------------|----------------|----------------|
| Normal setup (from Table 4.1) | 90 | 77 | 13 |
| Insulation on top cover (from Table 4.1) | 86 | 80 | 6 |
| Side heating source only (+ insulation on top cover) (from Table 4.1) | 86 | 82 | 4 |
| SATS (insulation applied as shown in Figure 4.6 (b)) | 87 | 82 | 5 |
| SATS (cover insulation removed) | 87 | 80 | 7 |

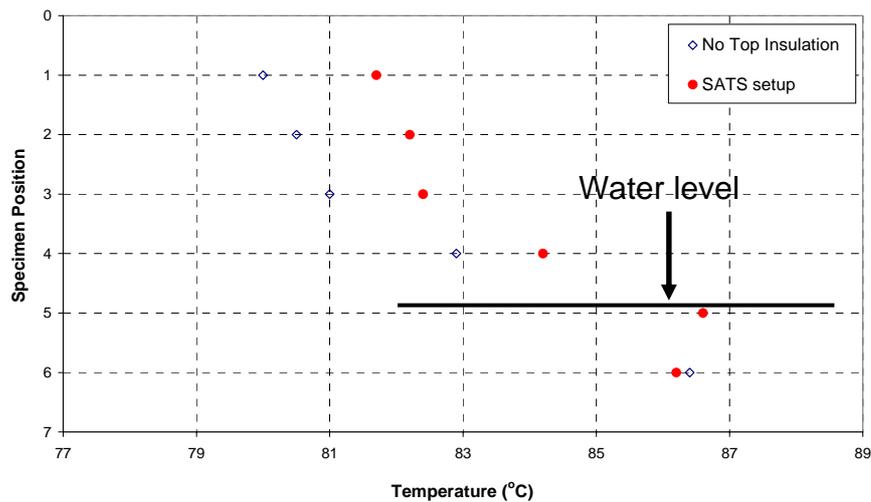


Figure 7.1 – Measured temperatures at each specimen position (SATS).

As seen in Table 7.1 and Figure 7.1, the temperature variation of the standard SATS protocol used in Chapter 5 was found to be around 5°C. However, it should be noted that the maximum temperature was found at just below the water surface (between 4th and 5th specimens in Figure 5.3) where no specimen was used. Also the temperature difference among the partially saturated specimens (Positions 1 ~ 4) as found to be much less (around 2°C). It is noted that the insulation applied in SATS (see Figure 4.6 (b)) appears to reduce the temperature variation up to 2°C, which is comparable to the 7°C reduction observed in the preliminary setup (see Table 7.1).

This difference is because that the temperature controlling thermocouple of the vessel could make direct contact with the partially filled water (see Figure 7.2) which results in a better temperature control (due to a better thermal conductivity of water) confirming the suitability of the standard SATS setup for the vessel design.

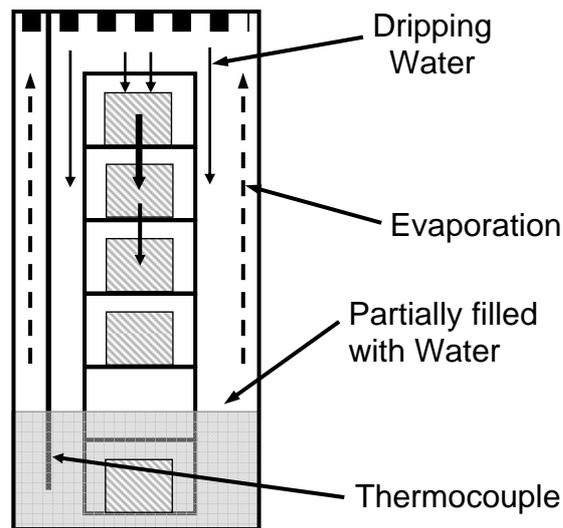


Figure 7.2 – Standard SATS setup in the pressure vessel.

7.2.2 Tray design

A new tray was designed and manufactured to have larger holes in each specimen shelf. This is to give a more increased and consistent water supply to the top specimen and eventually to lower specimens. As seen in Figure 7.3, the new tray has about five times larger hole area ($\phi 5$ mm, 36 holes) than the old tray ($\phi 2$ mm, 49 holes). Also the height of the empty space (between 4th and 5th specimens) has been reduced to 45 mm.

Figure 7.4 shows a comparison of the new tray results to the old tray results using the control mixtures (15B Acidic and 15B Basic). Both results are also compared to the previous data from Chapter 5 where identical mixtures were tested for repeatability (from Figure 5.7). As the results from both trays showed no significant difference, the new tray shown in Figure 7.3 has been adopted for the further study.

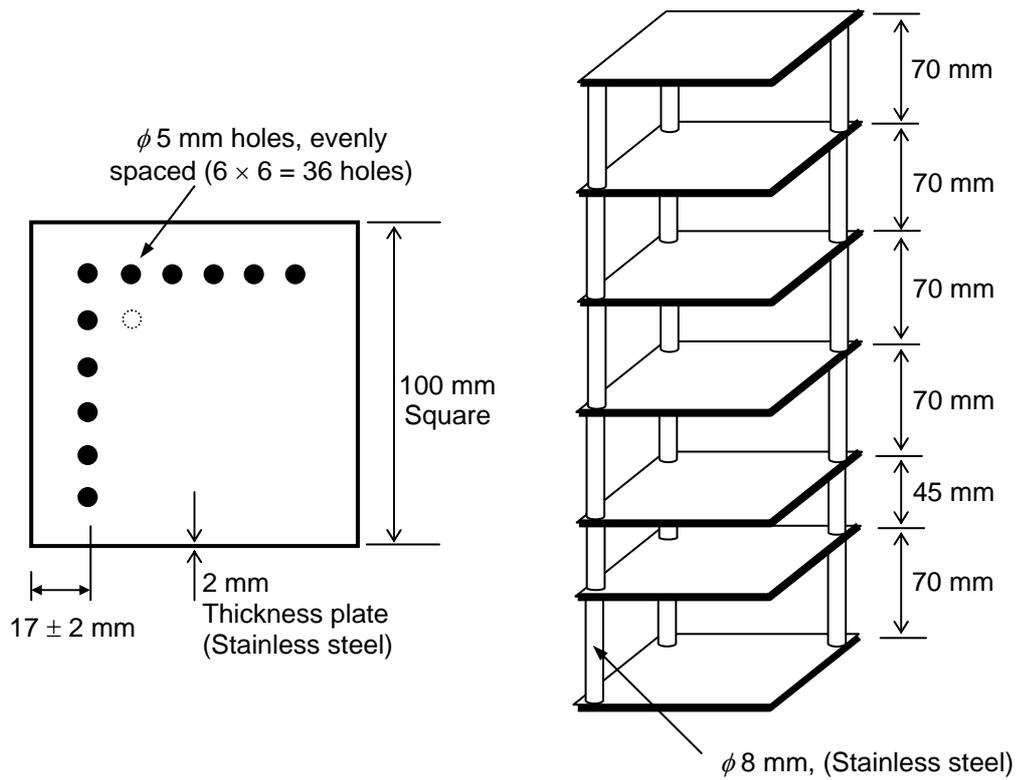


Figure 7.3 – Schematic and dimensions of the new specimen tray.

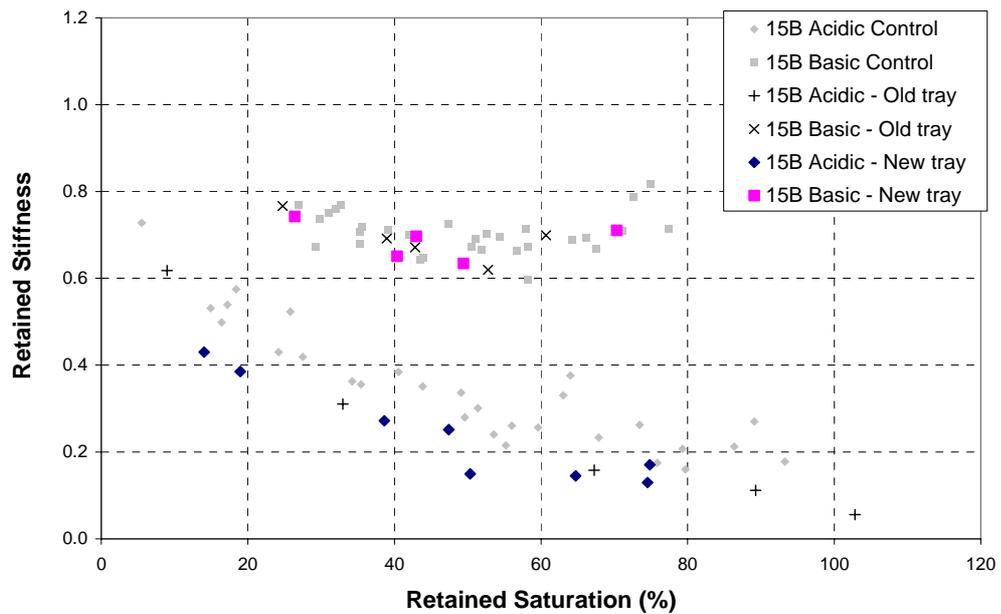


Figure 7.4 – Comparison of SATS results using old/new tray.

7.3 Pressure Condition

7.3.1 Half pressure SATS (1.0 MPa, 85°C, 65 hrs)

The most critical parameter of the SATS protocol is the applied pressure. The pressure is the key parameter to accelerate the binder oxidation (ageing) process through an increase amount of air and increased rate of diffusion. However, as the SATS protocol is applied on compacted specimen, the effect of this high pressure on the compacted specimen needs to be investigated. As the first step, the pressure applied to the SATS protocol was halved to 1.0 MPa, while other parameters remained the same. Both old and new trays were tested and results are presented in Figure 7.5.

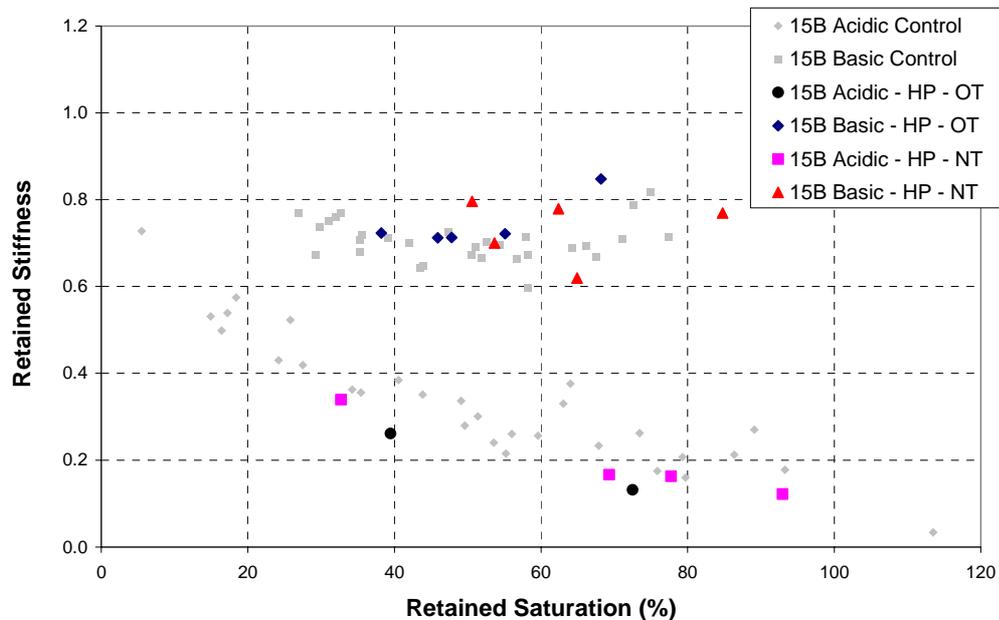


Figure 7.5 – Results of Half pressure SATS (1.0 MPa).

From Equation 5.3, it is clear that the specific humidity linearly increases as the pressure decreases (i.e. $\omega \propto P^{-1}$). Thus the reduced pressure will increase the amount of water vapour inside the vessel which will result in an increased amount of condensation beneath the lid, under the same temperature setup. Consequently, in a tightly concealed environment, a more accelerated water circulation (increased amount of dripping water supply to the specimens) is expected due to the increased amount of condensation.

In Figure 7.5 results show, as expected, generally higher retained saturations from most of the specimens (positions 1 ~ 4) with similar range of retained stiffnesses compared to the full pressure (standard SATS) test results. It is noted that a higher retained saturation range (50 ~ 85%) was found from 15B Basic with the new tray results. An investigation on recovered binders revealed that the level of ageing from the half pressure SATS is relatively lower than that from the full pressure test. More details are found in Chapter 9 (Section 9.4.4).

7.3.2 Atmospheric SATS (No added pressure, 85°C, 65 hrs)

This is to perform the SATS protocol in atmospheric pressure condition. No additional pressure was applied to the SATS protocol, while other parameters remained the same. Results are presented in Figure 7.6.

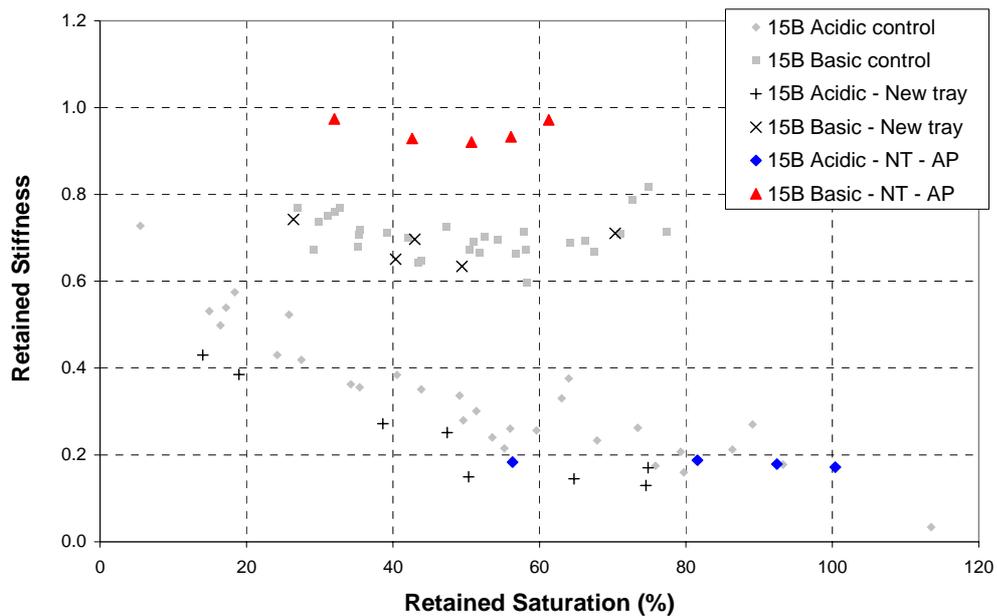


Figure 7.6 – Results of Atmospheric SATS.

From Figure 7.6, 15B Acidic shows generally higher retained saturation (over 60%) from most of the specimens (positions 1 ~ 4) with similar levels of retained stiffness (less than 0.3). However, the 15B Basic showed a similar range of retained saturation with virtually no reduction in the retained stiffness (0.9 ~ 1.0).

The increase level of retained saturation observed in 15B Acidic is thought to be due to a much more accelerated water circulation, compared to the half and full pressure setup. However, 15B Basic seems to be unaffected by the increased amount of water contact indicating much less sensitive property of the material against moisture damage. Moreover, the increase level of retained stiffness clearly indicated a certain amount of stiffness change, due to the applied pressure rather than moisture or ageing. An investigation on recovered binders revealed that the level of ageing from the Atmospheric SATS is significantly lower than those from the half pressure test and full pressure test. More details are found in Chapter 9 (Section 9.4.4).

7.4 Test Duration

The SATS test was performed for shorter test periods of 24 hours and 4 hours, while other parameters remained the same (2.1 MPa, 85°C). Results are presented in Figure 7.7.

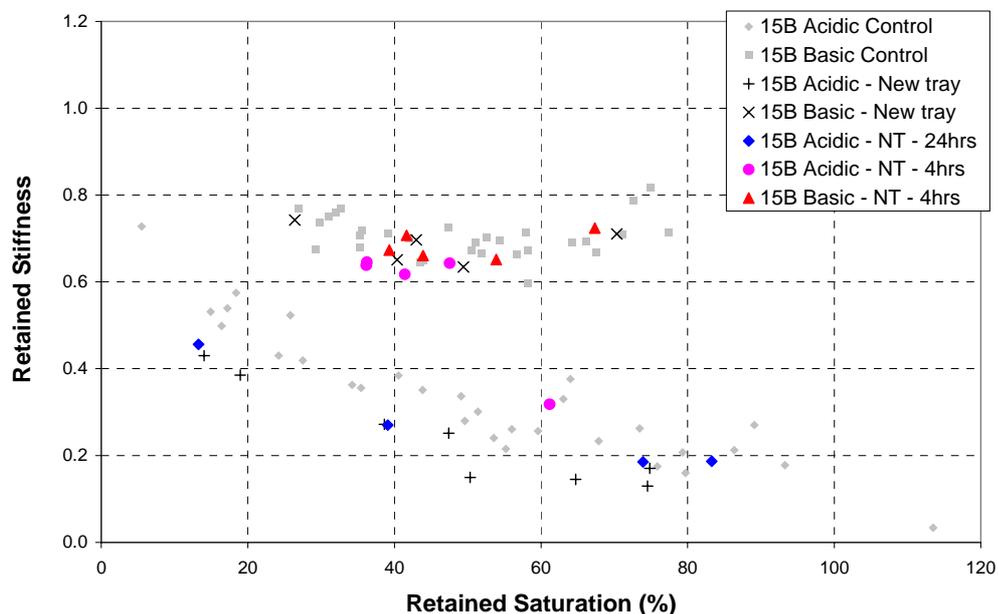


Figure 7.7 – Results of 24 hours and 4 hours SATS.

From Figure 7.7, it is clear that the level of stiffness reduction, commonly observed from 15B Acidic specimens so far, could be achieved in a very earlier stage of the SATS process (between 4 and 24 hours). It is interesting to note that the partially

saturated specimens (positions 1 ~ 4) from 4 hours SATS showed a very narrow range of retained saturation (around 40%) which is even lower than the initial saturation measured after the vacuum process (around 60%).

It can be assumed that, once the vacuum saturated specimens are placed in the vessel, a certain proportion of the initially saturated water (by the vacuum process) is commonly drained out of the specimens (above the water level, positions 1 ~ 4) resulting in a bit lower range of retained saturation than the initial saturation, as observed in '4 hours SATS' results. However, the condensed water from the lid starts to gradually re-saturate the specimens (from the top specimen) and, therefore results in the observed the retained saturation variation along the specimen position so far. The condensed water appears to re-saturate the specimens from around 4 hours after the process starts, and also this gradual re-saturation process appears to be stabilised within approximately 24 hours period. Thus the observed retained saturation variation of each specimen assumed to be maintained from around 24 hours to the completion of the test (65 hours).

Also it can be concluded that the observed retained saturation variation along the specimen position is mainly created by the dripping condensed water, rather than the initially saturated water, supporting the theory proposed in Section 5.5.2. 15B Basic specimens showed similar results to the full period SATS (standard SATS). An investigation on recovered binders revealed that the levels of ageing from the shorter period SATS tests are relatively lower than that from the full period SATS. More details are found in Chapter 9 (Section 9.4.5).

7.5 Temperature Condition

The SATS test was performed using lower temperatures of 60°C and 30°C, while other parameters remained the same (2.1 MPa, 65 hrs). Results are presented in Figure 7.8.

From Figure 7.8, the 15B Acidic specimens show a close relation to the applied temperatures. The results from 60°C SATS show an increased retained stiffness range (0.5 to 0.7), including the immersed specimen (Position 6), with a generally

reduced retained saturation range (25 ~ 60%). The results from 30°C SATS show a more increased retained stiffness range (around 0.8) with a generally reduced and narrower retained saturation range (40 ~ 55%), including the completely immersed specimen.

It is interesting to note that especially the immersed specimens from each setup showed a distinctive increase as the applied temperature decreased. For examples, the immersed 15B Acidic specimens from the standard SATS (85°C) commonly showed relatively lower retained stiffnesses (less than 0.2) compared to those of the specimens above the water level. However, the immersed specimen from 60°C SATS showed a similar retained stiffness (around 0.5) and even slightly higher retained stiffness (around 0.8) from the 30°C SATS, compared to those of the specimens above the water level from the same test. This indicates that the 15B Acidic is considerably sensitivity material to the water temperature and, together with the results from Atmospheric SATS (see Section 7.3.2), it can be proposed that the applied temperature is the key parameter to produce the target stiffness reduction (60% or more) in the SATS setup.

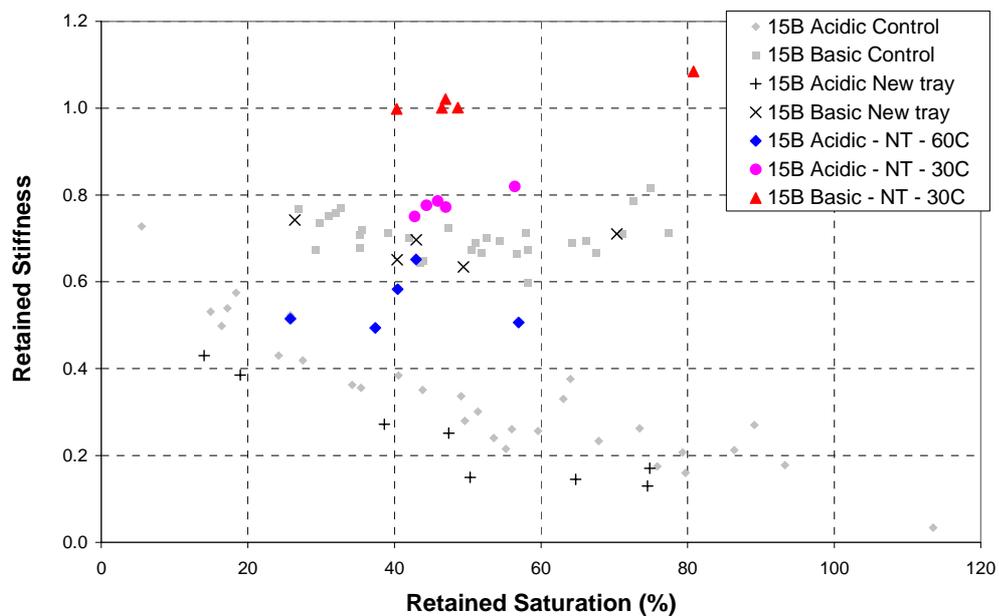


Figure 7.8 – Results of 60°C and 30°C SATS.

15B Basic, from Figure 7.8, shows virtually no change in its retained stiffness (around 1.0). This is a comparable result only to that from the Atmospheric SATS test (see Section 7.3.2). Considering that even at 4 hours SATS (see Section 7.4) showed a similar level of stiffness reduction, while the 15B Acidic showed a much increased retained stiffness compared to those from the standard SATS, it is clear that somehow the 30°C SATS appears to be unaffected (or less affected) by the applied pressure (2.1 MPa). An investigation on recovered binders revealed that the level of ageing from the 60°C SATS test is relatively lower than that from the standard SATS (85°C). More details are found in Chapter 9 (Section 9.4.6).

7.6 Mode of Pressure Damage

The most critical parameter of the SATS protocol is the high pressure (approximately 22 times of atmospheric pressure) which is used to accelerate the binder ageing in the mixture. However, from the investigation performed in the previous sections, it is clear that there is an influence to the retained stiffness caused by the high pressure.

The compacted specimen contains a certain proportion of air voids which are at atmospheric pressure before the SATS protocol. If the external pressure increases, the pressure inside the air voids also needs to increase to balance the external pressure. If the material is porous (e.g. aggregate) and the air voids are interconnected, the increased pressure will reach into the ‘open’ air voids freely. However the non-interconnected voids, such as air voids contained in bituminous binder, will be forced to be pressurised by other means. Examples of interconnected ‘open’ voids (a) and non-interconnected ‘closed’ voids (b) are shown in Figure 7.9 where the yellow parts represent the voids.

It is thought that there are two different extreme modes (Mode I and II) of the air voids pressurising process, as described in Figures 7.10 and 7.11 respectively. In the Mode I pressurising process (see Figure 7.10) the void containing bitumen binder is deformed (contracted) by the applied pressure until the void pressure becomes equivalent to the applied pressure (i.e. same amount of air in less volume of void). In the Mode II pressurising process (see Figure 7.11) one corner of the void

containing bitumen binder is ‘punctured’ providing a ‘channel’ for the pressurised air to infiltrate into the void, until becomes equivalent to the applied pressure (i.e. more amount of air in same volume of void). These processes are also likely to be taking place when the pressure is released (depressurising process) after the SATS protocol, in a reverse manner. For examples, in Mode I depressurising process, the air void containing binder will ‘expand’ to equalise the decreasing external pressure (same amount of air in more volume of void), while the pressure will be released through the pressure channel (puncture) in Mode II depressurising process.

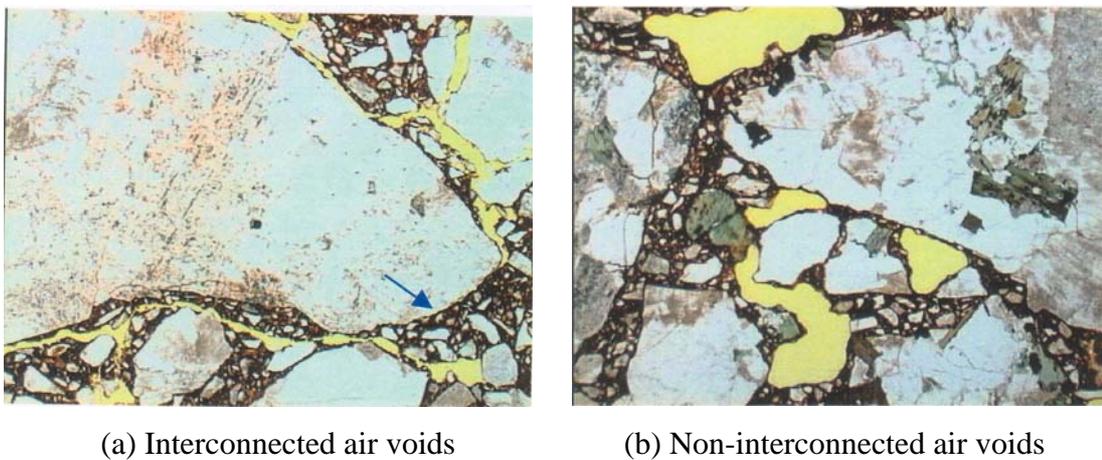


Figure 7.9 – Examples of interconnected air voids and non-interconnected air voids in a ‘compactd’ bituminous mixture.

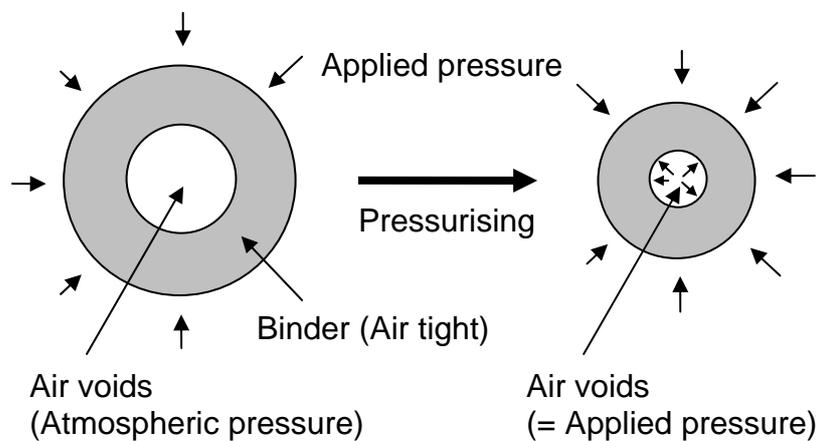


Figure 7.10 – Conceptual diagram of Mode I (deformation) void pressurising process.

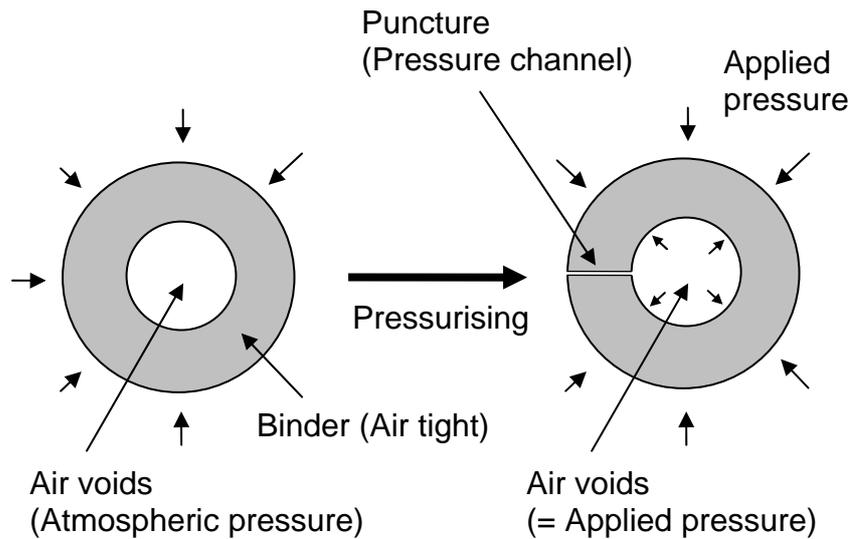


Figure 7.11 – Conceptual diagram of Mode II (puncture) void pressurising process.

The ‘punctures’ created in Mode II process are thought to be microscopic and the volumetric change is none (or minimal), giving minimum disturbance to the surrounding aggregate structure (see Figure 7.12). However, in Mode I depressurising process, the affected (deformed) area (or volume) of the binder is much greater (all around the void) giving maximum disturbance to the surrounding aggregate structure, due to the physical expansion of the void containing binder (see Figure 7.13). Therefore a physical damage to the compacted specimen, caused by the pressurising or depressurising process, is thought to be far greater in Mode I than Mode II.

It is logical to think that, considering the visco-elastic and temperature dependent characteristics of bituminous binder, the Mode I process is more likely to happen when the binder is in higher temperature having more viscous property, whereas the Mode II is more likely in the lower temperature with more elastic property. However in reality, as the above assumptions were made for the extreme cases, both modes of pressurising (or depressurising) process are likely happening together. A specimen, as an example, could be damaged by the Mode I process up to a certain proportion then move on to Mode II process. At a higher temperature Mode I damage is considered to be a dominant form but there will still be a minor proportion of Mode II damage and vice versa for the lower temperature case.

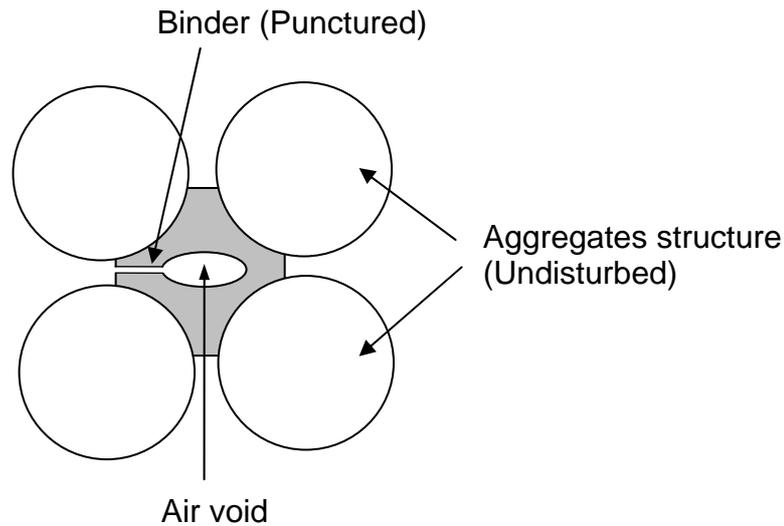


Figure 7.12 – Conceptual diagram of Mode II damage in depressurising process.

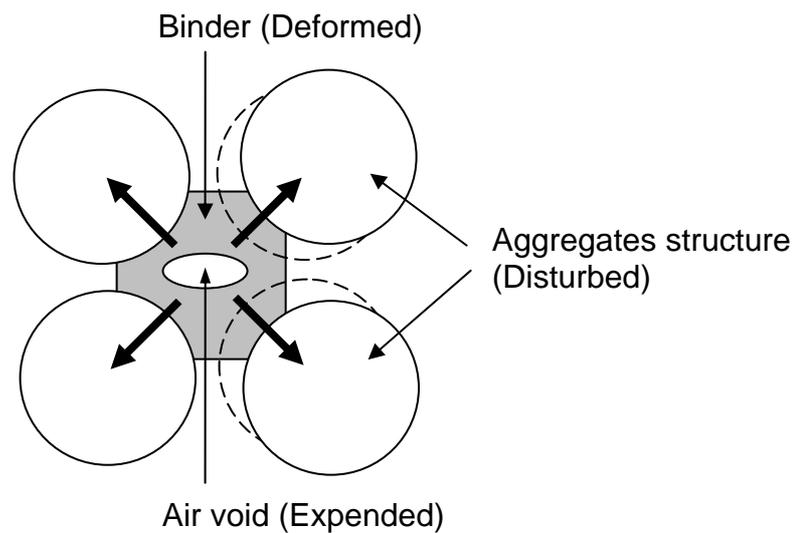


Figure 7.13 – Conceptual diagram of Mode I damage in depressurising process.

The exact relationship of Mode I and Mode II ratio as a function of temperature is unknown and considered to be beyond the scope of this study. However, based on the temperature dependent characteristics of bituminous binder, the following qualitative correlations could be proposed.

- Higher temperature (more viscose); Mode I > Mode II
- Lower temperature (more elastic); Mode I. < Mode II

Also at a same temperature;

- High pen grade binder (more viscose); Mode I > Mode II
- Low pen grade binder (more elastic); Mode I < Mode II

7.7 Summary and Conclusions

The developed 'standard SATS' protocol appears to fulfil the main objectives of the study, providing a durability test protocol which combines long-term binder ageing process and mixture moisture sensitivity process. However, from a further investigation on the parameters used in the protocol (i.e. pressure, temperature and test duration) it appears that the high pressure (introduced to accelerate binder ageing process) also induces a certain degree of specimen damage. Consequently two different modes of pressure related damage have been proposed and their expected properties are suggested.

Assuming that only bitumen binder is likely to be affected by the pressuring/depressurising process, a mixture with a more binder content and/or low air voids content is thought to be more susceptible to the pressurising/depressurising damage from the SATS protocol, since the mixture is likely to have a more closed 'non-interconnected' voids. Also, a mixture with a better adhesion property between aggregates and binder will be more susceptible to the pressurising/depressurising damage as the mixture will create more 'non-interconnected' voids along the interface of aggregate and binder.

The following chapter describes an investigation to prevent (or minimise) the pressure related damage discussed in this chapter.

Chapter 8. SATS Modification

8.1 Review of Pressure Damage

Investigation from Chapter 7 clearly indicated that there is a certain degree of damage (i.e. reduction in retained stiffness) caused by the pressure used in the SATS protocol. Thus the unexpected results observed in Chapter 6 are thought to be due to the influence of pressure related damage. Especially the 15E EME Basic mixture (see Section 6.4.1) is considered to be most affected by the pressure damage, showing unexpectedly low retained stiffnesses (around 0.5) with a clearly visible shape change (diametral expansion). It should be noted that the 15E EME mixture has the highest binder content (5.75%), the lowest air voids (4%) and the smallest maximum aggregates size (14 mm, compared to 28 mm DBM) which overall could contribute to produce a considerably more 'pressure sensitive' specimen (containing much more 'non-interconnected' air voids).

The following sections describe further modifications to the standard SATS protocol with the principal objective to remove (or reduce) the pressure related damage.

8.2 Depressurising Process Investigation

The pressure damage appears to be more related to the pressurising or depressurising process, rather than the absolute value of the applied pressure, since the 15B Basic specimens from the half pressure SATS (see Section 7.3.1) and the full pressure standard SATS showed a very similar retained stiffness range. In the standard SATS protocol (see Appendix D), the pressurising process takes place when the specimens are still in ambient temperature, since the pressure is applied during the first 20 minutes, immediately after the specimens are placed in the vessel following the vacuum saturation process in ambient temperature ($\approx 20^{\circ}\text{C}$). However the depressurising process takes place when the specimens are fully heated ($\approx 85^{\circ}\text{C}$). The study in Section 7.6 has suggested that the specimens are likely more susceptible to the pressure damage at a higher temperature (as Mode I damage is dominant), thus

the pressure damage of the SATS protocol is considered to be dominantly taking place in the depressurising process.

8.2.1 3 hours depressurisation SATS

In the standard SATS protocol, the vessel is depressurised within 30 minutes (see Appendix D). To investigate the effect of pressure release rate, a depressurising process with a considerably slower release rate was introduced. After the standard SATS protocol (2.1MPa, 85°C, 65 hours), the pressure was released uniformly over a 3 hour period and the results are presented in Figure 8.1.

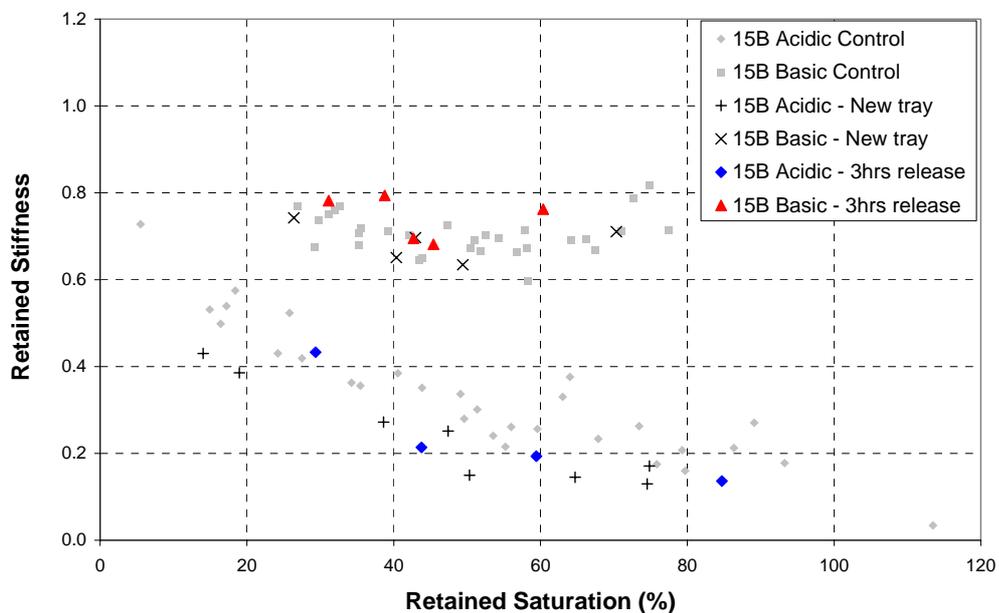


Figure 8.1 – Results of 3 hours depressurisation SATS.

From Figure 8.1, the results from the slower rate depressurisation SATS shows virtually no change in the retained stiffness indicating that the same (or similar) level of pressure damage has been occurred.

8.2.2 30°C depressurisation SATS

The objective of this trial is to release the pressure when the specimens are at (or close to) ambient temperature. After the standard SATS protocol (2.1 MPa, 85°C,

65 hours), an additional cooling period was allowed while the pressure (2.1 MPa) was still maintained. The target cooling temperature was chosen to be 30°C which took an additional 24 hours to reach after the heater was turned off. Since the temperature of the SATS PAV is only controlled by the incorporated heaters (i.e. no cooling unit installed), the target temperature needs to be slightly higher than ambient temperature to achieve the exact target temperature (30°C) every time regardless of any change in ambient temperature. The retained stiffnesses and retained saturations are presented in Figure 8.2.

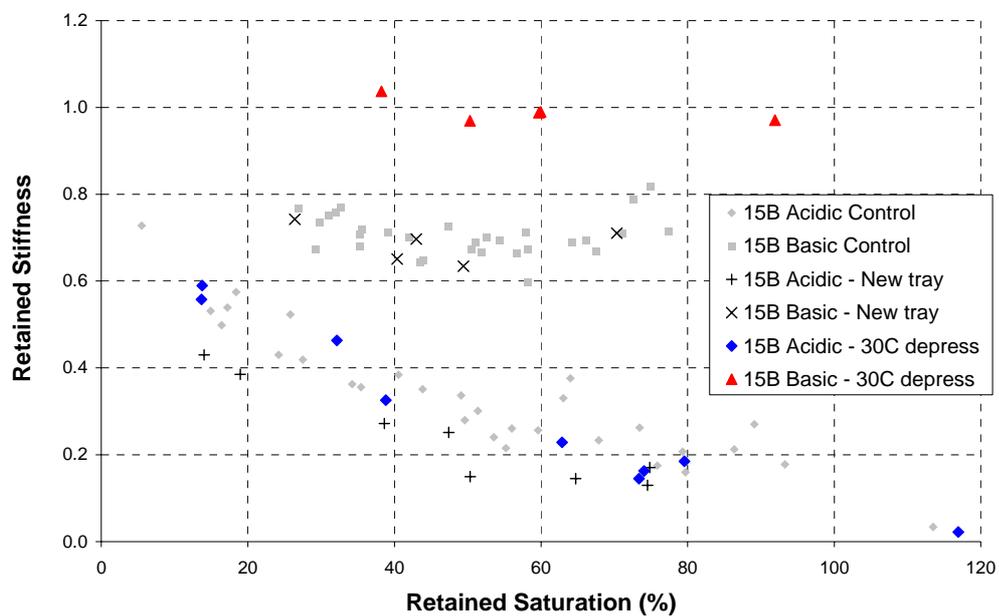


Figure 8.2 – Results of 30°C depressurisation SATS.

The 15B Basic mixture, from Figure 8.2, clearly shows increased retained stiffness values (around 1.0) comparable to those from Atmospheric SATS (see Section 7.3.2) indicating a much reduced pressure damage. It is also clear that 15B Acidic shows similar results to the standard SATS indicating that the poorer performance commonly observed in the Acidic mixtures so far is dominantly due to the moisture sensitivity of the material rather than the pressure effect.

The results support the pressure damage theory proposed in Section 7.6 and the temperature of 30°C appears to be low enough where the Mode II depressurising process is a dominant form (i.e. Mode I \ll Mode II). Thus the 30°C

depressurisation SATS protocol (referred to as ‘**modified SATS**’) has been adopted for further study. The reason that the 15B Acidic mixture is ‘insensitive’ to the pressure damage is believed to be linked with the generally poor performance of the material. When the test is completed (after 65 hours) the 15B Acidic specimens have already been severely damaged creating much more ‘interconnected’ voids (e.g. more cracks, more debonded binder from aggregates etc) which can provide enough number of pressure channels for the following depressurising process.

8.3 Modified SATS (2.1 MPa, 85°C, 65 hrs, 30°C Depression)

The modified SATS protocol, as described in Section 8.2.2, was applied on a series of mixtures selected from Chapter 6. Results from the modified SATS are presented in the following sections and are compared to those from the standard SATS (from Chapter 6).

8.3.1 15B Acidic (low air voids, high binder content)

Results of 15B Acidic with lower air voids or higher binder content (see Chapter 6 for more details) are presented in Figures 8.3 and 8.4 respectively.

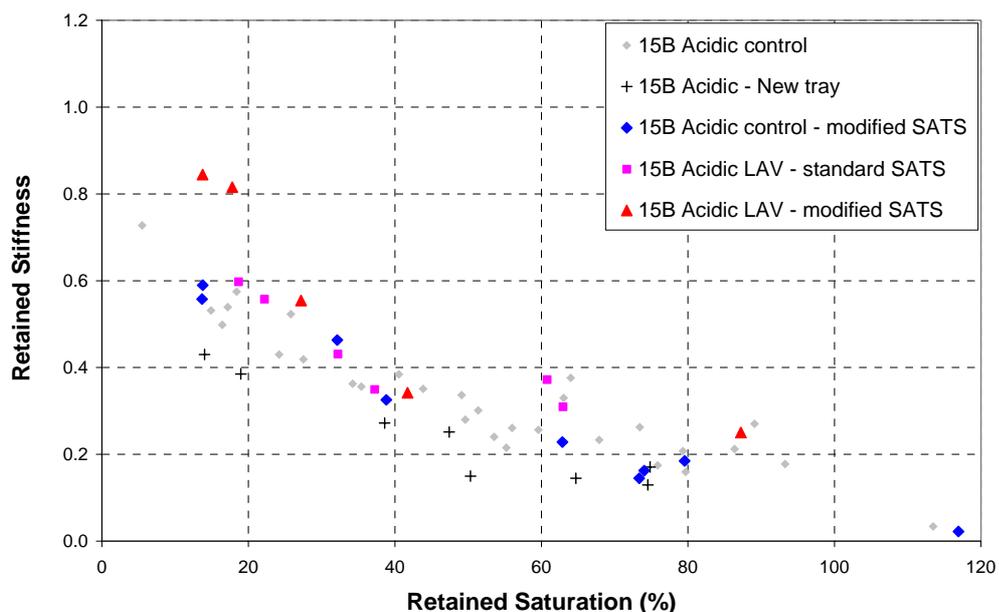


Figure 8.3 – Results of 15B Acidic low air voids (LAV).

From Figure 8.3, the 15B Acidic low air voids (LAV) specimens show similar retained stiffness and retained saturation trend to those of the control mixture (from modified SATS) and the low voids mixture (from standard SATS).

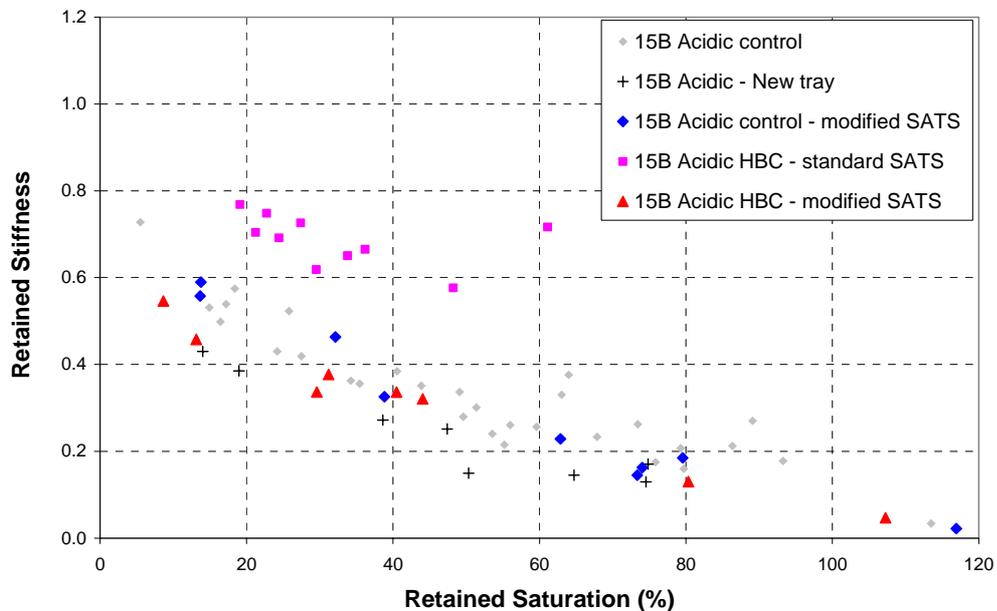


Figure 8.4 – Results of 15B Acidic high binder content (HBC).

From Figure 8.4, the 15B Acidic high binder content (HBC) specimens show similar retained stiffnesses and retained saturations to the 15B Acidic control mixture (from modified SATS). However it is notable that the results were poorer compared to the high binder content specimens tested using the standard SATS (from Chapter 6). This is thought to be due to the new tray (compared to the old tray used in the standard SATS protocol) and the further extended process time (65 hours of testing time + 24 hours of cooling time) of the modified SATS protocol, giving a relatively severer moisture damage condition (increased and prolonged water contact) than the standard SATS protocol. It also appears that, from Figures 8.3 and 8.4, the poorer performance of the 15B Acidic control mixture is not recognisably improved by change of the volumetric properties.

8.3.2 15B Basic (low air voids, high binder content)

Results from the 15B Basic specimens with lower air voids or higher binder contents (see Chapter 6 for more details) are presented in Figures 8.5 and 8.6 respectively.

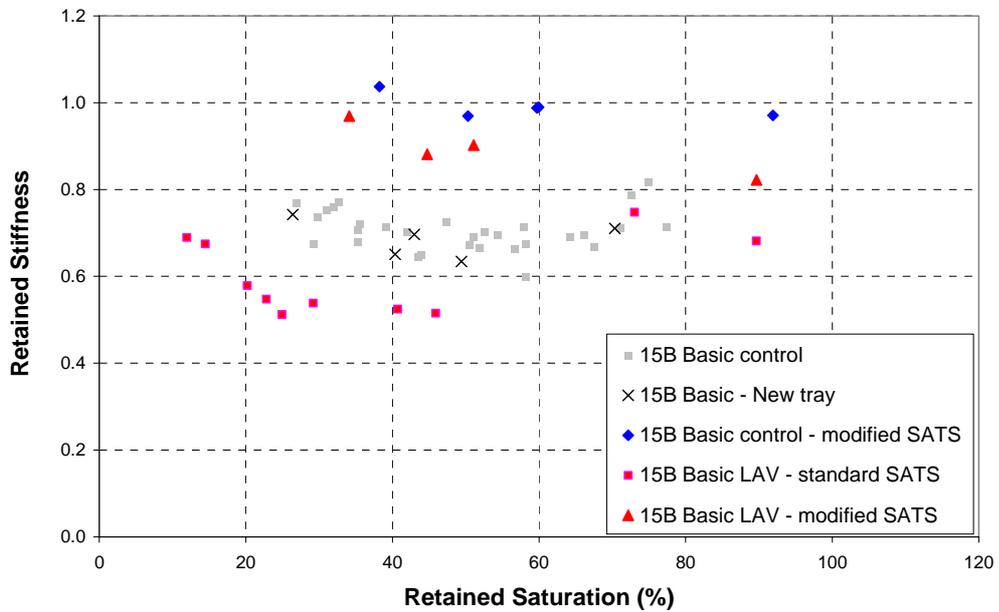


Figure 8.5 – Results of 15B Basic low air voids (LAV).

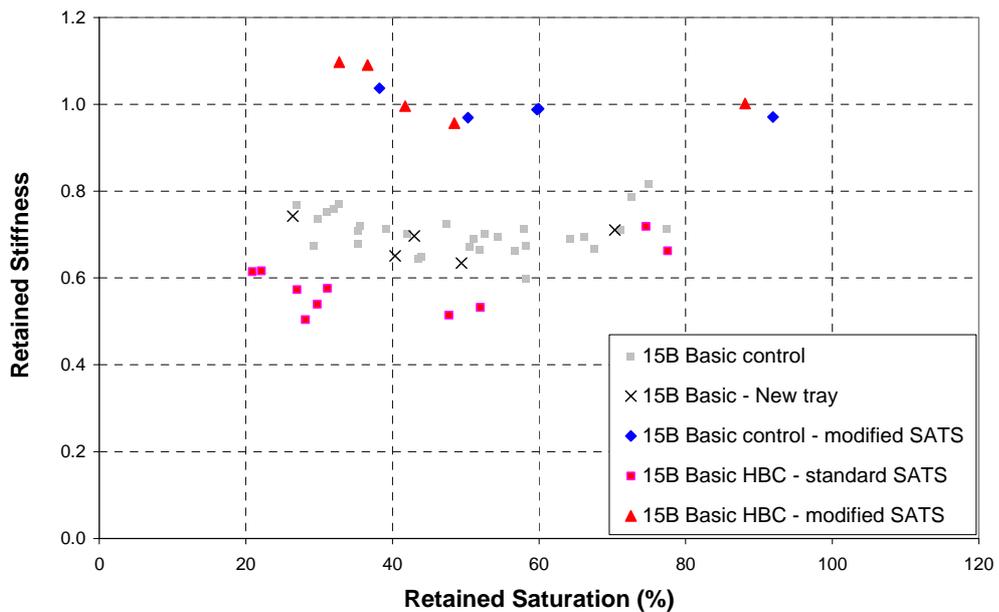


Figure 8.6 – Results of 15B Basic high binder content (HBC).

From Figures 8.5 and 8.6, the 15B Basic specimens with low air voids (LAV), or high binder content (HBC), commonly show much increased retained stiffness ranges, compared to those of the specimens tested using the standard SATS protocol (from Chapter 6) indicating a much reduced pressure damage. Also both mixtures commonly show comparable ranges of retained stiffnesses (LAV = 0.80 ~ 1.00, HBC = 0.95 ~ 1.10) to the control mixture tested using the modified SATS protocol (around 1.0). It is thought that the already well performing 15B Basic control mixture is not significantly affected by the volumetric changes used in this study.

8.3.3 15E EME Basic

Results of 15E EME Basic (see Chapter 6 for more details) are presented in Figure 8.7.

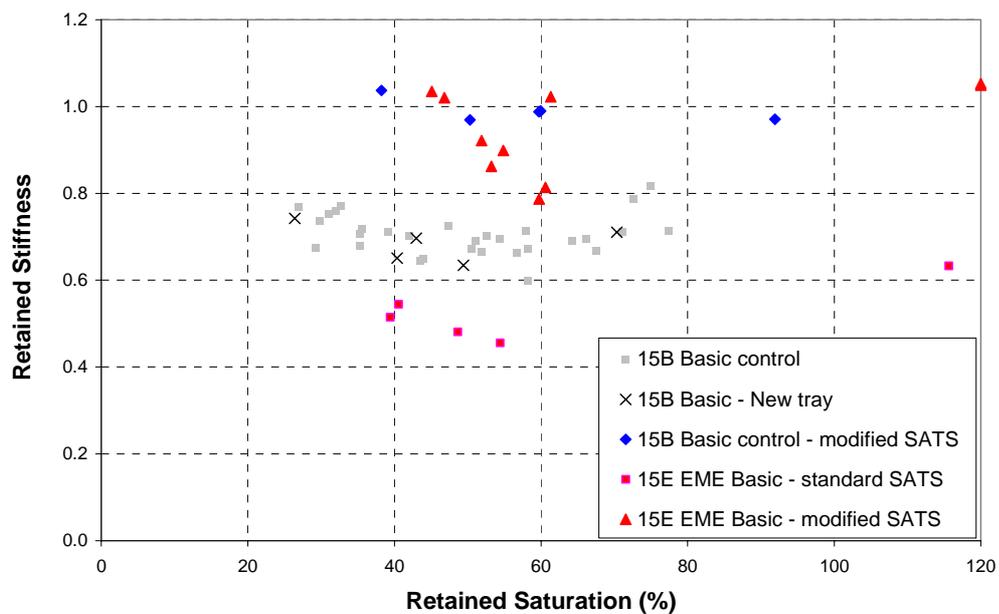


Figure 8.7 – Results of 15E EME Basic.

From Figure 8.7, the 15E EME Basic specimens show a much increased retained stiffness range (0.80 ~ 1.05, from specimens above water level), compared to that of the specimens tested using the standard SATS protocol (around 0.5, from Chapter 6) indicating a much reduced pressure damage. The visible swelling of the specimen observed in Figure 6.8 also significantly reduced showing virtually no change in the shape (see Figure 8.8 – (b)).

Since the 15E EME Basic mixture is thought to be more pressure sensitive, the marginally lower range of retained stiffnesses than 15B Basic control mixture (see Figure 8.7) is presumed to be due to a relatively higher level of pressure damage, rather than its true moisture sensitivity.



(a) Standard SATS (from Figure 6.8)

(b) Modified SATS

Figure 8.8 – Comparison of EME specimens before and after the SATS test (standard SATS and modified SATS).

It is notable that the immersed specimens (Position 6) typically showed a higher retained stiffness than those from the non-immersed specimens (Positions 1 ~ 4) which is also typically found from most of the Basic mixtures investigated in Chapters 5, 6 and 7. It is thought that the ‘pressurised’ water confines all around the specimen giving an extra support to the immersed specimen.

8.3.4 15B Acidic with HLF

Results of 15B Acidic with HLF (see Chapter 6 for more details) tested using the modified SATS protocol are presented in Figure 8.9. The 15B Acidic HLF specimens, from this figure, show a similar retained stiffness range (around 1.0)

comparable to that from the standard SATS. However, considering the generally increased retained saturation range (35 ~ 75%) due to the changed setup (see Section 8.3.1), it can be considered that the 15B Acidic HLF from the modified SATS performed slightly better than that from the standard SATS (i.e. higher retained stiffness at the same retained saturation).

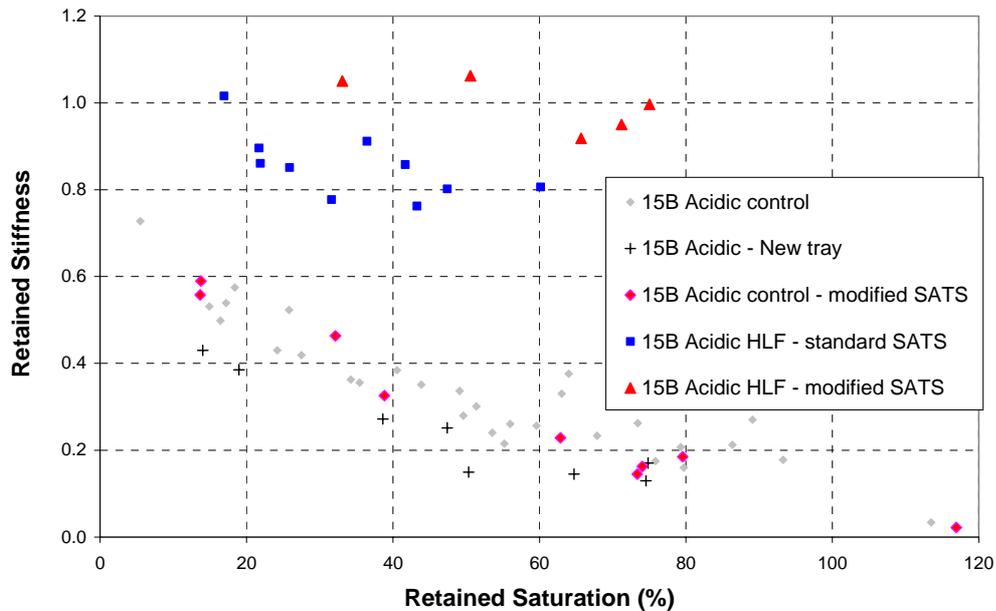


Figure 8.9 – Results of 15B Acidic with HLF (modified SATS).

This indicates that the 15B Acidic HLF mixture has more ‘non-interconnected’ voids than 15B Acidic control mixture, thus relatively more sensitive to the pressure damage. Consequently the modified SATS contributed to reduce the pressure related damage, although much less distinctive than the Basic mixtures.

8.3.5 Dry HiPAT ageing test with 30°C depressurisation process

The objective of this trial was to investigate the effect of the pressure release mode on the dry HiPAT aged specimens. Three of 15B Acidic and three of 15B Basic control specimens were prepared and placed in the SATS PAV using the old tray (the new tray can only contain five specimens), and then the HiPAT dry ageing protocol (2.1 MPa, 85°C, 65 hrs) was applied. The pressure was released as the same manner to the modified SATS protocol (i.e. depressurised at 30°C). Results

from the 15B Acidic specimens (from Positions 1, 3 and 5) and 15B Basic specimens (from Positions 2, 4 and 6) are presented in Figure 8.10.

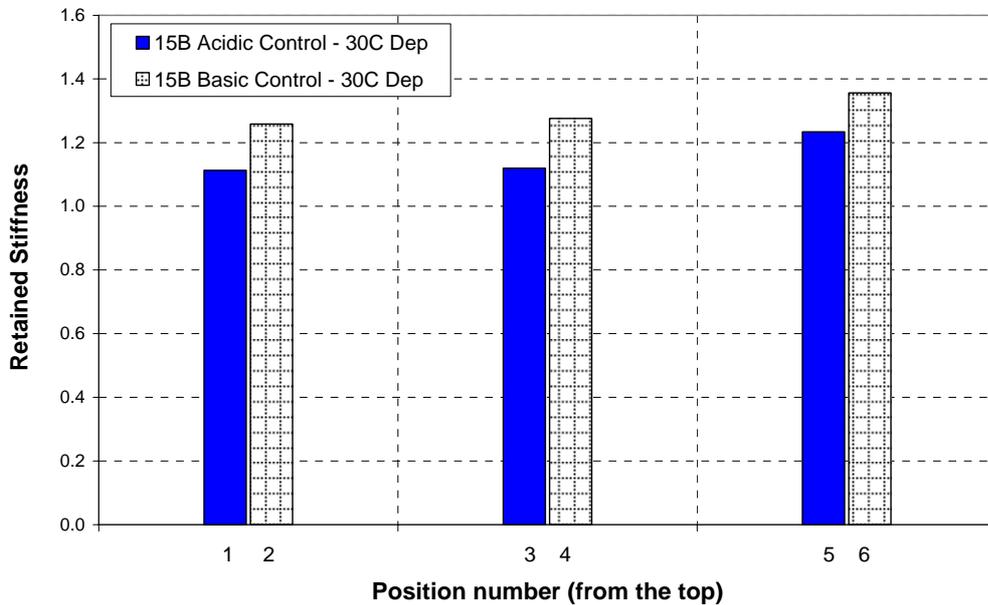


Figure 8.10 – Dry HiPAT ageing test with 30°C depressurisation process

As a comparison, another set of 15B Acidic and Basic specimens were prepared and underwent the same HiPAT dry ageing protocol, but the pressure was released in the same manner to the standard SATS protocol (i.e. depressurised at 85°C). Figure 8.11 shows the comparison of the average values of retained stiffnesses from the 30°C and 85°C depressurisation dry HiPAT aged specimens.

Figure 8.11 clearly displays that the 15B Basic was considerably affected by the pressure release mode confirming the 15B Basic is a more pressure sensitive material than 15B Acidic material. When the pressure was released at the high temperature (85°C) the ‘aged’ 15B Basic showed even lower retained stiffnesses (≈ 0.8) while the 15B Acidic showed a slightly increased value (≈ 1.05). However, when the pressure was released at the lower temperature (30°C) the 15B Basic clearly showed a much increased retained stiffness of 1.30 indicating a much reduced pressure damage. It is noted that the 15B Acidic also showed an increased value of 1.16 from the 30°C depressurisation protocol indicating a certain level of pressure sensitivity, although much less significant than that of the 15B Basic material.

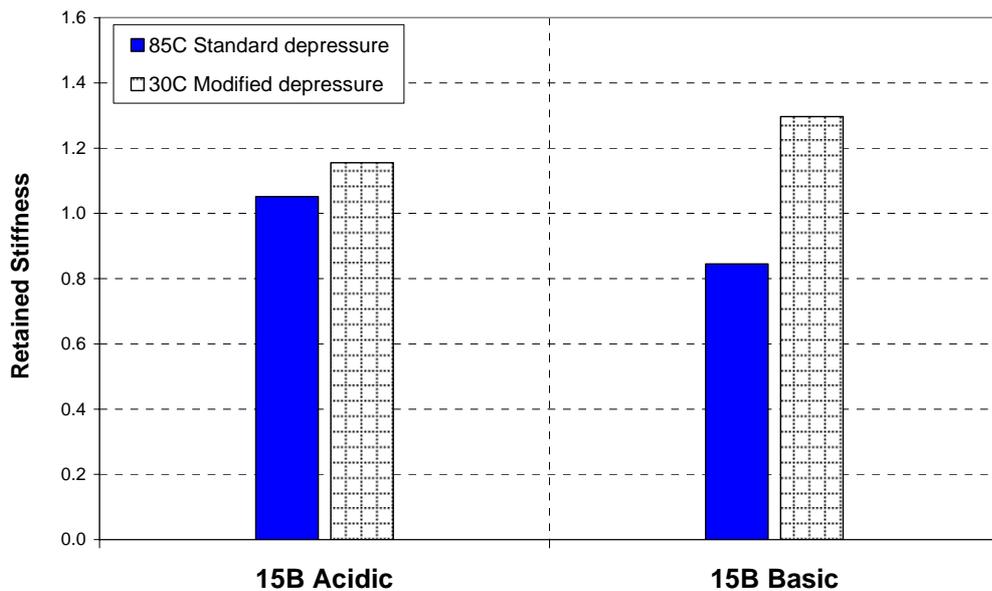


Figure 8.11 – Comparison of 30°C and 85°C depressurisation Dry HiPAT ageing tests.

8.4 Summary and Conclusions

The relatively high pressure used in the SATS protocol inevitably causes a certain degree of structural damage to the compacted specimen (particularly at the depressurising process). Thus, to remove (or reduce) the pressure related damage, a modification was applied on the standard SATS protocol by releasing the pressure at a much lower temperature of 30°C (instead of 85°C).

The modified SATS protocol (30°C depressurisation SATS) was applied to a limited range of mixtures and clearly displayed much reduced pressure damage (especially from the Basic mixture series). It also reveals that specimens containing more ‘non-interconnected’ voids (e.g. richer, denser and more homogeneous) are likely more prone to the damage caused by the depressurisation process.

In general, the standard SATS protocol used in Chapter 5 appear to be an acceptable approach, considering the distinctively ‘lean’ volumetric properties (i.e. High AV and Low BC) of the control materials focused on this study. Thus the results set produced in Chapter 5 using various combinations of binders and aggregates are

considered to be credible, especially for the Acidic materials which are nominally identical to the HMB trial site material. However it should be noted that, especially for the Basic materials, the presented results incorporate a considerable amount of pressure damage and the damage can be much reduced by the modified SATS protocol proposed in this chapter.

Therefore the modified SATS is believed to be a better protocol than the standard SATS, especially to be applied to a wider range of mixtures including the mixtures with a more pressure sensitive volumetric properties (e.g. Low AV or/and High BC).

Chapter 9. Recovered Binder Assessment

9.1 Introduction

One of the main objectives of the SATS test is to combine ageing and moisture conditioning, as described in Chapter 1. Normally, it could be predicted that ageing results in an increased stiffness, whereas moisture conditioning results in a reduced stiffness. The SATS protocol typically results in reduced retained stiffnesses under the combined effect of both conditioning procedures. A separate investigation was performed on the recovered binder properties from the tested specimens using the SATS protocol, to investigate the effect of the protocol in terms of ageing of the binders.

9.2 Materials

A number of samples of the binder were recovered from the 15B Acidic and Basic specimens, which had been tested using the SATS protocols under various conditions. The details of each specimen condition are accordingly presented in the following sections.

9.3 Binder Recovery and Testing (Stage 1)

The binders were recovered using the BS protocol [BSEN 12697-4, 2000] in sufficient quantities to allow Dynamic Shear Rheometer (DSR) testing to be undertaken. The DSR results of recovered binders were compared to those from the DSR tests on ‘unaged’ and aged binders (RTFOT and HiPAT), which were carried out as described in Appendix C. Figures 9.1 to 9.4 show the complex modulus (G^*) and phase angle (δ) master curves of the recovered 15B binders from the listed specimens (see Table 9.1), at a reference temperature of 25°C. Note that the DSR tests on the recovered binder were carried out in a temperature range of 10 ~ 50°C using the 8 mm standard spindle only for this stage.

Table 9.1 – Details of specimens for binder recovery investigation (stage 1).

| Series No. | | 1 | | 2 | | 3 | | 4 | |
|-----------------------------------|--------------------|---------------------------|------|--|------|--|------|--|--|
| Related chapters (section number) | | 3.3 | | 4.2 | | 5.4 | | 7.3.2 | |
| Mixture Type | | 15B Acidic | | 15B Acidic | | 15B Acidic | | 15B Acidic | |
| S A T S | Specimen Condition | Dry HiPAT Aged | | Preliminary SATS (partially saturated) | | Standard SATS (immersed) | | Atmospheric SATS | |
| | Water Condition | No initial or added water | | Initial vacuum saturation only | | Initial saturation + added water (Position 6 immersed) | | Initial saturation + added water (Position 6 immersed) | |
| | Pressure (MPa) | 2.1 | | 2.1 | | 2.1 | | Atmospheric | |
| | Temperature (°C) | 85 | | 85 | | 85 | | 85 | |
| Specimen Position | | 3 | 4 | 1 | 3 | 6 | 1 | 2 | |
| Retained Sat. (%) | | - | - | - | - | 93.3 | 71.1 | 66.5 | |
| Retained Stiffness | | 1.09 | 1.08 | 0.29 | 0.50 | 0.18 | 0.33 | 0.37 | |

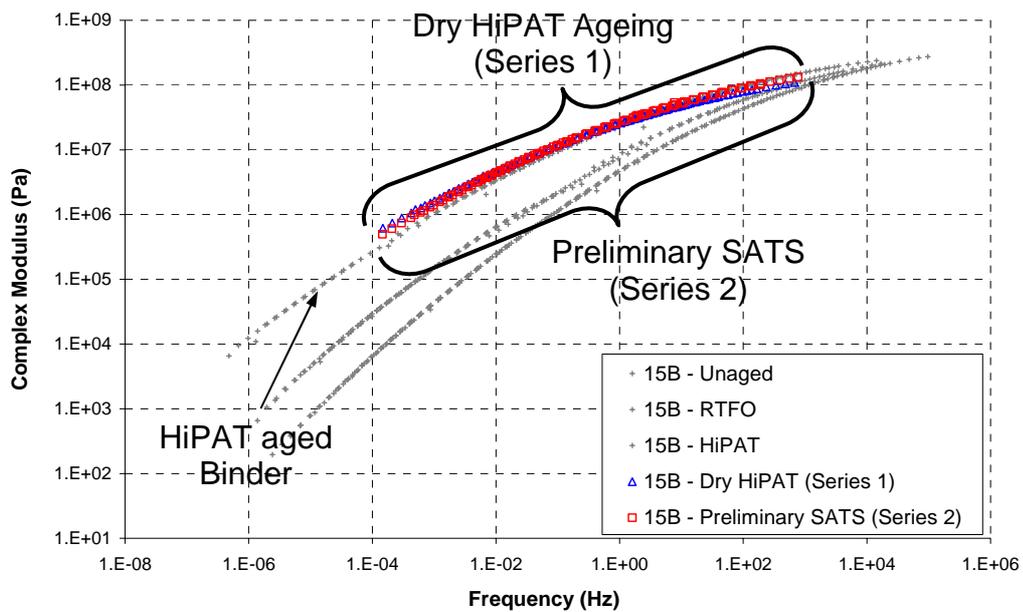


Figure 9.1 – G^* master curves of recovered binders from series 1 & 2.

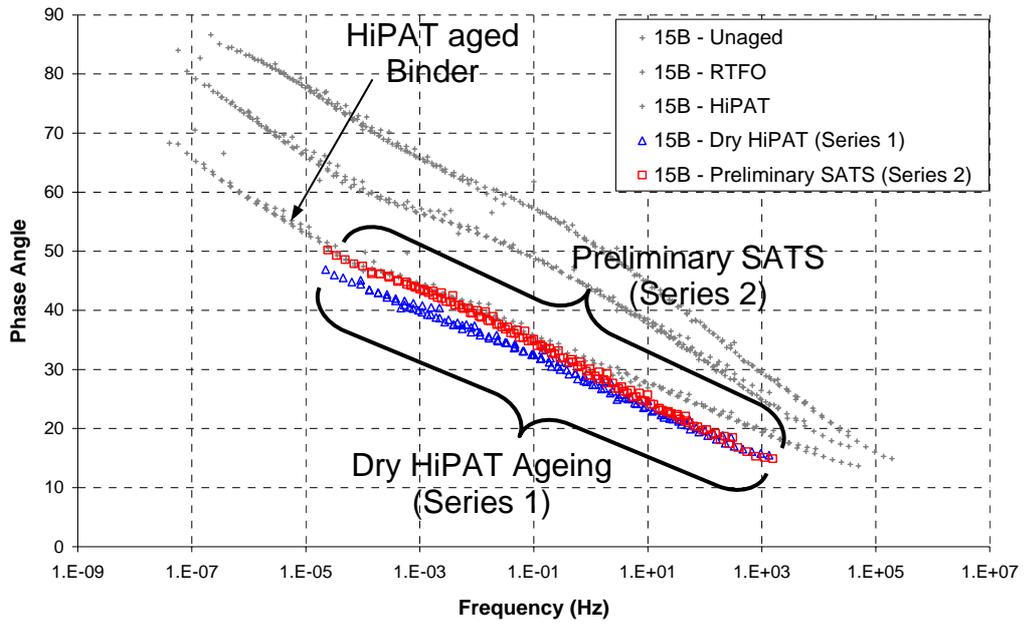


Figure 9.2 – δ master curves of recovered binders from series 1 & 2.

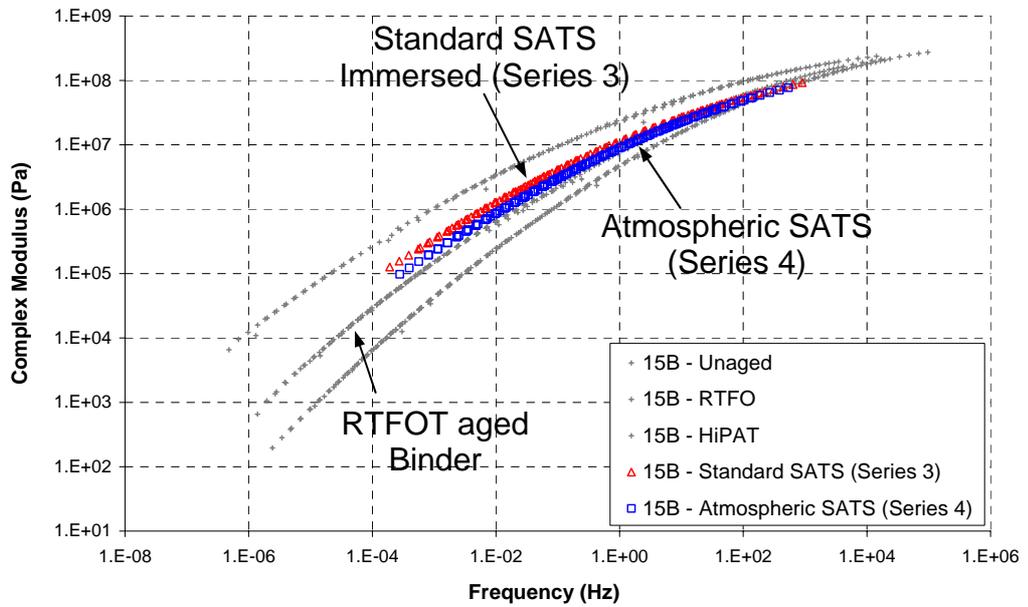


Figure 9.3 – G^* master curves of recovered binder from series 3 & 4.

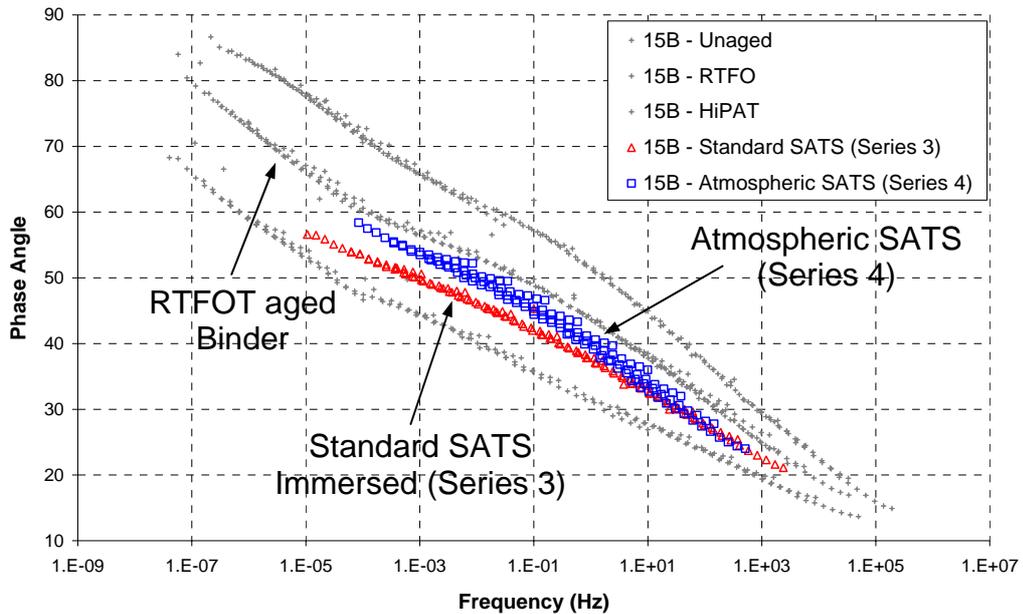


Figure 9.4 – δ master curves of recovered binder from series 3 & 4.

Figures 9.1 and 9.2 clearly show that the binders recovered after the preliminary SATS protocol (Series 2, initial vacuum saturation only, no added water) and the Dry HiPAT ageing process (Series 1) typically have very similar rheological characteristics to those of the HiPAT aged binder indicating a similar amount of ageing.

Figures 9.3 and 9.4 present the recovered binder DSR results from Atmospheric SATS (no added pressure) and immersed specimens from the standard SATS (2.1 MPa, 85°C, 65 hrs). Both sets of results typically showed considerably less ageing than the Series 1 and 2 (i.e. more comparable to RTFOT aged binder). It is interesting to note that the recovered binder from fully immersed specimens showed considerably less ageing than those from the Series 1 and 2 although the test conditions (pressure, temperature and test duration) are identical. It is thought that the specimens were ‘insulated’ from air by the surrounding water resulting in much less air contact than those from the specimens above water level.

9.4 Binder Recovery and Testing (Stage 2)

9.4.1 Binder ageing using the SATS PAV

The objective of these tests is to investigate whether the SATS PAV (ϕ 150 mm \times 435 mm, internal dimensions) can produce equivalent binder ageing to the standard PAV (ϕ 200 mm \times 230 mm, internal dimensions) used for the binder ageing. A smaller size pan (ϕ 87 mm, compared to standard ϕ 140 mm) containing 19 g of RTFOT of aged binder (to have the same binder film thickness of 3.2 mm) was placed in the SATS PAV and the same HiPAT protocol (2.1 MPa, 85°C, 65 hours) was applied. From results presented in Figures 9.5 and 9.6 it can be confirmed that, as expected, the HiPAT aged binder using SATS PAV are comparable to that of the standard HiPAT aged binder, regardless of the different vessel size.

9.4.2 Binder ageing on a function of specimen position

To investigate the binder ageing variation along the specimen position, binders were individually recovered from a set of 15B Acidic specimens (five specimens from Positions 1 ~ 6, Position 4 is empty) after it had been SATS tested (standard SATS with the new tray, as described in Section 7.2.2). Results are presented in Figures 9.7 and 9.8.

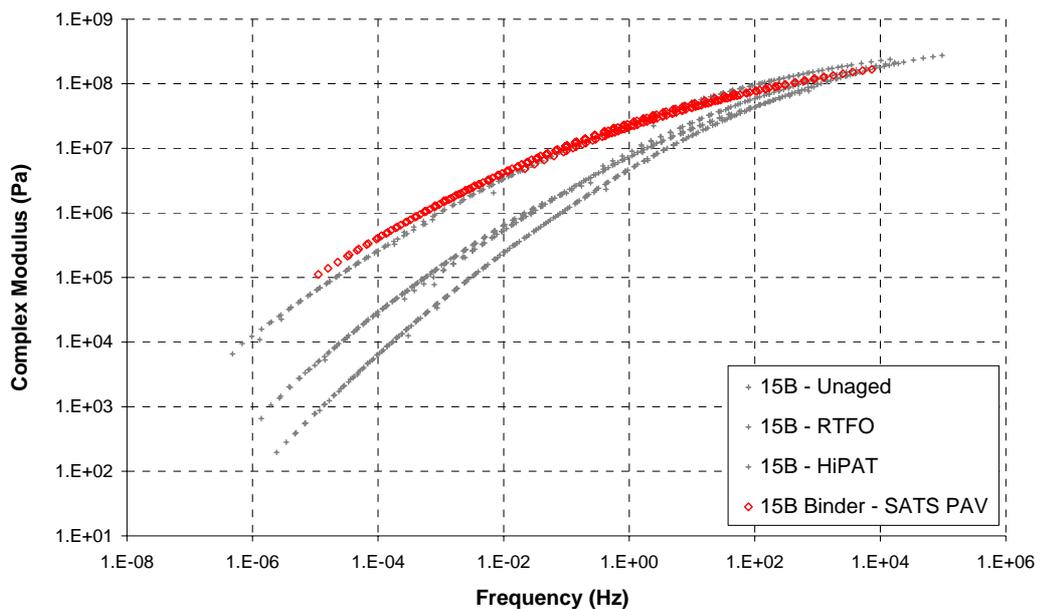


Figure 9.5 – G^* master curves of HiPAT aged binder using SATS PAV.

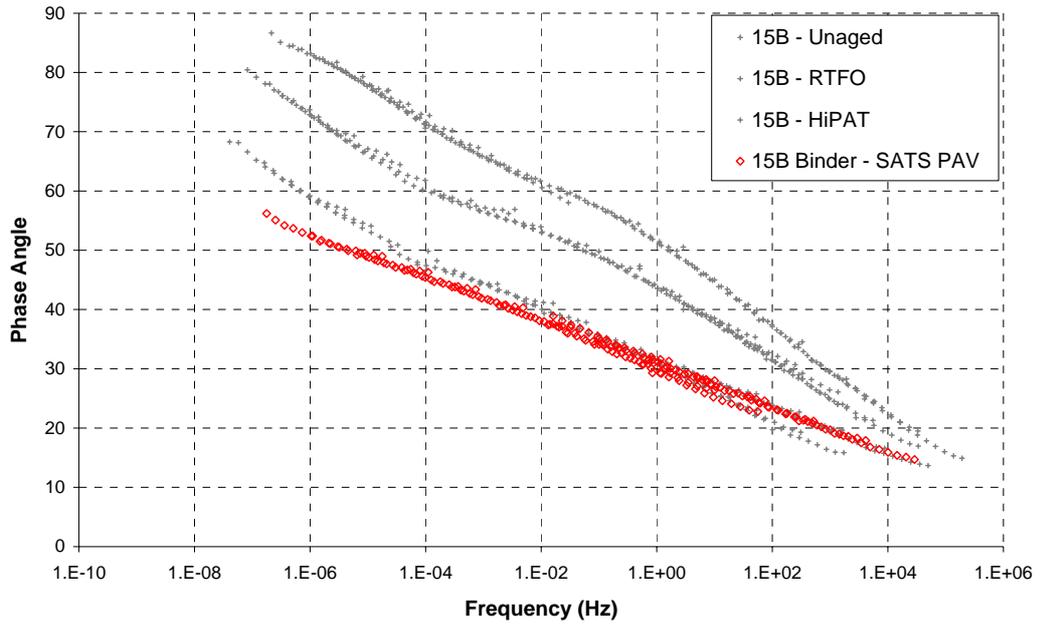


Figure 9.6 – δ master curves of HiPAT aged binder using SATS PAV.

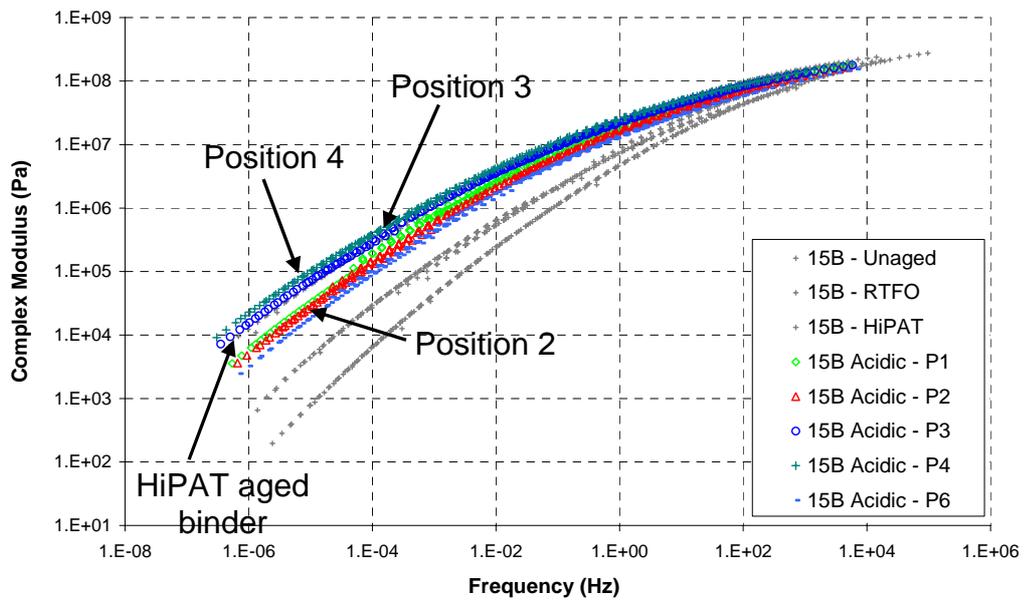


Figure 9.7 – G^* master curves of recovered binders from 15B Acidic SATS.

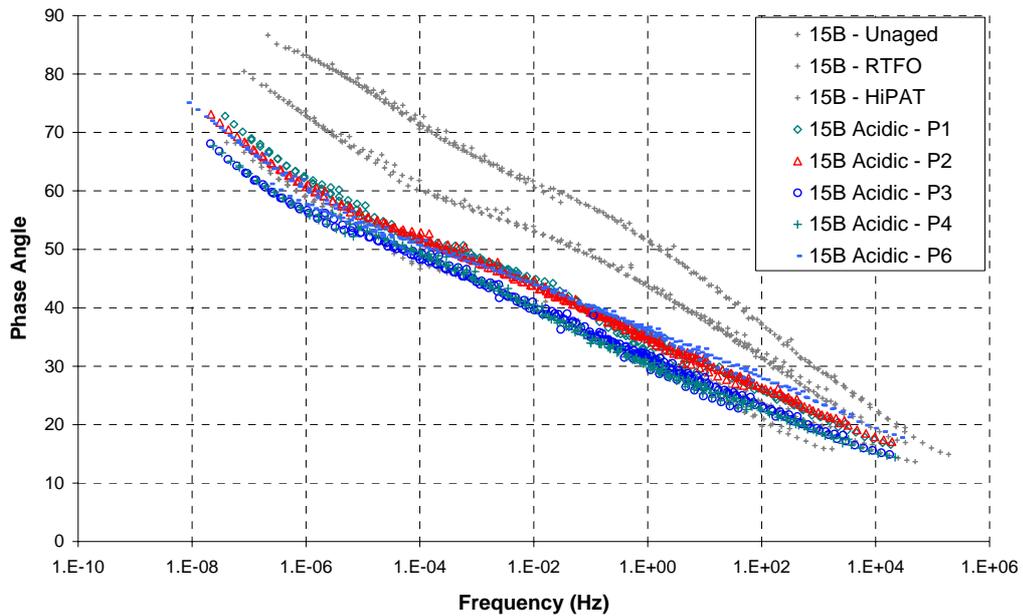


Figure 9.8 – δ master curves of recovered binders from 15B Acidic SATS.

From Figures 9.7 and 9.8, it is clear that there is a close correlation between the level of ageing and the specimen position. For example, from Figure 9.7, recovered binders from the specimens in Positions 1 or 2 show slightly lower complex modulus than HiPAT aged binder while the specimens in Positions 3 or 4 show slightly higher values. Since the dominantly affected variable by the specimen position is the retained saturation, the level of ageing is considered to be mostly dependent on the retained saturation. For a simpler presentation, complex modulus (G^*) at a particular frequency (@ 0.4 Hz) normalised using the complex modulus of HiPAT aged binder (G^*_{HiPAT} @ 0.4 Hz) are plotted against the retained saturation values from the corresponding specimens in Figure 9.9. Also the complex modulus (G^* @ 0.4 Hz) of recovered binder from the Dry HiPAT aged specimens (see Section 9.3 – Series 1) is added to the same graph, Figure 9.9 (data point = 0.0, 1.17).

From Figure 9.9 specimens from Positions 1, 2 and 6 (immersed), where the retained saturation are generally higher, show less ageing than HiPAT aged binder, whereas specimens from Positions 3 and 4, where the retained saturation are generally lower, show relatively more ageing than HiPAT aged binder. The retained saturation of the Dry HiPAT aged specimen is considered to be '0%' (x – axis) and, as expected,

showed a bit higher value (1.17, y – axis) than those from the saturated test. From Figure 9.9, a reasonable linear correlation ($R^2 = 0.84$) between retained saturation and ageing ratio (G^* / G^*_{HiPAT}) is obtained, as follows;

$$y = -0.0057 x + 1.1942 \quad (9.1)$$

where, y = ageing ratio (G^* / G^*_{HiPAT}) and x = retained saturation (%).

Thus, from Equation 9.1, it can be predicted that the HiPAT equivalent ageing (i.e. $G^* / G^*_{HiPAT} = 1.0$) can be achieved when the retained saturation is approximately 34%.

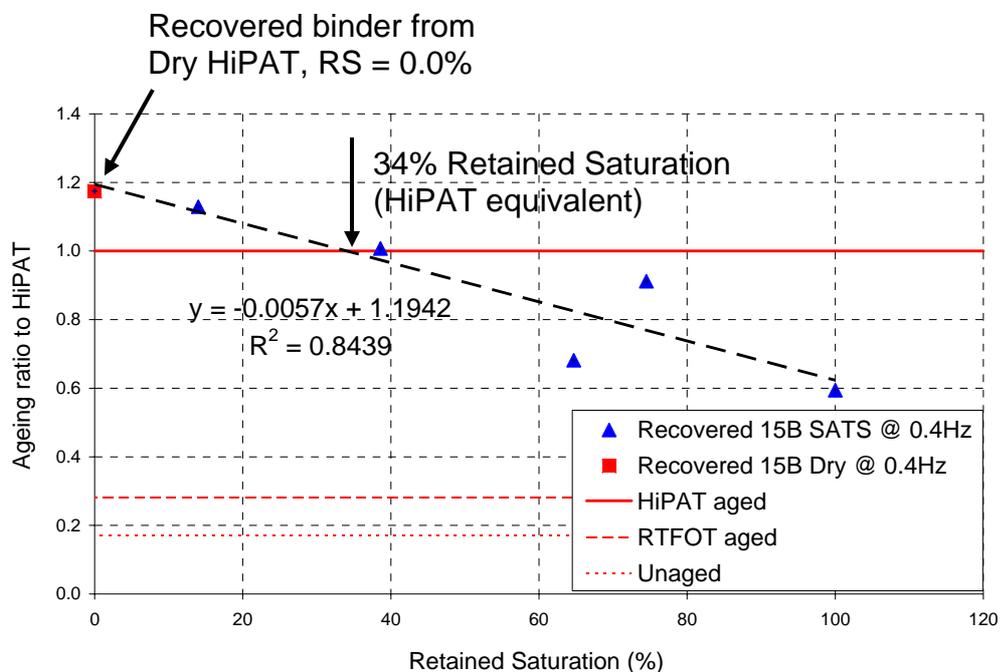


Figure 9.9 – Ageing ratio (@ 0.4 Hz) against the retained saturation.

9.4.3 Binder ageing from Basic and Acidic specimens

The objective of these tests is to investigate the effect of aggregates type on binder ageing. From Chapter 5 (Section 5.4) a set of recovered binder was obtained from 15B Acidic and 15B Basic SATS tested specimens (Positions 1 and 2 mixed). Results are presented in Figure 9.10 and 9.11 indicating virtually identical properties.

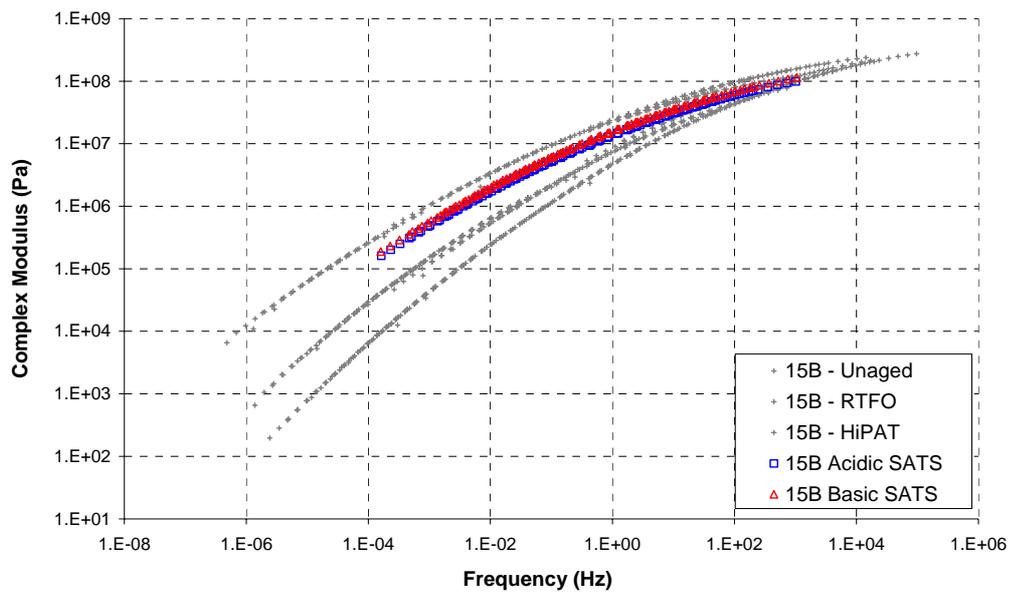


Figure 9.10 – G^* master curves of recovered binder from 15B Acidic/Basic.

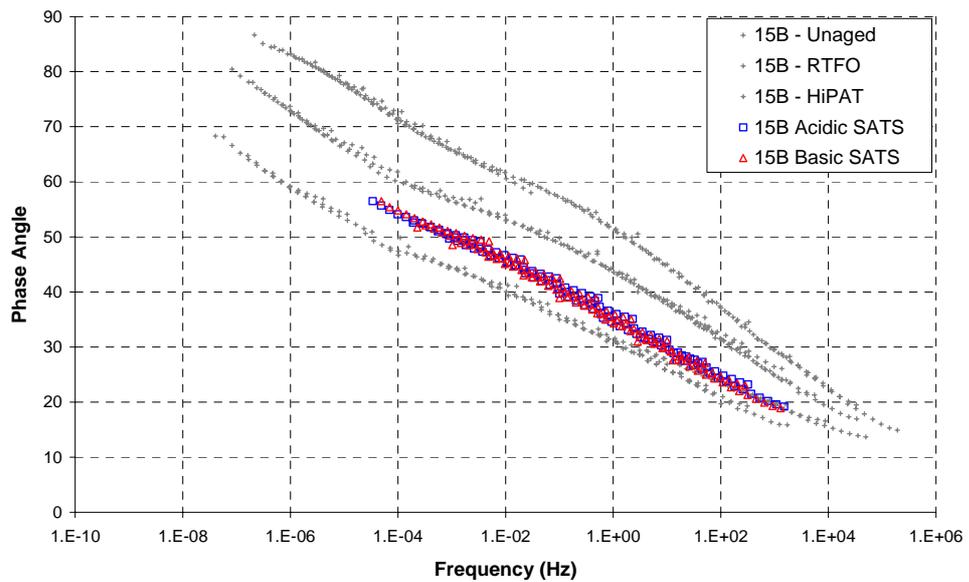


Figure 9.11 – δ master curves of recovered binders from 15B Acidic/Basic.

9.4.4 Applied pressure and binder ageing

The objective of these tests is to investigate the pressure effect on binder ageing. From Chapter 7 (Section 7.3.1) recovered binder was obtained from 15B Acidic half pressure SATS (position 1 and 2 mixed). Results are presented in Figure 9.12 and 9.13 (also compared to Atmospheric SATS in Section 9.3 – Series 4).

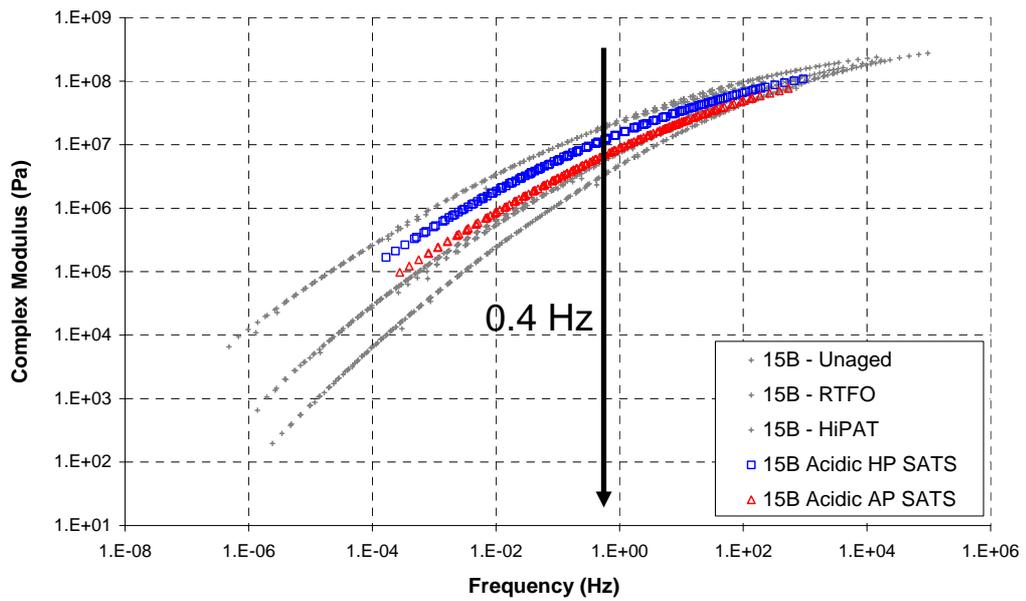


Figure 9.12 – G^* master curves of recovered binder from half pressure SATS.

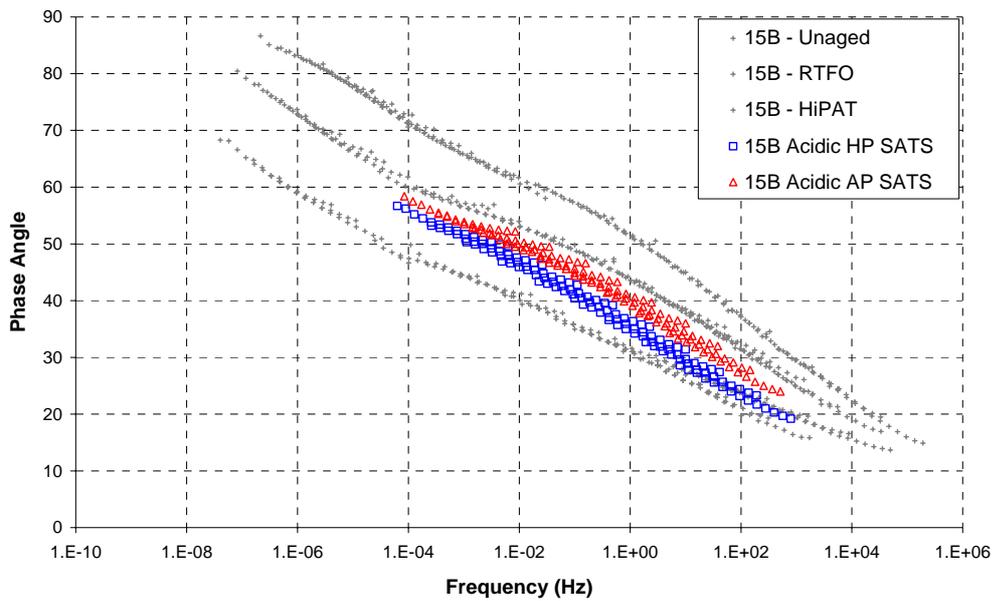


Figure 9.13 – δ master curves of recovered binders from half pressure SATS.

The ageing ratios (G^* / G^*_{HiPAT} @ 0.4 Hz, 0.64 for half pressure SATS, 0.36 for Atmospheric SATS) obtained at each retained saturation (85% and 69%) were normalised to a retained saturation of 34%, where the standard SATS showed ageing ratio of 1.0 (@ 0.4 Hz), using Equation 9.1. Results are presented in Figure 9.14 where ‘0.0 MPa’ (x – axis) represents atmospheric pressure.

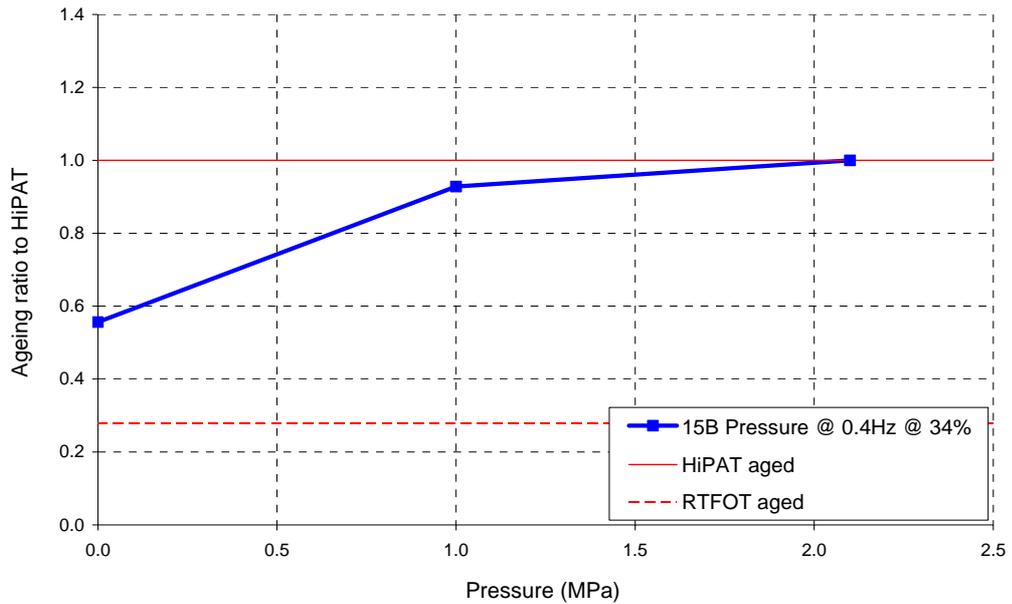


Figure 9.14 – Ageing against applied pressure @ 0.4 Hz @ 34% retained saturation.

9.4.5 Test duration and binder ageing

The objective of these tests is to investigate the correlation of test duration and binder ageing. From Chapter 7 (Section 7.4) a set of recovered binder was obtained from 15B Acidic 24 hours and 4 hours SATS (position 1). Results are presented in Figure 9.15 and 9.16.

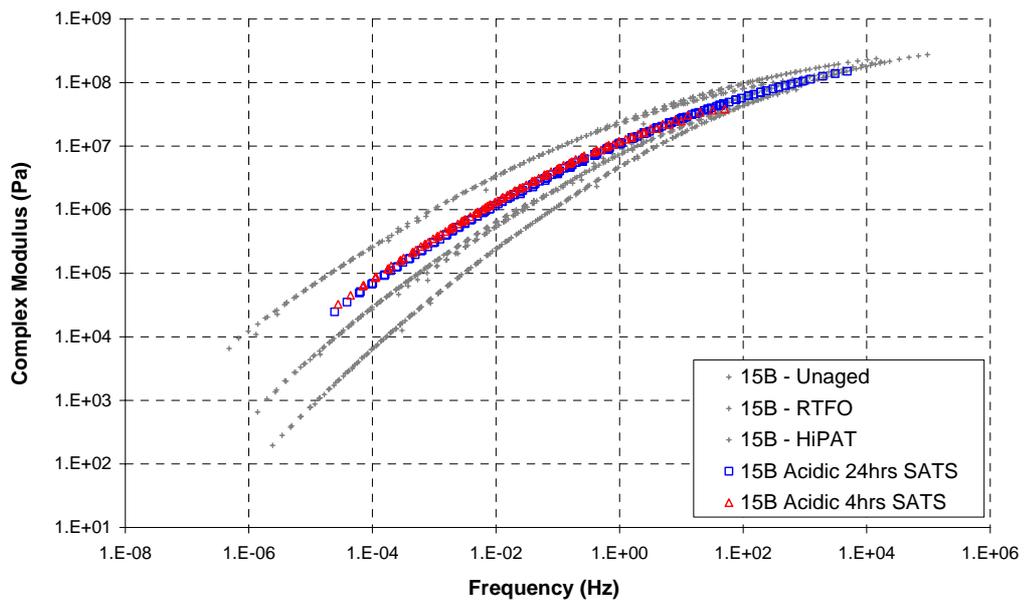


Figure 9.15 – G^* master curves of recovered binder from 24 and 4 hours SATS.

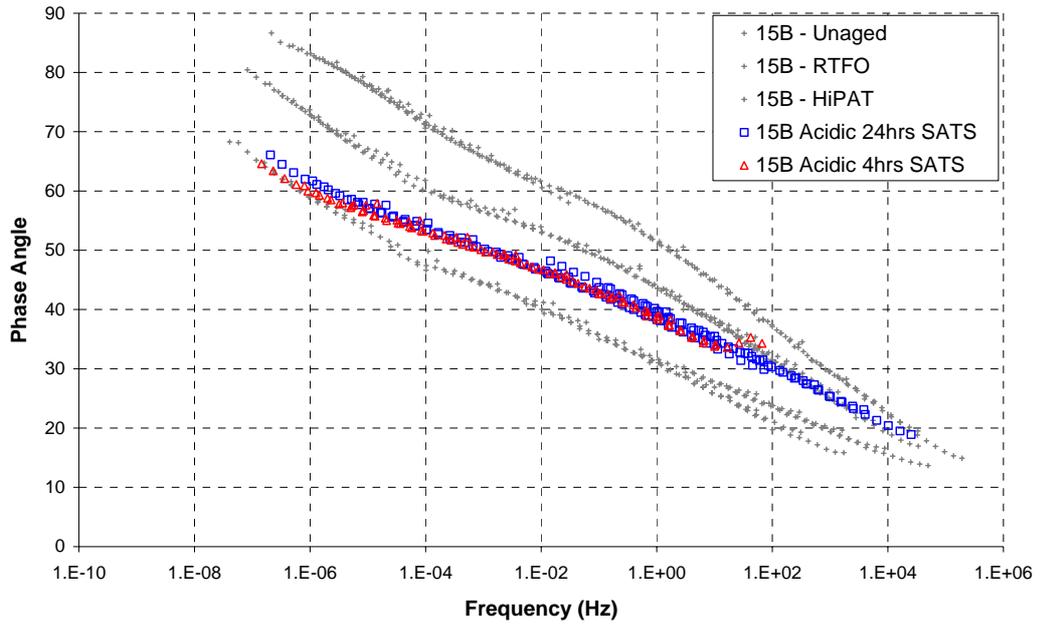


Figure 9.16 – δ master curves of recovered binders from 24 and 4 hours SATS.

The normalised ageing ratio (to a retained saturation of 34%) against the test duration, as described in the previous section, are presented in Figure 9.17.

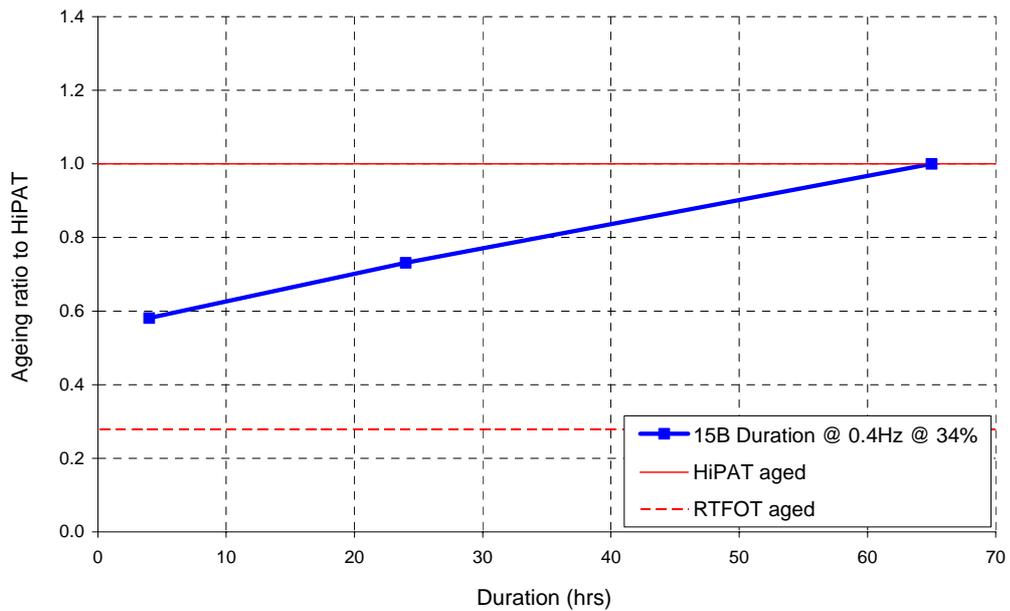


Figure 9.17 – Ageing against test duration @ 0.4 Hz @ 34% retained saturation.

9.4.6 Test temperature and binder ageing

The objective of these tests is to investigate the correlation of the applied temperature and binder ageing. From Chapter 7 (Section 7.5) a set of recovered binder was obtained from 15B Acidic 60°C SATS (position 1). Results are presented in Figure 9.18 and 9.19.

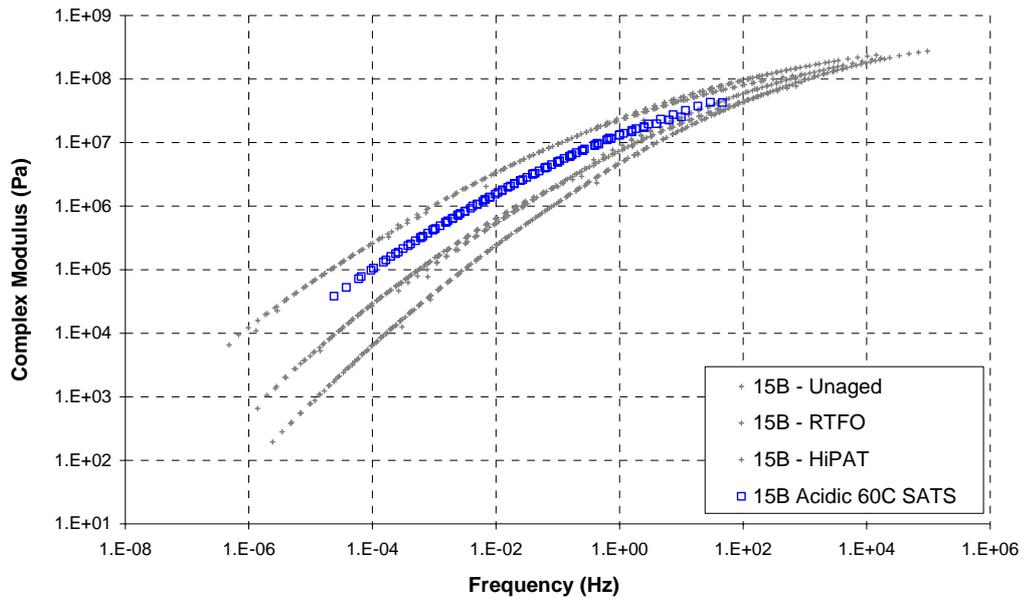


Figure 9.18 – G^* master curves of recovered binder from 60°C SATS.

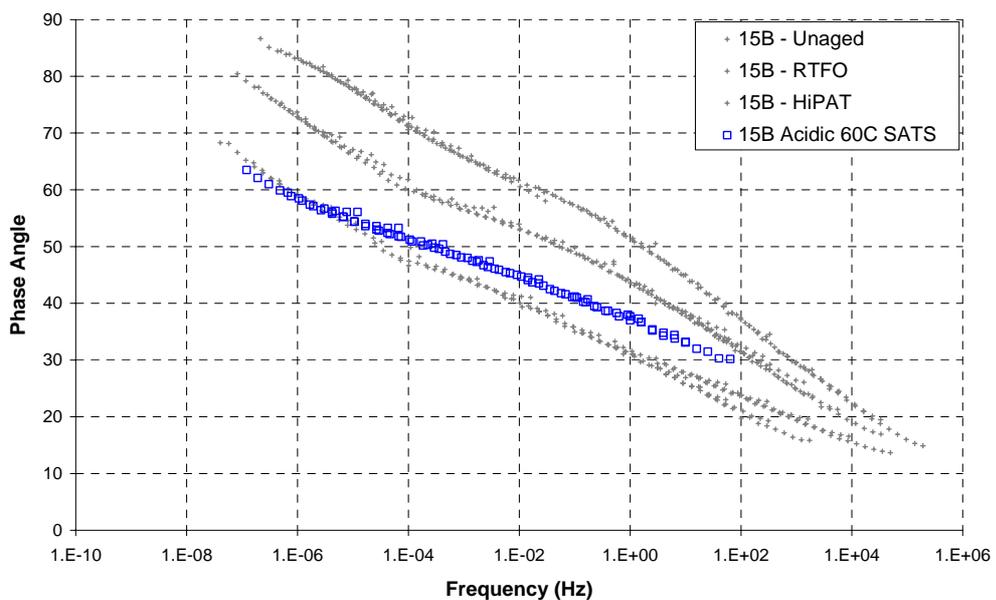


Figure 9.19 – δ master curves of recovered binders from 60°C SATS.

The normalised HiPAT ageing ratio (to a retained saturation of 34%) against the test temperature, as described in Section 9.4.4., are presented in Figure 9.20.

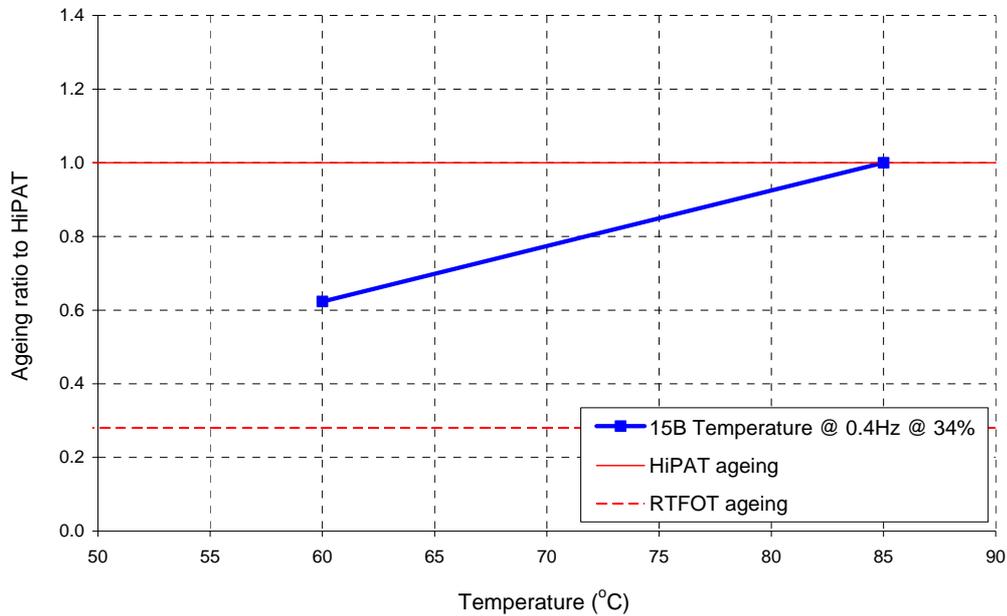


Figure 9.20 – Ageing against test temperature @ 0.4 Hz @ 34% retained saturation.

9.5 Summary and Conclusions

An extensive investigation on recovered binders from various SATS conditions were performed using the DSR test. Comparison of the preliminary SATS and Dry HiPAT ageing test showed similar level of ageing regardless of the existence of water. However, the fully immersed specimen from the standard SATS and the partially saturated specimens (above the water level) from the Atmospheric SATS commonly showed much lower levels of ageing compared to the partially saturated specimens (above the water level) from the standard SATS.

A more careful investigation on the ageing variation along the specimen position revealed that there is a clear trend of ageing level dependent on the specimen position. This is thought to be due to the saturation during the process. Air voids in a highly saturated specimen will have a more amount of water filled in the voids which prevents free air contact throughout the SATS process, therefore result in a relatively lower level of ageing. From a limited set of results a representative line

was fitted and a retained saturation of 34% has been found to be the HiPAT equivalent ageing condition (i.e. $G^* / G^*_{HiPAT} = 1.0$) for the 15B Acidic control mixture from the standard SATS protocol.

A series of recovered binder from various SATS conditions (lower pressures, shorter test durations and lower temperatures) revealed that, to achieve a HiPAT equivalent binder ageing from the SATS protocol within the same testing duration (65 hours), the pressure and temperature conditions used in the standard SATS (2.1 MPa and 85°C) are necessary.

Chapter 10. Data Interpretation

10.1 Retained Saturation

From a large number of SATS data observed in the previous chapters, it was noted that some results from the SATS test showed retained saturations higher than 100%. Saturation over 100% is not a realistic value, indicating a certain volumetric change took place during the test. For an example, a 15B Acidic result from Atmospheric SATS test performed in Section 7.3.2 (data not presented in Figure 7.6) showed a retained saturation value of 141% (from the immersed specimen) with clearly visible swelling (near disintegration stage, retained stiffness of 0.1). As the apparent volume of the specimen was expanded, the internal voids content was also supposedly increased.

A series of SATS tested specimens were selected and the air voids were measured again (referred to as ‘retained AV’) using the identical method to that described in Section 5.2.2. Table 10.1 (Column 5) shows the retained AV of 15B Acidic specimens which have been tested using the standard SATS, half pressure SATS or Atmospheric SATS protocol (see Section 7.3). The retained AV data of 15B Basic specimens are also presented in Table 10.2 (Column 5). Tables 10.1 and 10.2 also show the retained saturations calculated using the ‘initial AV’ (referred to as ‘ $Sat_{nominal}$ ’, in Column 6) and the retained saturation calculated using the ‘retained AV’ (referred to as ‘ Sat_{actual} ’, in Column 7). The reason of selecting specimens from SATS tests using different pressures was to observe whether the applied pressure particularly contributed to change the volumetric properties.

From Table 10.1 (15B Acidic series), it is noted that the retained saturations ($Sat_{nominal}$) over 100% are commonly associated with very low retained stiffnesses (≤ 0.1), regardless of the magnitude of applied pressure. This low retained stiffness clearly indicates that the specimens are in a near disintegrating state with a much increased internal voids due to the creation of cracks or adhesion failure between binder and aggregate. It can be found from Table 10.2 that the 15B Basic specimen from Atmospheric SATS displays virtually unchanged air voids resulting in

unchanged retained saturations (Nominal/Actual ratio ≈ 1.0). 15B Basic specimens from the pressurised SATS protocols commonly show increased Nominal/Actual ratio (1.2 ~ 1.4) due to a pressure related damage (see Section 7.3.2 for a more discussion). This fact generally agrees with the finding from Chapters 7 and 8, where 15B Acidic mixtures are considered to be pressure insusceptible and 15B Basic mixtures to be pressure susceptible. However, in general, it should be noted that the bigger volumetric change (i.e. higher Nominal/Actual ratio) is associated with the lower retained stiffness values (i.e. more damage), regardless of the applied pressure or material types.

Table 10.1 – Air voids and retained saturation chart of 15B Acidic specimens.

| 15B Acidic control | | Retained stiffness | Initial AV | Retained AV | $Sat_{nominal}$ (%) | Sat_{actual} (%) | Nominal /Actual |
|--------------------|---|--------------------|------------|-------------|---------------------|--------------------|-----------------|
| Standard SATS | 1 | 0.73 | 8.02 | 9.57 | 5.5 | 4.5 | 1.21 |
| | 2 | 0.16 | 9.24 | 12.77 | 79.7 | 55.5 | 1.44 |
| | 3 | 0.01 | 7.83 | 15.53 | 142.1 | 65.6 | 2.17 |
| Half P SATS | 1 | 0.05 | 7.89 | 11.72 | 96.8 | 62.5 | 1.55 |
| | 2 | 0.05 | 9.19 | 11.94 | 68.5 | 51.2 | 1.34 |
| | 3 | 0.13 | 7.36 | 10.59 | 72.5 | 48.6 | 1.49 |
| | 4 | 0.26 | 7.31 | 9.72 | 39.5 | 31.2 | 1.27 |
| Atmospheric SATS | 1 | 0.27 | 9.51 | 10.57 | 81.6 | 74.7 | 1.09 |
| | 2 | 0.25 | 7.98 | 9.60 | 92.4 | 77.9 | 1.19 |
| | 3 | 0.10 | 7.93 | 11.73 | 140.8 | 93.0 | 1.51 |

Table 10.2 – Air voids and retained saturation chart of 15B Basic specimens.

| 15B Basic control | | Retained stiffness | Initial AV | Retained AV | $Sat_{nominal}$ (%) | Sat_{actual} (%) | Nominal /Actual |
|-------------------|---|--------------------|------------|-------------|---------------------|--------------------|-----------------|
| Standard SATS | 1 | 0.77 | 7.43 | 10.13 | 27.0 | 19.2 | 1.40 |
| Half P SATS | 1 | 0.80 | 9.68 | 11.28 | 50.6 | 42.6 | 1.19 |
| | 2 | 0.70 | 7.30 | 9.24 | 53.7 | 41.5 | 1.29 |
| | 3 | 0.77 | 7.32 | 9.18 | 84.8 | 66.3 | 1.28 |
| Atmospheric SATS | 1 | 0.92 | 8.71 | 8.88 | 50.7 | 49.7 | 1.02 |
| | 2 | 0.93 | 8.50 | 8.52 | 56.1 | 55.9 | 1.00 |
| | 3 | 0.93 | 8.91 | 8.95 | 42.6 | 42.4 | 1.00 |
| | 4 | 0.97 | 10.4 | 10.13 | 32.0 | 31.7 | 1.01 |
| | 5 | 0.97 | 9.61 | 9.93 | 61.3 | 59.1 | 1.04 |

Figure 10.1 shows a plot of Nominal/Actual ratio against retained stiffness of individual specimens (from Tables 10.1 and 10.2). Two separate curve fittings (in a power form) were performed on 15B Acidic data (Equation 10.1) and 15B Basic data (Equation 10.2), as follows.

- 15B Acidic ‘Nominal/Actual’ ratio (y) and retained stiffness (x)

$$y = 1.043 x^{-0.1394} \quad (10.1)$$

- 15B Basic ‘Nominal/Actual’ ratio (y) and retained stiffness (x)

$$y = 0.966 x^{-1.0046} \quad (10.2)$$

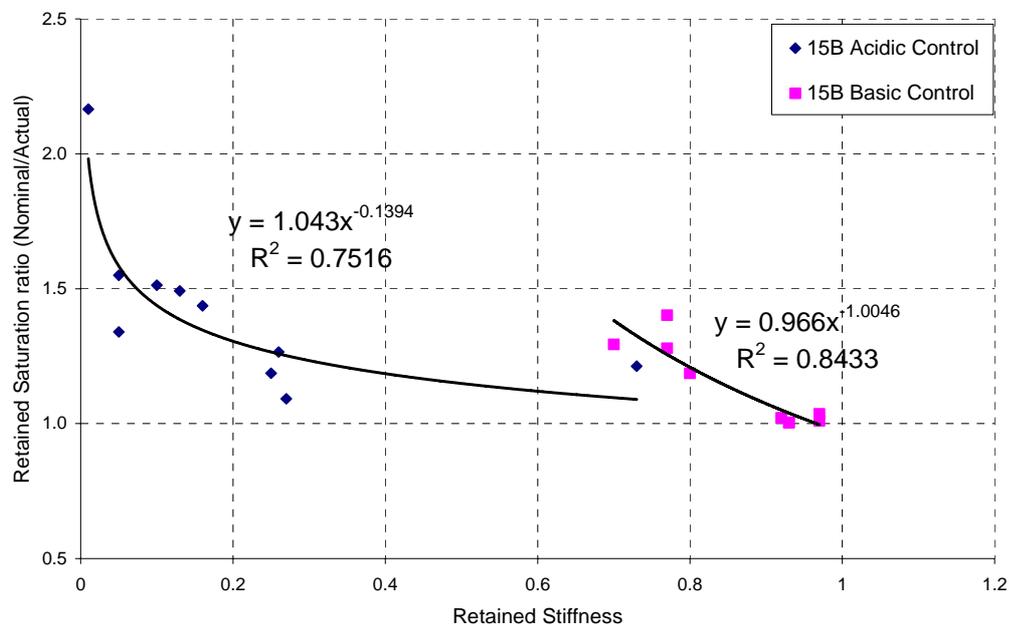


Figure 10.1 – Correlation between Nominal/Actual ratio and retained stiffness.

This is to get a reasonable correlation ($R^2 > 0.75$) between the Nominal/Actual ratios and the corresponding retained stiffnesses. Equations 10.1 and 10.2 were used to calculate the Sat_{actual} values from a typical SATS data plot (from Section 5.4), as presented in Figure 10.2. This figure clearly displays that the Sat_{actual} values are generally lower than the $Sat_{nominal}$ values, and the difference varies depending on the corresponding retained stiffnesses (i.e. higher retained stiffness \rightarrow smaller difference, lower retained stiffness \rightarrow bigger difference). The Sat_{actual} (i.e. retained saturation based on ‘retained AV’) of 15B Acidic and 15B Basic SATS test data

shown in Figure 5.7 (see Section 5.5) are compared with its $Sat_{nominal}$ data (i.e. retained saturation) in Figure 10.3.

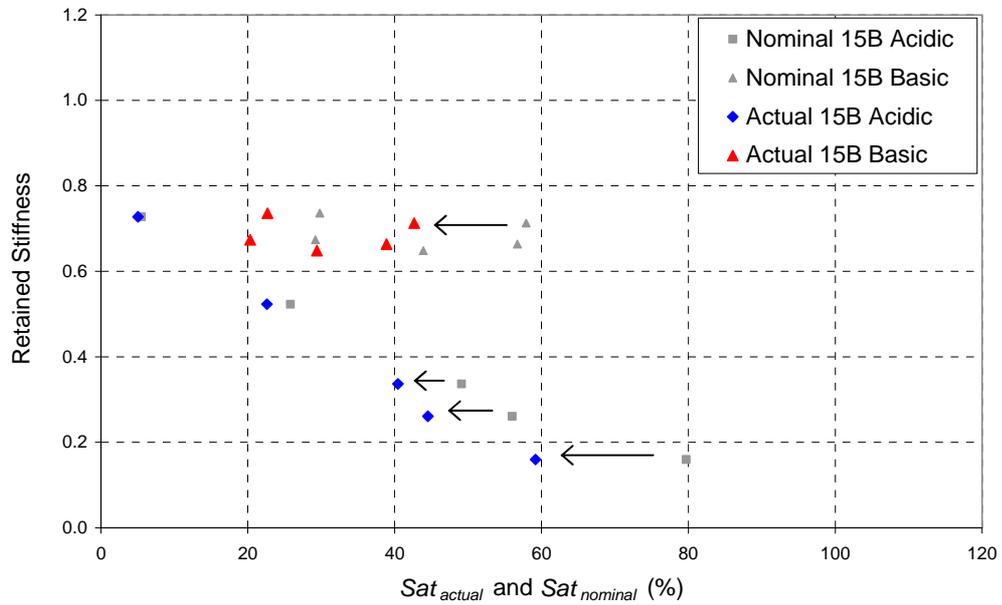


Figure 10.2 – Examples of Sat_{actual} and $Sat_{nominal}$ of SATS test.

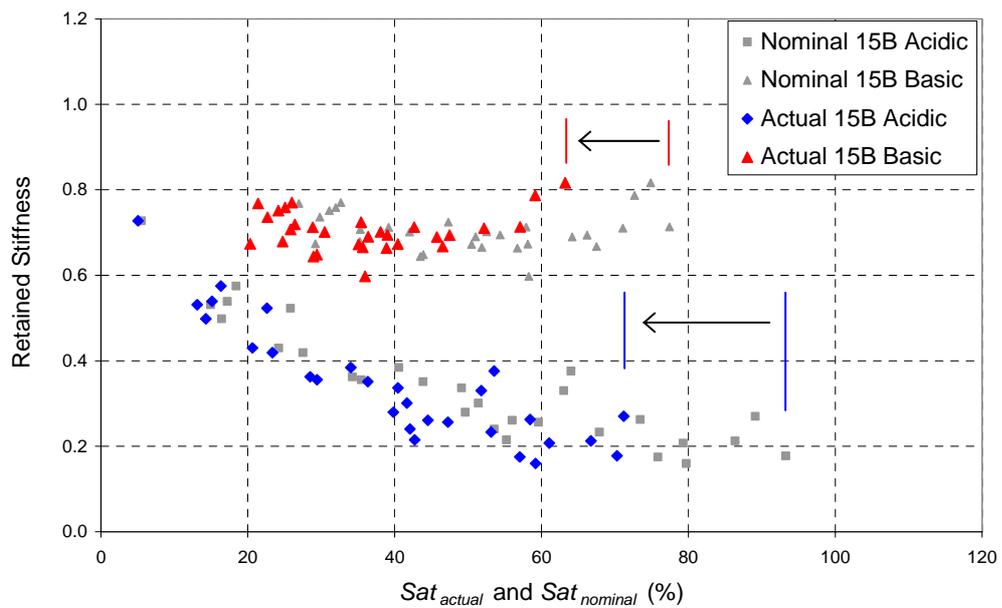


Figure 10.3 – Comparison of Sat_{actual} and $Sat_{nominal}$ of the main SATS test series.

Therefore, from Figures 10.2 and 10.3, it can be concluded that, for the 15B control materials used in the main series in Section 5.5, the presented retained saturations (i.e. $Sat_{nominal}$) are considered to be up to 20% higher (for 15B Acidic) and up to 15% higher (for 15B Basic) than the volumetric change considered ‘actual’ retained saturations (i.e. Sat_{actual}).

10.2 Retained Stiffness and Moisture Sensitivity

The retained stiffness data (referred to as ‘nominal’ retained stiffness) obtained from the SATS test is calculated based on the initial ‘un-conditioned’ stiffness of the specimen, as follows.

$$RS_{nominal} = \frac{S_{conditioned}}{S_{initial}} \quad (10.3)$$

where;

$RS_{nominal}$ = Nominal retained stiffness of a specimen (MPa/MPa)

$S_{initial}$ = Stiffness modulus of un-conditioned specimen (MPa)

$S_{conditioned}$ = Stiffness modulus of conditioned specimen (MPa)

For an example, if a specimen shows an initial stiffness modulus ($S_{initial}$) of 10,000 MPa and a conditioned stiffness of 5,000 MPa after the SATS test ($S_{conditioned}$), the retained stiffness ($RS_{nominal}$) of the specimen is calculated to be 0.5 (5,000 MPa / 10,000 MPa).

However, the binder in the mixture is hardened (aged), as the SATS protocol ages the specimen as well as inducing moisture damage (see Chapter 9 for the recovered binder assessment). Thus, a certain degree of mixture stiffness increase (by **ageing factor**) is expected during the SATS process, although this appears to be masked by the stiffness reduction caused by moisture damage (by **moisture factor**). The trial of Dry HiPAT test (see Section 8.3.5 for more details of the test setup) was used to investigate the mixture hardening (ageing factor) during the SATS process, without the presence of moisture. However, it is difficult to accept that the result (1.15 for 15B Acidic, 1.30 for 15B Basic) fully represents the expected stiffness increase

entirely due to the ‘ageing factor’ of SATS protocol, as a certain degree of pressure damage (by **pressure factor**) is presumed to be included in the results (see Section 7.6 for more discussions on the pressure damage).

Therefore the SATS conditioned stiffness modulus ($S_{conditioned}$) of a specimen can be expressed as follows.

$$S_{conditioned} = S_{initial} \times f_{ageing} \times f_{pressure} \times f_{moisture} \quad (10.4)$$

where;

$S_{initial}$ = Stiffness modulus of unconditioned specimen (MPa)

$S_{conditioned}$ = Stiffness modulus of conditioned specimen (MPa)

f_{ageing} = Ageing factor (age hardening of mixture)

$f_{pressure}$ = Pressure factor (pressure damage)

$f_{moisture}$ = Moisture factor (moisture sensitivity)

Consequently, once the ageing factor (f_{ageing}) and pressure factor ($f_{pressure}$) are identified, the ‘true’ moisture sensitivity ($f_{moisture}$) of a SATS conditioned specimen can be obtained as follows.

$$f_{moisture} = \frac{S_{conditioned}}{S_{initial} \times f_{ageing} \times f_{pressure}} = \frac{RS_{nominal}}{f_{ageing} \times f_{pressure}} \quad (10.5)$$

10.3 Ageing Hardening (f_{ageing})

The binder in the mixture from the SATS test is expected to be aged equivalent to the HiPAT binder ageing level (in a specific retained saturation of 34%, see Section 9.4.2). It is easily predicted that, if the binder in the mixture hardens (binder stiffness increases), the mixture stiffness will also increase. However, the correlation between the binder stiffness increase and the resultant mixture stiffness increase does not seem to be linear. For an example, the DSR test performed on the HiPAT aged 15B binder shows approximately 3.5 times higher complex modulus (G^*) than that of the RTFOT aged 15B binder (see Appendix C). If this ratio is directly applied on

the mixture stiffness, then the stiffness of 15B Acidic or Basic specimens (due to binder ageing) would increase up to 35,000 MPa ($3.5 \times$ unconditioned mixture stiffness ($S_{initial}$) of 10,000 MPa), which is an unrealistically high value for the bituminous materials (the binder of ‘unconditioned’ specimens was defined to have same complex modulus to that of RTFOT aged binder).

To find the correlation between binder stiffness change and resultant mixture stiffness change, the **Shell BANDS 2.0** software [Shell Bitumen, 1998] has been used. This programme is based on a binder stiffness prediction nomograph produced by Van der Poel [1954] and a mixture stiffness prediction nomograph produced by Bonnaure et al. [1977]. Van der Poel [1954] produced the nomograph (see Figure 10.4) based on the data from series of stiffness determination tests conducted on forty bitumen binders at many temperatures and frequencies, using both creep and dynamic tests. This nomograph is to predict the stiffness modulus of a bitumen for any conditions of temperature and loading time, using only Penetration and Softening point data, within a practically acceptable range (factor of 2). Bonnaure et al. [1977] conducted series of dynamic tests (2 point and 3 point bending tests) on twelve bituminous mixtures of vastly different composition but representative of mixtures used in a number of countries. As a result, they produced the nomograph shown in Figure 10.5 for predicting the stiffness modulus of a bituminous material. The input required for this nomograph are a) stiffness modulus of bitumen (Pa) obtained from the Van der Poel nomograph, b) volume of bitumen (V_b , %) and c) volume of mineral aggregates (V_a , %). It should be noted that the stiffness modulus of bitumen was from the ‘recovered’ binder from the tested mixtures, not from the initial ‘fresh’ binder to make the mixtures.

10.3.1 Binder stiffness prediction (Van der Poel Nomograph)

The Penetration and Softening point data of unaged, RTFOT aged or HiPAT aged 15B binders (see Table 10.3, data also presented in Appendix C) are used to predict binder stiffness using the **BANDS** software. As observed in Table 10.3, the measured ‘tensile’ stiffnesses ($G^* \times 3$) from DSR test (under 2 Hz sinusoidal loading and 20°C test temperature) are considerably lower (around 60%) than the **BANDS** predicted binder stiffnesses (using Penetration and Softening point test data, for a

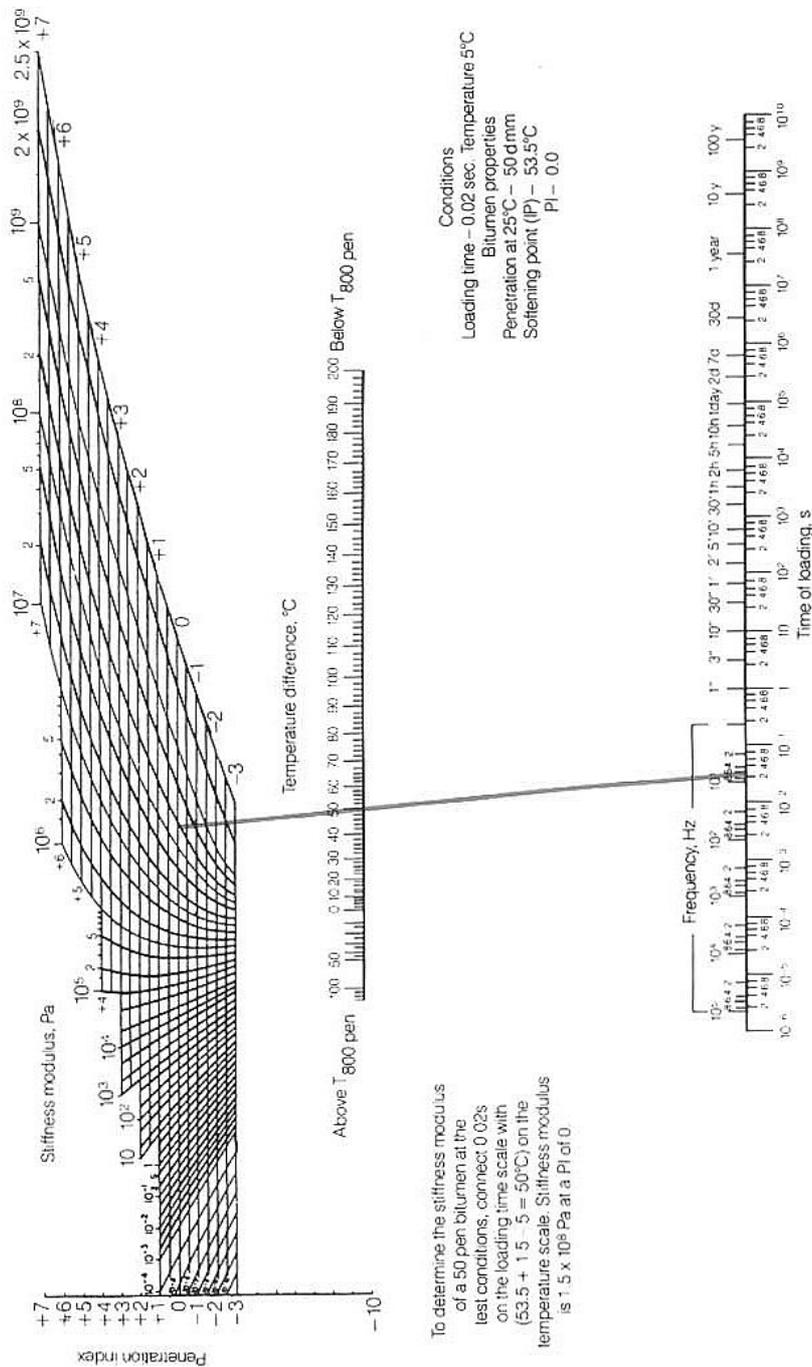


Figure 10.4 – Nomograph for determining stiffness modulus of bitumens [Van der Poel, 1954].

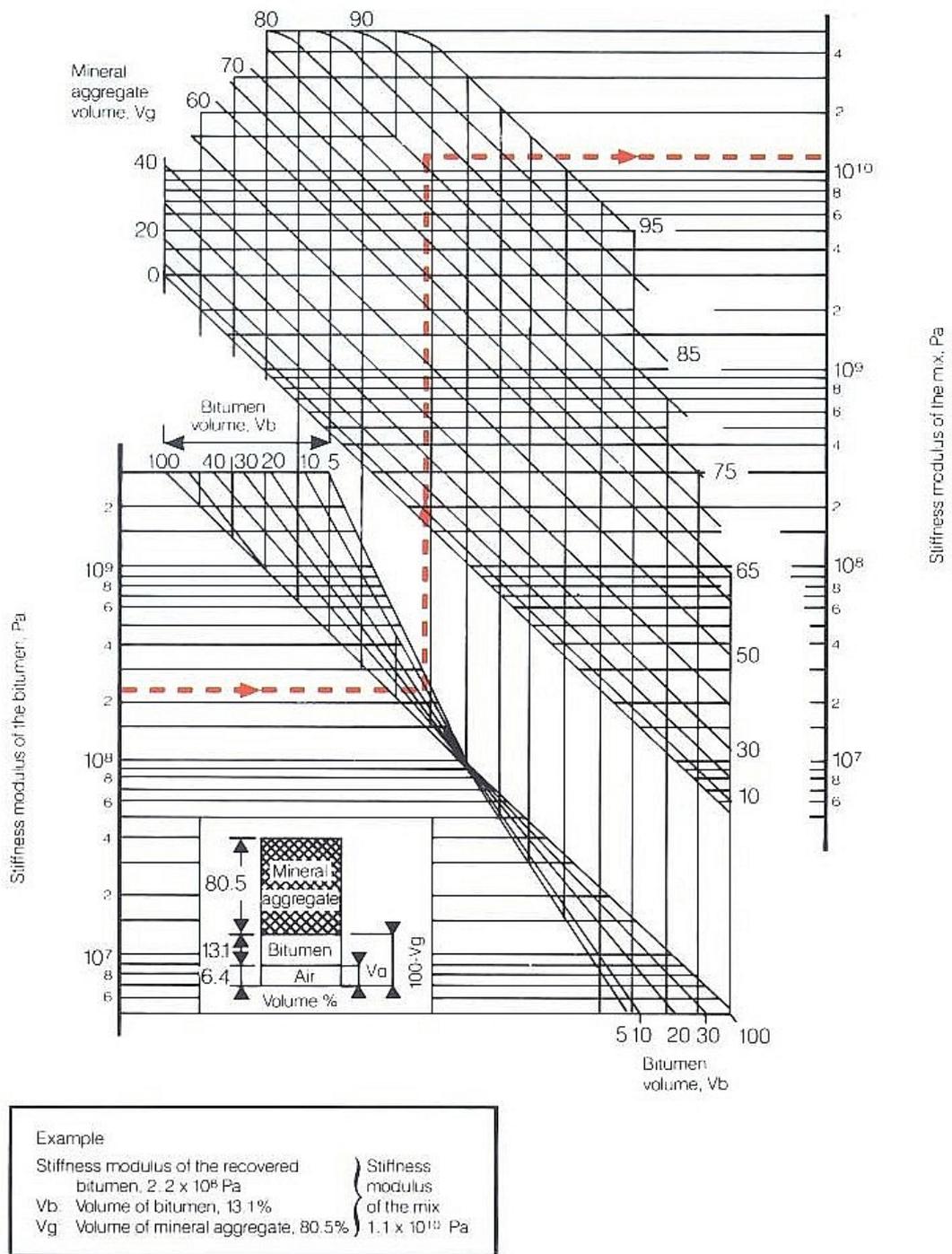


Figure 10.5 – Nomograph for predicting the stiffness modulus of bituminous mixtures [Bonnaure et al., 1977].

frequency of 2 Hz and temperature of 20°C). The frequency of 2 Hz was chosen to be a comparable loading condition to the ITSM testing condition (rise time = 0.125 second), based on Figure 10.6. Consequently, instead of directly using the tensile stiffness ($G^* \times 3$) measured from DSR test, the BANDS predicted binder stiffness (using the Penetration and Softening point test data, Rows 2 and 3 of Table 10.3) was used as the BANDS mixture stiffness prediction input.

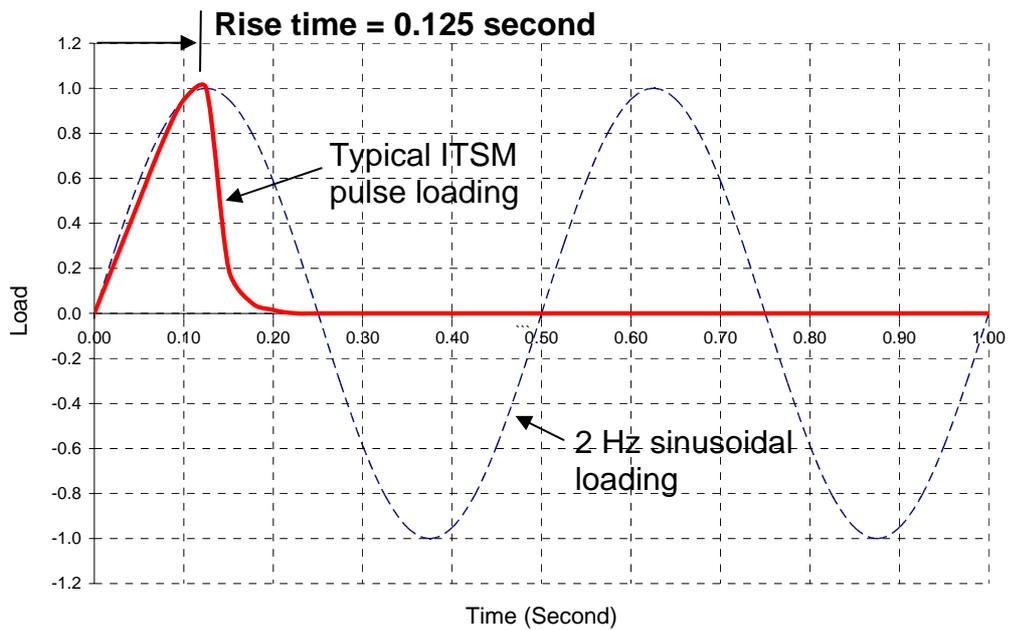


Figure 10.6 – Comparison of 2 Hz sinusoidal load and ITSM pulse load (rise time = 0.125 second).

Figure 10.7 shows the Penetration and Softening point data (unaged, RTFOT aged or HiPAT aged) plotted against the corresponding DSR G^* values of the identical binders (measured at 0.4 Hz and 25°C). The G^* measured at the frequency of 0.4 Hz and the test temperature of 25°C was chosen to be the representative G^* value, since all the related analysis in this thesis (i.e. DSR master curves and the recovered binder assessment) are based on the same condition. For a simpler presentation the G^* values were normalized to the G^*_{HiPAT} (i.e. G^* / G^*_{HiPAT} , Row 8 of Table 10.3) in x – axis. Curve fittings were performed for the both data set (Penetration and Softening point) and, from Figure 10.7, power curves were found to be satisfactory for both cases ($R^2 > 0.99$), as follows;

Table 10.3 – Properties (Penetration, Softening point and DSR G^*) of 15B binders (at different ageing schemes) and BANDS predicted binder stiffness.

| 15B Binder | Unaged | RTFOT | HiPAT |
|---|--------|-------|--|
| Penetration | 15.0 | 11.0 | 6.0 |
| Softening Point (°C) | 68.0 | 75.4 | 96.0 |
| BAND predicted 'tensile' stiffness @ 2 Hz @ 20°C (MPa) | 73.6 | 92.8 | 158.0 |
| DSR measured 'shear' modulus (G^*) @ 2 Hz @ 20°C (MPa) | 14.2 | 17.3 | 40.6 |
| DSR measured 'tensile' stiffness ($G^* \times 3$) @ 2 Hz @ 20°C (MPa) | 42.6 | 51.9 | 121.8 |
| DSR measured complex modulus (G^*) @ 0.4 Hz @ 25°C (MPa) | 2.8 | 4.7 | 16.5 (G^*_{HiPAT}) |
| DSR G^* Ratio @ 0.4 Hz @ 25°C (normalised to G^*_{HiPAT}) | 0.17 | 0.28 | 1.00 |

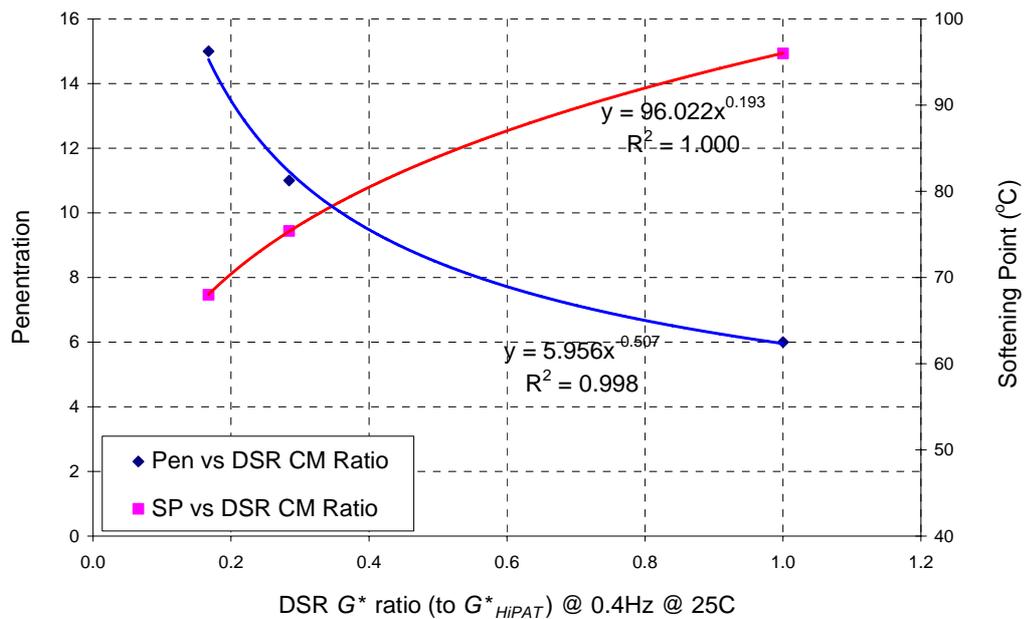


Figure 10.7 – Correlation between the DSR measured G^* ratio (to G^*_{HiPAT}) and BANDS binder stiffness prediction input (Pen and SP data).

- Penetration (y) and G^* ratio (x)

$$y = 5.956 x^{-0.507} \quad (10.6)$$

- Softening point (y) and G^* ratio (x)

$$y = 96.022 x^{0.193} \quad (10.7)$$

Equations 10.6 and 10.7 were used to convert the G^* ratios (to G^*_{HiPAT}) from the DSR test (measured under 0.4 Hz sinusoidal loading frequency and 25°C test temperature) to the BANDS predicted binder stiffness (for 2 Hz loading frequency and temperature of 20°C, equivalent to the ITSM mixture testing condition), as presented in Table 10.4.

Table 10.4 – BANDS predicted mixture stiffnesses.

| $\frac{DSR}{G^*}$ G^*_{HiPAT} | Predicted Pen | Predicted SP (°C) | 15B Binder stiffness (MPa) | 15B Acidic stiffness (MPa) | 15B Basic stiffness (MPa) |
|------------------------------------|---------------|-------------------|----------------------------|----------------------------|---------------------------|
| 0.1 | 19.14 | 61.57 | 62.8 | 6680 | 6720 |
| 0.2 | 13.47 | 70.38 | 80.0 | 7730 | 7770 |
| 0.3 | 10.97 | 76.11 | 91.3 | 8360 | 8400 |
| 0.4 | 9.48 | 80.46 | 105.0 | 9040 | 9080 |
| 0.5 | 8.46 | 84.00 | 114.0 | 9400 | 9440 |
| 0.6 | 7.72 | 87.01 | 125.0 | 9820 | 9860 |
| 0.7 | 7.14 | 89.63 | 138.0 | 10300 | 10300 |
| 0.8 | 6.67 | 91.97 | 146.0 | 10600 | 10600 |
| 0.9 | 6.28 | 94.09 | 153.0 | 10800 | 10900 |
| (G^*_{HiPAT}) 1.0 | 5.96 | 96.02 | 160.0 | 11000 | 11100 |
| 1.1 | 5.68 | 97.80 | 167.0 | 11300 | 11300 |
| 1.2 | 5.43 | 99.46 | 176.0 | 11600 | 11600 |

10.3.2 Mixture stiffness prediction (Bonnaure Nomograph)

The BANDS predicted binder stiffness was then used to predict the mixture stiffness. Table 10.4 shows each predicted data to the corresponding DSR G^* ratio (to G^*_{HiPAT} , Column 1). The Penetration and Softening point data (Columns 2 and 3)

were calculated by inputting G^* ratio (Column 1) to Equations 10.6 and 10.7, respectively. The Penetration and Softening point data (Columns 2 and 3) were then inputted to BANDS to calculate the corresponding binder stiffnesses (Column 4). Columns 5 and 6 show the predicted stiffnesses of 15B Acidic and 15B Basic mixtures using the binder stiffness data (Column 4) and the following input parameters (calculated from the volumetric information of both mixtures, see Chapter 5);

- 15B Acidic mixture: $V_v = 8.0\%$, $V_b = 8.91\%$, $V_a = 83.09\%$;
- 15B Basic mixture: $V_v = 8.0\%$, $V_b = 8.86\%$, $V_a = 83.14\%$.

As shown in Figure 10.8 a curve fitting was performed on the predicted mixture stiffnesses against the DSR G^* ratio (to G^*_{HiPAT}) to get a correlation equation. Since the predicted stiffnesses of 15B Acidic and 15B Basic mixtures showed virtually identical values, the curve fitting was performed on the 15B Acidic data only. From Figure 10.8, a power curve was found to be satisfactory ($R^2 > 0.99$), as follows;

- BANDS predicted mixture stiffness (y) and G^* ratio (x)

$$y = 11061.595 x^{0.223} \quad (10.8)$$

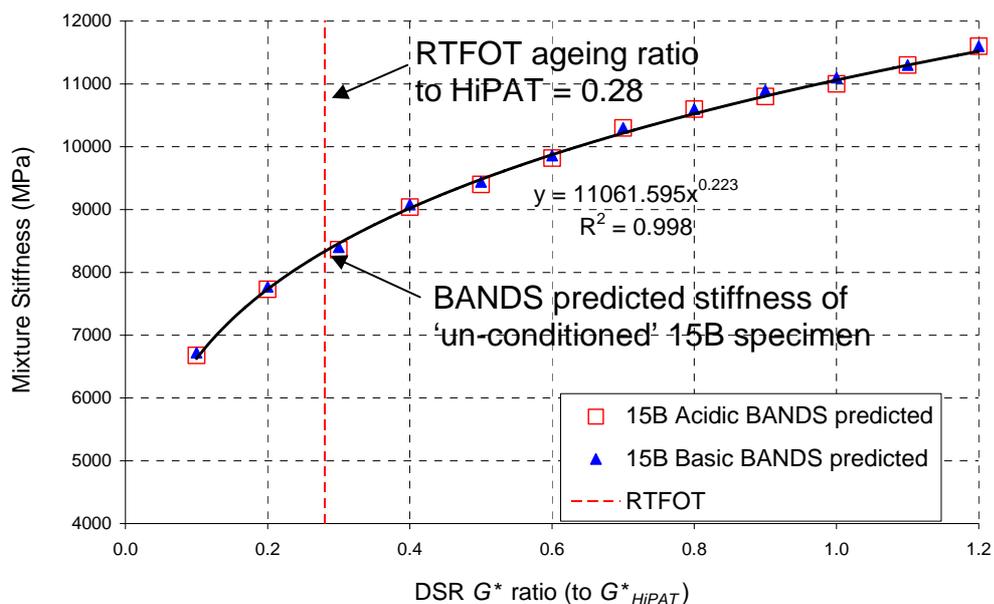


Figure 10.8 – BANDS predicted stiffness of 15B mixtures against DSR G^* ratio.

The predicted mixture stiffnesses of Figure 10.8 are re-plotted in Figure 10.9 where the mixture stiffnesses (y – axis) are normalised to the BANDS predicted stiffness of ‘un-conditioned’ specimen (i.e. 8328 MPa @ G^* ratio of 0.28, from Equation 10.8). The normalised mixture stiffness is the ageing factor (f_{ageing} , see Equation 10.4) which predicts the stiffness increase of the ‘un-conditioned’ specimen (i.e. $S_{initial} \times f_{ageing}$) due to the binder hardening. This is based on an assumption that the binder in the ‘un-conditioned’ specimen has the same properties to those of RTFOT aged binder (due to the short-term ageing during the mixing and compaction process).

To get a general correlation between the binder hardening (G^* ratio) and the resultant mixture ageing factor (f_{ageing}), a curve fitting was performed again and, from the Figure 10.9, a power curve was found to be satisfactory ($R^2 = 1.00$), as follows;

- Ageing factor (f_{ageing}) (y) and G^* ratio (x)

$$y = 1.328 x^{0.223} \quad (10.9)$$

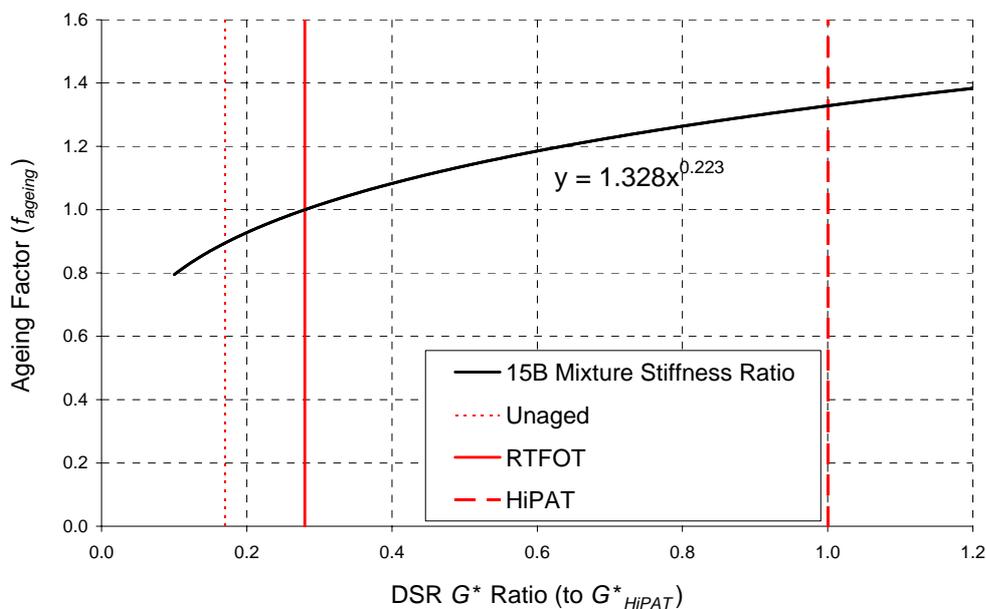


Figure 10.9 – Ageing factor (f_{ageing}) of 15B mixtures against DSR G^* ratio.

For an example, from Equation 10.9, the stiffness of the un-conditioned 15B specimen ($S_{initial}$) is expected to rise 33% (i.e. $S_{initial} \times f_{ageing}$ of 1.33), if the binder in the mixtures hardens to a HiPAT equivalent level (i.e. G^* ratio = 1.0).

Figure 10.10 shows predicted mixture stiffness increase during the SATS test. A series of binder was recovered from the shorter period SATS protocol (4 hours and 24 hours SATS, see Section 9.4.5 for more details) and the measured G^* was normalised to the G^*_{HiPAT} to get G^* ratio. The G^* ratio of 0.58 and 0.74 were obtained from 4 hours SATS and 24 hours SATS respectively and these values were inputted in Equation 10.9 to get the corresponding ageing factors (f_{ageing}). The results are presented in Figure 10.10 demonstrating the ageing factors (f_{ageing}) change (i.e. increase) as SATS test time elapses.

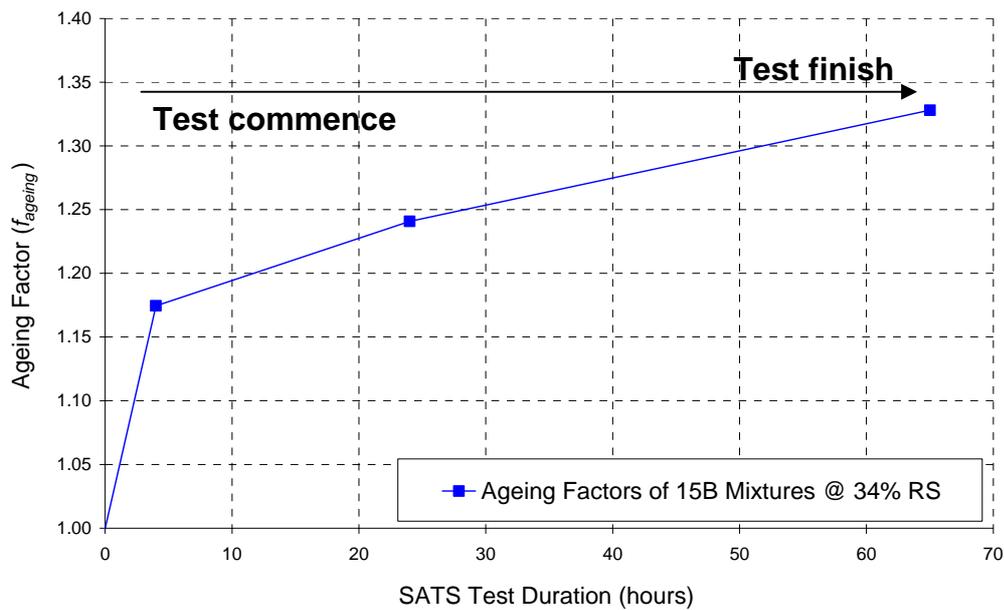


Figure 10.10 – Expected ageing factor (f_{ageing}) change during the SATS process.

10.4 Pressure Damage ($f_{pressure}$)

From Equation 10.4, the stiffness modulus ($S_{DryHiPAT}$) of a specimen from the Dry HiPAT ageing test (see Section 8.3.5) can be expressed as follows, since the moisture factor ($f_{moisture}$) is 1.0 (i.e. no moisture damage).

$$S_{DryHiPAT} = S_{initial} \times f_{ageing} \times f_{pressure} \quad (10.10)$$

The G^* ratio (to G^*_{HiPAT}) of a specimen from the Dry HiPAT test is calculated to be 1.19 from Equation 9.1 (when retained saturation is 0.0%, see Section 9.4.2) and therefore the ageing factor (f_{ageing}) of the Dry HiPAT tested specimen, from Equation 10.9, is calculated to be 1.38.

Thus, from Equation 10.10, the pressure factor ($f_{pressure}$) of specimen (for a pressure of 2.1 MPa) can be obtained as follows.

$$f_{pressure} = \frac{S_{DryHiPAT}}{1.38 \times S_{initial}} = 0.72 \times \frac{S_{DryHiPAT}}{S_{initial}} \quad (10.11)$$

Columns 2 and 3 of Table 10.5 show the Dry HiPAT ageing test (depressurised at 85°C) results of 15B Acidic and 15B Basic mixtures and the calculated pressure factor ($f_{pressure}$) from Equation 10.11, whereas Columns 4 and 5 shows those from the Dry HiPAT test depressurised at 30°C.

Table 10.5 – Dry HiPAT results (85°C and 30°C depressurisation) and $f_{pressure}$.

| Specimen | Dry HiPAT (85°C depress) | | Dry HiPAT (30°C depress) | |
|--------------------|------------------------------------|----------------|------------------------------------|----------------|
| | $\frac{S_{DryHiPAT}}{S_{initial}}$ | $f_{pressure}$ | $\frac{S_{DryHiPAT}}{S_{initial}}$ | $f_{pressure}$ |
| 15B Acidic control | 1.05 | 0.76 | 1.16 | 0.84 |
| 15B Basic control | 0.85 | 0.61 | 1.30 | 0.94 |

Table 10.5 clearly demonstrates that Basic mixture has more pressure damage (i.e. lower $f_{pressure}$) than that of Acidic mixture, from 85°C depressurisation Dry HiPAT test. However, the pressure damage of Basic mixture was much reduced (i.e. higher $f_{pressure}$) by depressurising at 30°C, suggesting that the modified SATS protocol (depressurising at 30°C, see Section 8.3) can significantly reduce the pressure damage, as proposed.

10.5 Moisture Sensitivity ($f_{moisture}$) of SATS Data

From the Shell BANDS 2.0 software and the Dry HiPAT ageing test, the ageing factor (f_{ageing}) and the pressure factor ($f_{pressure}$) have been identified respectively. For examples, if a specimen shows an initial stiffness of 10,000 MPa ($S_{initial}$) and a conditioned stiffness of 5,000 MPa after the SATS test ($S_{conditioned}$), the nominal retained stiffness of the specimen ($RS_{nominal}$) is calculated to be 0.50 (5,000 MPa / 10,000 MPa), from Equation 10.3. However, from Equations 10.5 and 10.9, the ‘true’ moisture sensitivity ($f_{moisture}$) of the specimen is calculated as follows, if the binder in the specimen was aged to HiPAT equivalent level (i.e. G^* ratio = 1.0) and tested using the standard SATS, for 15B Acidic specimens ($f_{pressure} = 0.76$);

$$f_{moisture} = \frac{5,000}{10,000 \times 1.33 \times 0.76} = 0.49$$

For 15B Basic specimens ($f_{pressure} = 0.61$),

$$f_{moisture} = \frac{5,000}{10,000 \times 1.33 \times 0.61} = 0.62$$

If the specimen was tested using the modified SATS, for 15B Acidic specimens ($f_{pressure} = 0.84$),

$$f_{moisture} = \frac{5,000}{10,000 \times 1.33 \times 0.84} = 0.45$$

For 15B Basic specimens ($f_{pressure} = 0.94$),

$$f_{moisture} = \frac{5,000}{10,000 \times 1.33 \times 0.94} = 0.40$$

10.5.1 Moisture sensitivity ($f_{moisture}$) and retained saturation

From the recovered binder assessment performed in Chapter 9, it is clear that the ageing levels of the SATS tested specimens vary depending on its retained saturation (see Section 9.4.2). The correlation, Equation 9.1, is from the standard SATS protocol and it is easily assumed that the different testing setup will have a different correlation. For an example, Atmospheric SATS test performed in Section 7.3.2 will have a different correlation equation between ageing ratio and retained saturation. From Figure 10.11, a line fitting was performed on recovered binder data points (three data points available from the recovered binder assessment) from the Atmospheric SATS, as follows;

$$y = -0.0026 x + 0.6200 \quad (10.12)$$

where, $y = G^*$ ratio (to G^*_{HIPAT}) and $x =$ retained saturation (%).

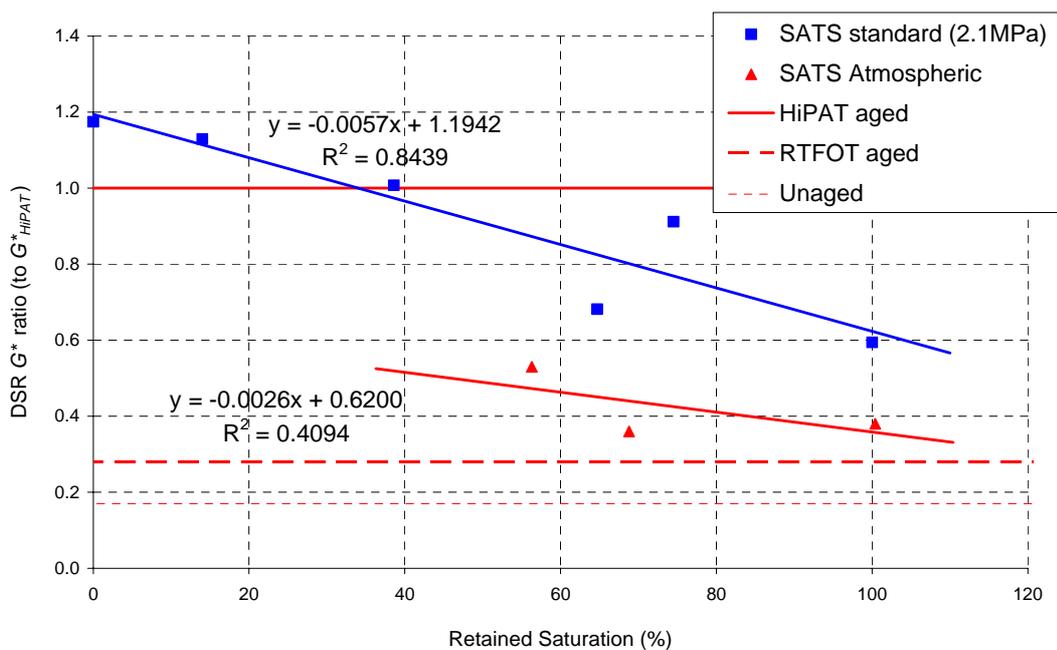


Figure 10.11 – Correlation lines of G^* ratio and retained saturation for the standard SATS and Atmospheric SATS protocols.

Equations 9.1 (for standard SATS) and 10.12 (for Atmospheric SATS) have been used to produce a master chart, Figure 10.12, showing ageing factor and pressure factor (i.e. $f_{ageing} \times f_{pressure}$) according to the material type (15B Acidic and Basic), testing conditions (standard SATS and Atmospheric SATS) and corresponding retained saturations. It should be noted that the pressure factor ($f_{pressure}$) of Atmospheric SATS is 1.0 (i.e. no pressure damage), thus $f_{ageing} \times f_{pressure} = f_{ageing}$.

Separate curve fittings were performed and a 2nd order polynomial was found to be satisfactory for each case ($R^2 > 0.99$). The correlations for 15B Acidic and 15B Basic from the standard SATS are expressed in Equations in 10.13 and 10.14, respectively. The correlation for Atmospheric SATS is expressed in Equation 10.15, as follows;

$$y = -0.000004 x^2 - 0.001007 x + 1.048922 \quad (10.13)$$

$$y = -0.000003 x^2 - 0.000808 x + 0.841898 \quad (10.14)$$

$$y = -0.000003 x^2 - 0.001041 x + 1.192980 \quad (10.15)$$

where, $y = f_{ageing} \times f_{pressure}$, and $x =$ retained saturation (%).

Figure 10.13 presents another master chart showing ageing and pressure factor (i.e. $f_{ageing} \times f_{pressure}$) of 15B Acidic and 15B Basic specimens from the modified SATS protocol. The same ageing factor (f_{ageing}) of standard SATS (i.e. using Equations 9.1 and 10.9) was directly applied to the modified SATS and the pressure factors ($f_{pressure}$) were from Table 10.5 (from Dry HiPAT 30°C depressurisation test).

The correlations for 15B Acidic and 15B Basic from the modified SATS are expressed in Equations in 10.16 and 10.17, respectively.

$$y = -0.000004 x^2 - 0.001113 x + 1.159335 \quad (10.16)$$

$$y = -0.000005 x^2 - 0.001245 x + 1.297351 \quad (10.17)$$

where, $y = f_{ageing} \times f_{pressure}$, and $x =$ retained saturation (%).

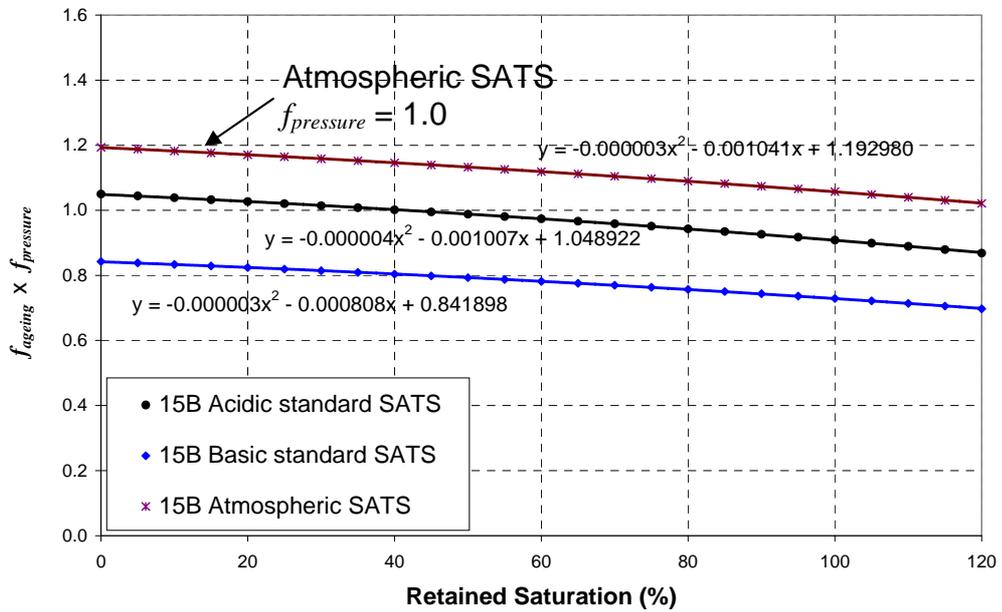


Figure 10.12 – Master chart showing $f_{ageing} \times f_{pressure}$ of standard SATS and Atmospheric SATS.

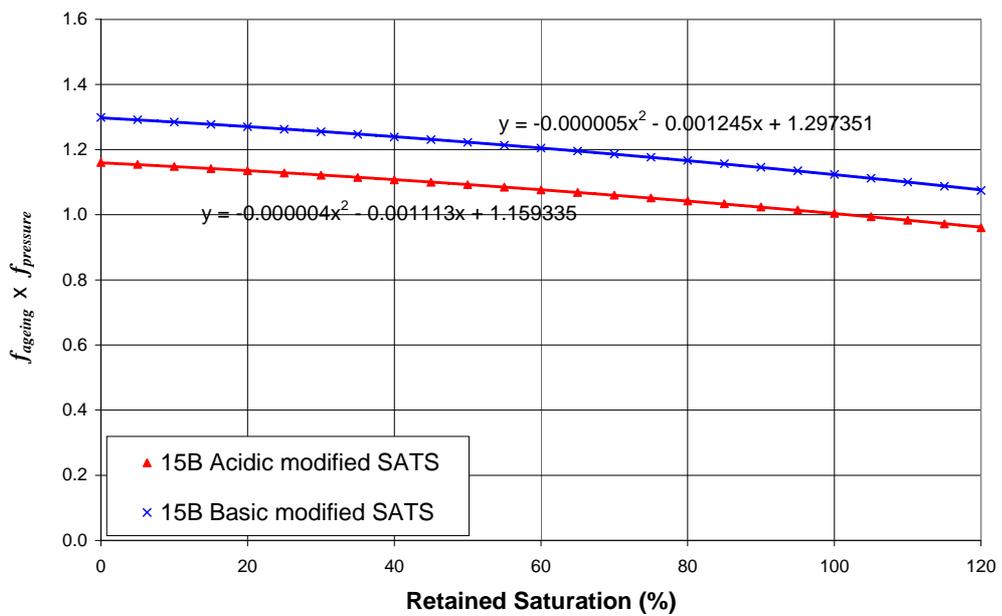


Figure 10.13 – Master chart showing $f_{ageing} \times f_{pressure}$ of modified SATS

For examples, if a specimen shows an initial stiffness of 10,000 MPa and a conditioned stiffness of 5,000 MPa (i.e. $RS_{nominal} = 0.5$) with a retained saturation of 50%, the ageing and pressure factors ($f_{ageing} \times f_{pressure}$) of the specimen are calculated, as follows;

- 15B Acidic standard SATS → 0.99 (from Equation 10.13),
- 15B Basic standard SATS → 0.79 (from Equation 10.14),
- 15B Atmospheric SATS → 1.13 (from Equation 10.15),
- 15B Acidic modified SATS → 1.09 (from Equation 10.16), and
- 15B Basic modified SATS → 1.22 (from Equation 10.17).

Thus, from Equation 10.5, the ‘true’ moisture sensitivity ($f_{moisture}$) of the specimen is calculated, as follows;

- 15B Acidic standard SATS → 0.51,
- 15B Basic standard SATS → 0.64,
- 15B Atmospheric SATS → 0.44,
- 15B Acidic modified SATS → 0.46, and
- 15B Basic modified SATS → 0.41.

This generally agrees that, Atmospheric SATS and the modified SATS have relatively severer moisture damage condition than that of standard SATS. This is assumed to be due to a more accelerated water circulation of Atmospheric SATS

(see Section 7.3 for more discussions), and due to the increased and prolonged water contact of the modified SATS (see Section 8.3.1 for more discussions).

Figure 10.14 shows typical examples of retained stiffness ($RS_{nominal}$) and moisture sensitivity ($f_{moisture}$) of the 15B Acidic material tested using the standard, Atmospheric or modified SATS.

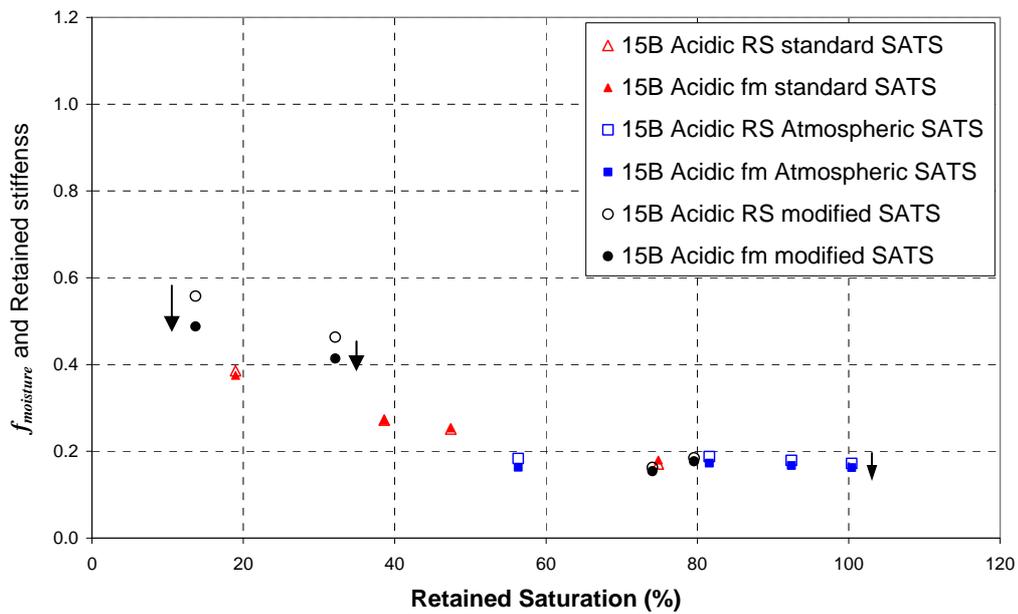


Figure 10.14 – Examples of retained stiffness ($RS_{nominal}$) and moisture sensitivity ($f_{moisture}$) of 15B Acidic material.

As observed in Figure 10.14, $f_{moisture}$ of 15B Acidic material commonly show relatively lower values than its $RS_{nominal}$, although the difference is very marginal (virtually identical values for the standard SATS).

Figure 10.15 shows typical examples of retained stiffness ($RS_{nominal}$) and moisture sensitivity ($f_{moisture}$) of the 15B Basic material tested using the standard, Atmospheric or modified SATS.

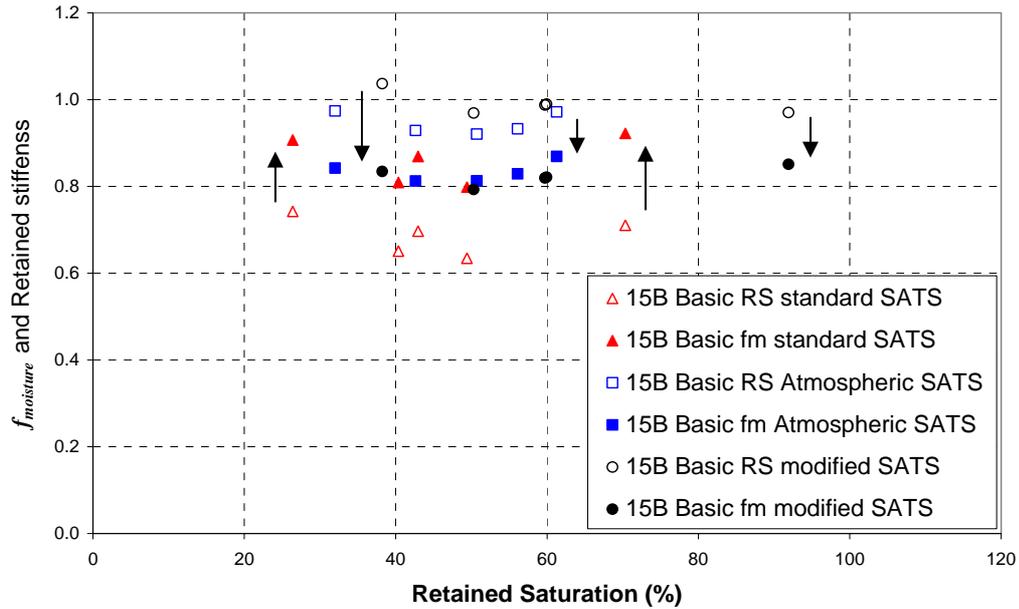


Figure 10.15 – Examples of retained stiffness ($RS_{nominal}$) and moisture sensitivity ($f_{moisture}$) of 15B Basic material.

As observed in Figure 10.15, $f_{moisture}$ of 15B Basic from the standard SATS shows relatively higher values than its $RS_{nominal}$. However, $f_{moisture}$ of 15B Basic from Atmospheric SATS and the modified SATS commonly show relatively lower values than its $RS_{nominal}$. Consequently, regardless of the different test condition, 15B Basic material commonly showed very similar moisture sensitivity values ($f_{moisture}$) of between 0.8 ~ 0.9. Therefore, it can be concluded that, from the comparison of $f_{moisture}$ of the standard SATS and Atmospheric SATS, the considerably more aged binder of the standard SATS did not significantly alter the moisture sensitivity of the mixture.

The moisture sensitivity ($f_{moisture}$) of 15B Acidic and 15B Basic SATS main test series data shown in Figure 5.7 (see Section 5.5) are compared with its retained stiffness (i.e. $RS_{nominal}$) in Figure 10.16. It can be concluded that, from Figure 10.16, the moisture sensitivity ($f_{moisture}$) of the 15B Acidic data from the standard SATS protocol are considered to be virtually identical to its retained stiffnesses. However, the moisture sensitivity of 15B Basic material is considered to be higher (up to 0.25), depending on the corresponding retained saturation, than its retained stiffness.

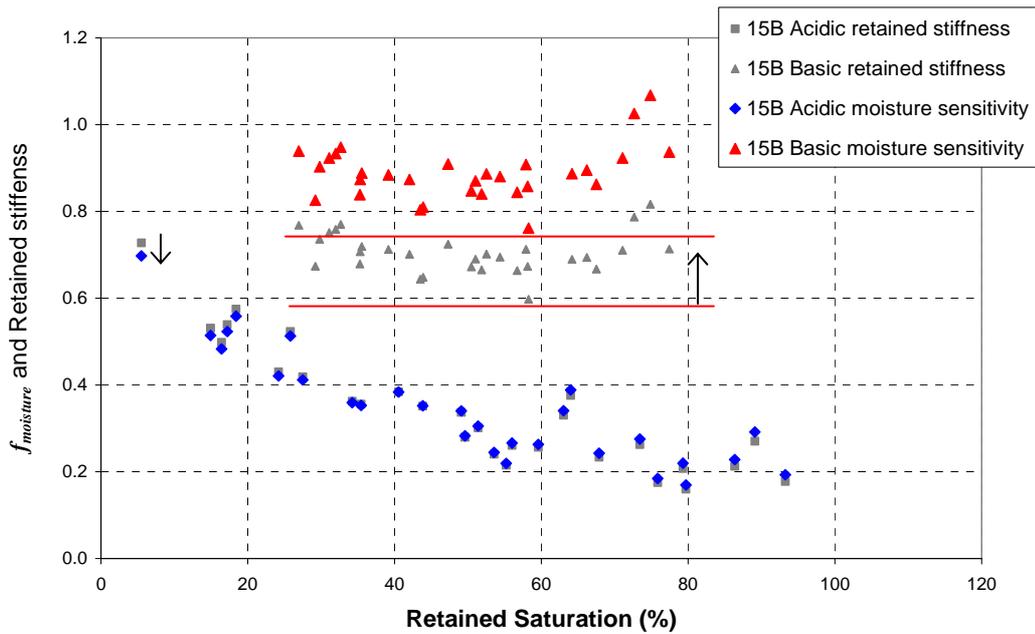


Figure 10.16 – Comparison of $f_{moisture}$ and retained stiffness of the SATS main test series.

10.6 Summary and Conclusions

The internal volumetric change during the SATS process has been investigated. It is considered that the volumetric change (increment of air voids) is associated with retained stiffness. For an example, if a specimen is severely damaged showing very low retained stiffness, the number of voids and/or the size of existing voids are believed to be increased due to creation of cracks or adhesion failure between aggregates and bituminous binder.

Representative correlations were obtained from a limited number of specimens and applied to the main data series to observe the ‘actual’ retained saturation (Sat_{actual}), considering the volumetric change taking place during the SATS process. For the selected types of materials (15B Acidic and Basic control), the difference between ‘ $Sat_{nominal}$ ’ and ‘ Sat_{actual} ’ appears to vary in a range of up to 20%, associated with its corresponding retained stiffnesses.

It is believed that the correlation of retained saturation to retained stiffness is a material dependent factor. For an example, the EME 2 material (used in Section

6.4.1) is likely to have a different correlation equation, as it has different volumetric properties. However, it still can be believed that the less damaged specimens (higher retained stiffness) will have less volumetric change. For an example, the EME 2 specimens tested in Section 6.4.1, which showed relatively low retained stiffness with clearly visible expansion of specimen size, will have higher volumetric change, while the identical specimens tested in Section 8.3.3 (using the modified SATS) will have much less volumetric change.

The ‘true’ moisture sensitivity (i.e. $f_{moisture}$) of the material from the SATS protocol has been investigated (by extracting ageing factor and pressure factor). The correlation between binder ageing (G^* ratio) and the expected mixture stiffness increase (i.e. f_{ageing}) has been obtained using the Shell BANDS 2.0 software. The correlation has been used to predict stiffness increase curve for the 15B materials (only by f_{ageing}) and, from this curve, the pressure damage (i.e. $f_{pressure}$) caused by the applied high pressure of the SATS protocol has been identified. Consequently, the moisture sensitivity of the material used in this thesis has been identified.

From the investigation of the ‘true’ moisture sensitivity which showed very similar values regardless of different binder ageing level (especially from 15B Basic mixture), it can be concluded that, for the limited material range covered in this study, the ageing of the binder did not significantly alter the moisture sensitivity of the mixture.

Chapter 11. Summary, Conclusions and Recommendations for Future Work

11.1 General Summary

Due to concerns over ageing and moisture sensitivity of HMB materials, from on-going monitoring of the initial trial site, a research programme was established to investigate the long-term durability of the material. The main objective of the research programme was to develop a laboratory testing procedure able to reproduce the material properties observed in the HMB trial site, with a specific interest in the ageing and moisture sensitivity combined long-term durability of the HMB materials.

Since none of the existing durability protocols are found to be able (or designed) to do the purpose, the prime concern was given to develop a new protocol which would combine the effect of the both aspects (ageing and water damage) together in one process. More specifically, the protocol needs to be able to simulate 60% or more reduction in mixture stiffness due to moisture damage, combined with 10 or more years of 'long-term' binder ageing.

11.1.1 Literature review (Chapter 2)

The main aim of the literature review was to provide the reader with the relevant background knowledge applicable to this study (particularly durability test methods). Therefore, the chapter was divided into three main sections reviewing 'Introduction to Pavement Engineering', 'Ageing Tests for Bituminous Materials' and 'Moisture Sensitivity Tests for Bituminous Materials'.

The binder ageing can be divided into short and long term ageing. The 'short-term' describes that the ageing process (mainly volatile loss) occurred during the asphalt mixture construction process (heating up, mixing, delivery and compaction) and the 'long-term' describes that the progressive oxidation process of the in-place material in the field during its service life.

Moisture sensitivity tests generally have a conditioning and an evaluation phase. The conditioning processes associated with most test methods are to simulate the deterioration of the asphalt mixture in the field exposure conditions, in an accelerated manner to be tested in a reasonably shorter periods of time. Normally the acceleration is achieved by immersing the material in an elevated temperature.

Most moisture sensitivity test procedures on the compacted mixture measure the loss of strength and stiffness of an asphalt mixture due to moisture conditioning. The most common way of quantifying the damage caused by the conditioning process is to directly compare the ‘conditioned’ mechanical properties to those of ‘unconditioned’. The mechanical properties can be evaluated using various mechanical tests which can be divided into destructive (compression, stability, indirect tensile strength and fatigue) and non-destructive (indirect tensile stiffness and resilient modulus) approaches.

11.1.2 Development of SATS Protocol (Chapters 3 and 4)

Chapter 3 presented a series of preliminary tests. Among the various trials investigated in this chapter, to find a suitable durability test method which fulfils the given objectives of this study, the HiPAT ageing using vacuum saturated specimens showed the most interesting behaviour. Firstly it was the only process which was severe enough to produce the target stiffness reduction within reasonably shorter period of time. Secondly it was the only process which had ageing and moisture sensitivity schemes in ‘one process’.

The investigation on the recovered binder extracted from the specimens which had been through the above protocol (referred to as preliminary SATS) clearly demonstrated a comparable level of binder ageing to the HiPAT aged binder. Knowing that the binder in the mixture will be ‘long-term’ aged according to the given HiPAT ageing conditions, while the compacted specimens are ‘moisture damaged’ by saturated water in the pressure vessel, the SATS protocol showed a strong possibility to be a suitable durability test method which fulfils the given objectives of the study.

Chapter 4 described the details of SATS protocol development procedure. The preliminary version of SATS protocol (preliminary SATS) was performed on laboratory manufactured specimens. The variability investigation using nominally identical sets of specimens revealed that the test conditions vary dependent on the specimen position within the vessel and the key factor is believed to be the retained saturation.

Among the various modifications to optimise the SATS protocol, considering the practicability and repeatability of the protocol, the semi immersion SATS protocol appeared to be the most suitable approach, as it produces from a very dry specimen to a completely immersed specimen with gradually saturated specimens between them.

11.1.3 SATS Durability Test (Chapters 5 and 6)

Chapter 5 presented SATS durability tests results performed on a series of mixtures including a mixture nominally identical to that used in the HMB trial site. Generally the Basic mixture series showed a better performance than the Acidic mixture series. For Acidic aggregate mixture series the retained stiffness (at a particular retained saturation) was compared favourably with the stiffness modulus reduction observed from the initial trial site.

An investigation of test variability showed a reasonably good repeatability with the mixtures selected. No statistically clear difference was found from nominally identical mixtures that contained binders from different sources. The retained saturation variation at each specimen position was also investigated and generally showed a practically acceptable range of variation.

Chapter 6 presented the SATS test results performed on a wider range of mixtures. The results from Acidic mixtures with HLF showed superior performances. However, the already good performance of the Basic mixtures was not significantly improved by the adding of HLF. The effect of anti-stripping agent was also investigated and showed some improved performance.

Investigation on mixture variability showed an unexpected results showing well compacted or richer mixture performing poorer than the control mixture, especially for the Basic mixtures. 15E Basic EME material also showed worse performance than expected.

11.1.4 SATS Parameters and Modification (Chapters 7 and 8)

Chapter 7 presented a more carefully investigation on the parameters used in the SATS protocol. It appears that the applied pressure, to induce the accelerate binder ageing, is also inducing a certain degree of specimen damage. Consequently two different modes of pressure related damage were proposed and their expected properties are suggested.

Chapter 8 described the modified SATS protocol which was developed to reduce the pressure damage observed in the previous chapters. The modified SATS protocol was applied to a limited range of mixtures and clearly displayed much reduced level of pressure damage (especially from the Basic mixture series).

11.1.5 Recovered Binder Assessment (Chapter 9)

Chapter 9 presented an extensive investigation on recovered binders from various SATS conditions. Comparison of saturated SATS and dry SATS test showed similar level of ageing regardless of the existence of water. However, the fully immersed specimen from the standard SATS and Atmospheric SATS commonly showed much less level of ageing compared to the specimens above the water level from the standard SATS.

A more careful investigation on the ageing variation along the specimen position revealed that there is a clear trend of ageing level dependent on the specimen position. From a limited set of results a representative line was fitted and a retained saturation of 34% was found to be the HiPAT equivalent ageing condition for the 15B Acidic standard mixture from the standard SATS protocol.

11.1.6 Data Interpretation (Chapter 10)

Chapter 10 presented a further investigation to interpret the nominal SATS data obtained in the previous chapters more accurately. The internal volumetric change during the SATS process was investigated. It was considered that the volumetric change is associated with retained stiffnesses. Representative correlations were obtained from a limited number of specimens and applied to the main data series to observe the ‘actual’ retained saturation (i.e. Sat_{actual}), considering the volumetric change during the SATS test process.

The ‘true’ moisture sensitivity ($f_{moisture}$), by extracting the ageing factor (f_{ageing}) and pressure factor ($f_{pressure}$), was investigated. The correlation between binder ageing (expressed in G^* ratio to G^*_{HiPAT}) and the ageing factor (i.e. stiffness increase due to binder ageing in the mixture) was obtained using the Shell BANDS 2.0 software. The ageing factor was used to predict ‘un-damaged’ stiffness increment curve for the 15B materials, and from this curve, the pressure factor (i.e. stiffness reduction caused by the applied high pressure of the SATS protocol) was identified. Consequently the ‘true’ moisture sensitivity (i.e. stiffness reduction caused by the moisture damage only) of the material was obtained and compared to the ‘nominal’ retained stiffness.

11.2 Conclusions

The following conclusions have been drawn from the various chapters of this thesis.

- From the worldwide questionnaire survey (Appendix B), the most commonly used tests for short-term and long-term ageing simulation are the RTFOT and PAV protocols respectively.
- A temperature of 85°C appears to be the maximum applicable temperature to age the compacted specimen.
- The existing durability protocols are designed to investigate one aspect only (i.e. ageing test for ageing sensitivity, moisture sensitivity test for moisture damage).

- In long-term service condition, pavements undergo both aspects of ageing and moisture damage together (i.e. materials damaged by water infiltration while the binder in the material is being aged).
- Binder of the HiPAT aged specimen is expected to be aged comparable to the HiPAT aged binder, regardless of existence of moisture in the pressure vessel.
- The retained saturation is the key factor to control the magnitude of damage created by the SATS protocol.
- In the SATS test setup, the retained saturation variation depending on the specimen position is inevitable and therefore needs to be accepted as a test parameter.
- Retained saturation variation depending on the specimen position in the pressure vessel is beneficial since the sensitivity of a material to the various amount of moisture could be obtained.
- For the mixture range investigated in Chapter 5, the SATS protocol successfully discriminated the durability of mixtures against the combined effect of ageing and moisture damage, on the basis of a measured reduction in stiffness similar to that observed in the initial HMB trial site.
- Variability investigation on the SATS protocol showed a practically acceptable level of variation of retained stiffnesses and retained saturations.
- The high pressure applied to accelerate the ageing process of binder in the compacted specimens also induces a certain degree of damage (stiffness reduction) to the specimen.
- The pressure damage mainly occurs during the depressurising process.
- Magnitude of the pressure damage is closely related to the proportion of Mode I (deformation) and Mode II (puncture) damage.

- A mixture with a higher binder content and/or lower air voids is more susceptible to the pressure damage from the SATS protocol, as the mixture is likely to have a more closed ‘non-interconnected’ voids.
- A mixture with a better adhesion property between aggregates and binder is likely more susceptible to the pressure damage as the mixture will create more ‘non-interconnected’ voids along the interface of aggregates and binder.
- A mixture with a softer binder is thought to be more susceptible to the pressure damage as the binder will have more viscose properties at a same pressurising (or depressurising) temperature.
- Considering the distinctively ‘lean’ volumetric properties of the materials focused on this study, the SATS data produced in Chapter 5 using various combinations of binders and aggregates are considered to be credible, especially for the Acidic materials which are nominally identical to the HMB trial site material.
- For the Basic materials, the presented data in Chapter 5 incorporates a considerable amount of pressure damage and the damage can be reduced by the modified SATS protocol.
- The modified SATS protocol, presented in Chapter 8, is a better protocol, especially to be applied to a wider range of mixtures including the mixtures with more pressure sensitive volumetric properties.
- For the 15B Granite mixtures the ageing level of binder in the standard SATS tested specimens varies depending on its retained saturation and the HiPAT binder equivalent ageing can be found in a retained saturation of 34%.
- To achieve a HiPAT equivalent binder ageing from the SATS protocol within the same testing duration (65 hours), the pressure and temperature conditions (2.1 MPa and 85°C) used in the standard SATS are necessary.

- Initial volumetric properties of the specimen can change during the SATS process, and can be correlated to its retained stiffness.
- The retained stiffness used to present SATS test results is a combined function of ageing factor, pressure factor and moisture factor.
- By extracting the ageing factor and pressure factor, the moisture sensitivity of the material from the SATS test can be identified.
- From the limited material range covered in this study, the ageing of the binder did not significantly alter the moisture sensitivity of the mixture.
- Considering the variability of the SATS test and the pressure damage incorporated, materials tested using the SATS protocol need to be ranked in a broad range.
- From the limited range of materials observed in this study, the following guideline of durability performance can be suggested (in a retained saturation range higher than 40%).
 - A (Retained stiffness > 0.8) – excellent performance.
 - B (0.8 > Retained stiffness > 0.4) – acceptable performance.
 - C (Retained stiffness < 0.4) – poor performance.

11.3 Recommendations for Future Work

For this thesis, the standard SATS (presented in Chapter 5) was the main testing protocol under investigation. However, from the findings in the later stage, the modified SATS (30°C depressurising) clearly demonstrated to be a better and more recommendable protocol (in Chapter 8). Thus the modified SATS protocol needs to be accepted as ‘the’ SATS protocol (instead of the standard SATS). Consequently, to understand the test variability and recovered binder properties of the modified SATS protocol, the variability investigation performed in Chapter 5 and the

recovered binder assessment performed in Chapter 9 are recommended to be performed on the modified SATS protocol as the same manners.

The performance ranking guideline proposed in the previous section is only an example, based on a limited range of HMB materials covered in this study. A more credible and acceptable guideline can only be produced by testing of a much wider range of HMB mixtures, desirably linked with the field performance.

Since the prime objective of the research presented in this thesis was to reproduce the material properties observed in the initial HMB trial site, it was inevitable to produce a testing protocol specifically focused on a particular material type (i.e. HMB with lean volumetric properties). Although this initial goal has been successfully achieved, it is still desirable for the protocol to be applicable to a wider range of mixture types. Thus mixtures with relatively softer binders (e.g. 25 Pen and 35 Pen grade binders) need to be investigated to find the limit of the applicability of the SATS protocol. Also more various types of aggregates and gradings need to be reviewed.

The correlation of retained saturation and retained stiffness obtained in Chapter 10 was based on a limited number of specimens only. Thus to make the correlations more accurate and credible, a more number of specimens needs to be investigated. Also it should be noted that only the control specimens (i.e. 15B Acidic/Basic with High AV/Low BC) were observed in this investigation. Since mixtures with different properties (e.g. Low AV, High BC or EME material) are expected to have different correlation curves, it is desirable to observe the volumetric change of such materials, as the same manner used for the control mixtures.

The correlation of binder hardening and resultant mixture stiffness increase was estimated based on the nomographs produced using different range of materials and testing conditions to those used in this study. Thus it is desirable to produce the correlation using the identical material and testing conditions used in this study. This can be achieved by a series of investigation on recovered binders from conventional dry oven aged specimens (identical mixtures used in this study) under various ageing period. This investigation can also be used to find an identical oven

ageing time equivalent to the HiPAT binder ageing protocol without the pressure factor. Ultimately this investigation can lead to develop an alternative SATS protocol without concerns on pressure damage. Although a much longer testing time is expected, the alternative protocol can effectively be used as a comparison protocol to the SATS protocol developed in this study.

Appendix A - Core Logs from HMB Field Site

(Selected examples of cores in poor,
intermediate and good conditions)

Appendix B – Questionnaire Survey of Durability
Tests on Bituminous Material

Appendix C – Binder Characterisation Tests

- Penetration Test
- Softening Point Test
- Rotational Viscosity Test
- Dynamic Shear Rheometer Test

Appendix D – Draft of SATS Protocol

Note:

1. This protocol is referred to as 'standard SATS' in the main chapters.
2. The term 'retained stiffness' used in the main chapters is referred to as 'Stiffness Ratio'
3. The term 'retained saturation' used in the main chapters is referred to as 'Saturation after Conditioning'.

Appendix E – Individual Test Data Charts

Appendix F – Manual of Contracted Document for Highway Works: Clause 953

Method for the Assessment of Durability of Compacted Asphalt
Mixtures using the Saturation Ageing Tensile Stiffness (SATS) Test

Note:

1. This protocol is referred to as 'modified SATS' in the main chapters.
2. The term 'retained stiffness' used in the main chapters is referred to as 'Stiffness Ratio'
3. The term 'retained saturation' used in the main chapters is referred to as 'Saturation after Conditioning'.

CORE LOG



Job number : RG0644
 Sample number : 01-35
 Core number : 7
 Date cored : 31/03/2001

Location : A14, Milton - FEN Ditton
 Westbound, Lane 1 Ch 100, N/S
 Nominal diameter : 150 mm

| Layer | Depth | | Thickness (mm) | Material description * | Binder B/T/C/N | Aggregate | |
|-------|-------|-----|-------------------|------------------------|-------------------|-----------|------|
| | From | To | | | | Size | Type |
| 1 | 0 | 40 | 40 | Thin Surfacing | B | 14 | CR |
| | | | | Layers 1&2 debonded | | | |
| 2 | 40 | 127 | 87 | DBM Upper RoadBase | B | 20 | CR |
| | | | | Layers 2&3 debonded | | | |
| 3 | 127 | 264 | 137 | DBM Lower RoadBase | B | 40 | CR |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |



* The material description given (such as hot rolled asphalt or dense bitumen macadam) is generic only and is based upon a visual assessment of the material. Any layer description (such as base course or roadbase) has been similarly assessed, using best engineering judgement.

- Key :-**
- Binder
 B = Bitumen
 T = Tar
 C = Cement
 N = None
- Aggregate Size
 40 = 40 mm
 28 = 28 mm
 20 = 20 mm
 14 = 10-14 mm
 6 = 3-6mm
- Aggregate Type
 CR = Crushed rock
 G = Gravel
 S = Slag
 O = Other

Prepared by :-
 NAL

Checked by :-

Date :- 02/05/2001

The scale is for guidance only. It does not necessarily reflect the actual thicknesses of individual layer(s). The actual layer thicknesses are presented in the above table.

Appendix B – Survey of Durability Tests

From the literature review (see Chapter 2), it is evident that there are a considerably large number of ageing and moisture sensitivity tests that have been and/or are being used to assess the durability of bituminous binders and asphalt mixtures. In order to identify which of these tests are currently applicable, a survey of bitumen and asphalt practitioners was undertaken. The survey questionnaires (see Section B-1) were sent to forty organisations (see Section B-2) worldwide, with a 50% response rate (20/40). Based on the results collected, the following summaries are presented:

Short-term ageing of bituminous binders

17 of the 20 organisations undertook short-term ageing tests;

94% of these used the RTFOT (CEN EN 12607-1, AASHTO T240, ASTM D2872) as their standard test;

One organisation used the TFOT (CEN EN 12607-2);

One organisation used the RFT (CEN EN 12607-3, DIN 52016);

One organisation used UV radiation.

Long-term ageing of bituminous binders

17 of the 20 organisations undertook long-term ageing tests;

94% of these used the RTFOT + PAV (AASHTO PP1) as their standard test;

One organisation used the RTFOT + HiPAT (BBA HAPAS SG4 Doc 062);

One organisation used the RCAT (Verhasselt, 2000);

One organisation used UV radiation.

Short-term ageing of asphalt mixtures

9 of the 20 organisations undertook short-term ageing tests, specifically;

AASHTO PP2, or;

2 – 4 hours @ 135°C on loose aggregate.

Long-term ageing of asphalt mixtures

8 of the 20 organisations undertook long-term ageing tests, specifically;

AASHTO PP2, or;

2 – 8 days @ 85°C, or;

BBA HAPAS SG3/98/173, Appendix A12.

Moisture damage (stripping) tests on loose aggregates

11 of the 20 organisations undertook stripping tests on loose aggregates, specifically;

Rolling Bottle Method (CEN prEN 12697-11), or;

Boiling Water Test (ASTM D3625), or;

Boiling Water Stripping Test (BRRC method ME65/91), or;

Ancona Stripping Test (Bocci and Colagrande, 1993).

Moisture damage tests on compacted asphalt mixtures

All 20 organisations undertook moisture damage tests on compacted asphalt mixtures;

25% used the Modified Lottman Procedure (AASHTO T283);

15% used the Duriez Procedure (NF P 98 251-1);

10% used the Lottman Procedure. The remainder used;

Environmental Conditioning System (ECS) (AASHTO TP34), or;

Effect of water on tensile strength (ASTM D4867), or;

Water sensitivity protocol (BBA HAPAS SG3/98/173 Appendix A2, or;

Retained Marshall Stability (AASHTO T245).

None of the organisations indicated that they undertook combined ageing and moisture damage tests to assess the ageing and/or stripping resistance of bituminous materials.

B-1: Moisture Sensitivity and Ageing Questionnaire

Name of Organisation:

Name of Contact Person:

Ageing

1 Ageing tests performed on bituminous binders

1.1 Does your organisation use **short-term ageing** (relative hardening that occurs during the mixing and laying process) procedures for bituminous binders?

YES NO

If YES, please list which test method(s) is/are used and whether the test methods are standard procedures or internal (in-house) test protocols.

1.2 Does your organisation use **long-term ageing** (relative hardening that occurs in the field) procedures for bituminous binders?

YES NO

If YES, please list which test method(s) is/are used and whether the test methods are standard procedures or internal (in-house) test protocols.

2 Ageing tests performed on bituminous mixtures

2.1 Does your organisation use **short-term ageing** (relative hardening that occurs during the mixing and laying process) procedures for bituminous mixtures?

YES NO

If YES, please list which test method(s) is/are used and whether the test methods are standard procedures or internal (in-house) test protocols.

2.2 Does your organisation use **long-term ageing** (relative hardening that occurs in the field) procedures for bituminous mixtures?

YES NO

If YES, please list which test method(s) is/are used and whether the test methods are standard procedures or internal (in-house) test protocols.

Moisture Damage

1 Moisture tests performed on loose coated aggregate

Does your organisation use moisture damage tests on loose coated aggregates?

YES NO

If YES, please list which test method(s) is/are used and whether the test methods are standard procedures or internal (in-house) test protocols.

2 Moisture tests performed on compacted bituminous mixtures

Does your organisation use moisture damage tests on compacted bituminous mixtures?

YES NO

If YES, please list which test method(s) is/are used and whether the test methods are standard procedures or internal (in-house) test protocols.

Combined Moisture Damage & Ageing

Has your organisation used a combined moisture damage/ageing test to assess the ageing of bituminous materials in the presence of water in one test rather than in two separate tests?

YES NO

If YES, please list the test method(s) used and whether the test method(s) is/are standard procedures or internal (in-house) test protocols.

B-2: List of Organisations Contacted (responded - ✓)

| | |
|--|---|
| Bitumes et Specialites Shell Global Solutions (France) | ✓ |
| Penn State University (USA) | x |
| University of Wisconsin (USA) | ✓ |
| Fortum Oil and Gas Oy (NESTE) (Finland) | x |
| NCAT, Auburn University (USA) | ✓ |
| Jean Lefebvre (France) | ✓ |
| University of Illinois at Urbana-Champaign (USA) | x |
| BP Bitumen | ✓ |
| Laboratoire Central des Ponts et Chaussees (LCPC) (France) | ✓ |
| Oldcastle Inc (USA) | x |
| ENTPE (France) | x |
| Scott Wilson Pavement Engineering | ✓ |
| Universita Degli Studi Di Parma (Italy) | ✓ |
| Refined Bitumen Association | x |
| University of California at Berkeley (USA) | ✓ |
| Royal Institute of Technology (Sweden) | ✓ |
| CRREL (USA) | x |
| Delft University (The Netherlands) | x |
| ARRB (Australia) | ✓ |
| Tarmac | x |
| University of Florida (USA) | x |
| AB Nynas Petroleum (Sweden) | ✓ |
| SHRP Equipment Corporation (USA) | x |
| Auburn University (USA) | ✓ |
| Dansk Vejteknologi (Denmark) | x |
| Cowl (Denmark) | x |
| Ooms Avenhorn Holdings bv (The Netherlands) | ✓ |
| Belgium Road Research Centre (Belgium) | ✓ |
| Transportek, CSIR (South Africa) | x |
| Danish Road Institute (Denmark) | ✓ |
| Arizona State University (USA) | x |
| Heritage Research Group (USA) | ✓ |
| Koch Materials (USA) | ✓ |
| Foster Yeoman | x |
| Hanson | x |
| Aggregate Industries | x |
| Lafarge | x |
| Esso Bitumen | x |
| TotalFinaElf | ✓ |
| Staffordshire Highways Laboratory | ✓ |

Appendix C. Binder Characterisation Tests

C.1 Introduction

This chapter describes the binder characterisation tests performed on the four binders used in the research. A series of tests were performed on the unconditioned binders as well as after short term (RTFOT) and long term (HiPAT) ageing.

C.1.1 Binders

Three 15 penetration grade binders (referred to as 15B, 15D and 15E) and a 50 penetration grade binder (referred to as 50C) were tested.

C.1.2 Ageing Processes

The above binders were subjected to short term (RTFOT) and long term (HiPAT) ageing. Details of these two procedures can be found respectively, in references [EN 12607-1, 1999; BBA/HAPAS SG4, 2001].

C.2 Penetration Test

The penetration test [BS 2000-49, 2000] was performed at 25°C and 40°C for this project. Table C.1 shows the individual results.

C.3 Softening Point Test

The Softening Point test was performed as described in references [BS 2000-58, 2000]. The results are presented in Table C.2.

Table C.1 – Penetration test results

| Binder | 15B | | 15D | | 15E | | 50C | |
|--------|-----|----|-----|----|-----|----|-----|-----|
| | 25 | 40 | 25 | 40 | 25 | 50 | 25 | 40 |
| Unaged | 15 | 62 | 14 | 39 | 15 | 70 | 53 | 263 |
| RTFOT | 11 | 36 | 8 | 30 | 13 | 41 | 35 | 146 |
| HiPAT | 6 | 20 | 4 | 16 | 7 | 25 | 17 | 69 |

Table C.2 – Softening point test results

| Binder | 15B | 15D | 15E | 50C |
|--------|------|------|------|------|
| Unaged | 68.0 | 73.0 | 62.0 | 49.0 |
| RTFOT | 75.4 | 79.0 | 67.0 | 55.0 |
| HiPAT | 96.0 | 98.0 | 78.0 | 65.0 |

C.4 Rotational Viscosity Test

This test was performed to investigate the viscosity of binder as described in reference [BS 2000-505, 2003]. The test was performed in a temperature range of 60°C to 135°C. The results are presented in Table C.3.

C.5 Bitumen Test Data Chart

The results from the penetration, softening point and viscosity tests for 15B, 15D, 15E and 50C are presented using the bitumen test data chart (BTDC) in Figures C.1 to C.4.

Table C.3 – Rotational viscosity test results

| 15B Unaged | | 15D Unaged | | 15E Unaged | | 50C Unaged | |
|------------|------------------|------------|------------------|------------|------------------|------------|------------------|
| Temp. | Viscosity (Pa.s) |
| 60 | 2280.0 | 60 | | 60 | 2300.0 | 60 | 335.0 |
| 80 | 343.4 | 80 | 960.3 | 80 | 154.3 | 100 | 4.4 |
| 100 | 34.2 | 100 | 83.5 | 100 | 17.0 | 135 | 0.5 |
| 135 | 2.1 | 135 | 4.0 | 135 | 1.4 | | |
| | | | | | | | |
| 15B RTFOT | | 15D RTFOT | | 15E RTFOT | | 50C RTFOT | |
| Temp. | Viscosity (Pa.s) |
| 80 | 1452.3 | 80 | 0.0 | 80 | 345.1 | 60 | 883.2 |
| 100 | 95.5 | 100 | 188.3 | 100 | 32.9 | 100 | 8.0 |
| 135 | 4.3 | 135 | 6.9 | 135 | 2.0 | 135 | 0.8 |
| | | | | | | | |
| 15B HiPAT | | 15D HiPAT | | 15E HiPAT | | 50C HiPAT | |
| Temp. | Viscosity (Pa.s) |
| 100 | 1311.5 | 80 | 0.0 | 80 | 1174.0 | 60 | 2300.0 |
| 135 | 24.4 | 100 | 1481.0 | 100 | 86.4 | 80 | 217.2 |
| | | 135 | 26.1 | 135 | 4.1 | 100 | 23.7 |
| | | | | | | 135 | 1.5 |

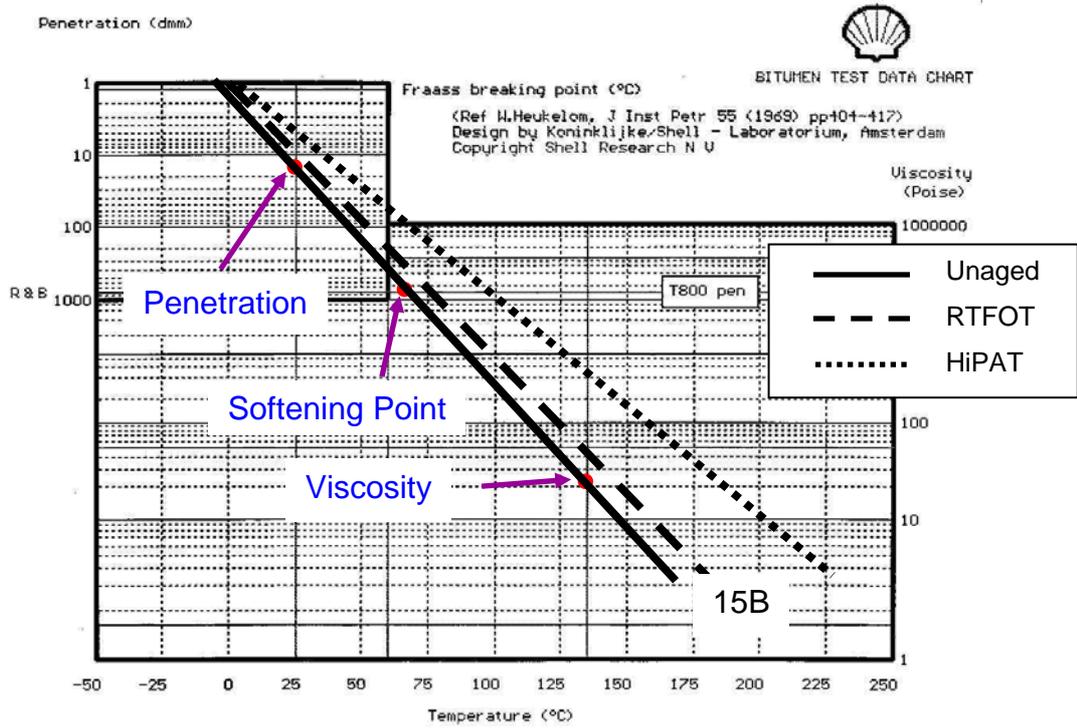


Figure C.1 – BTDC plot of 15B binder

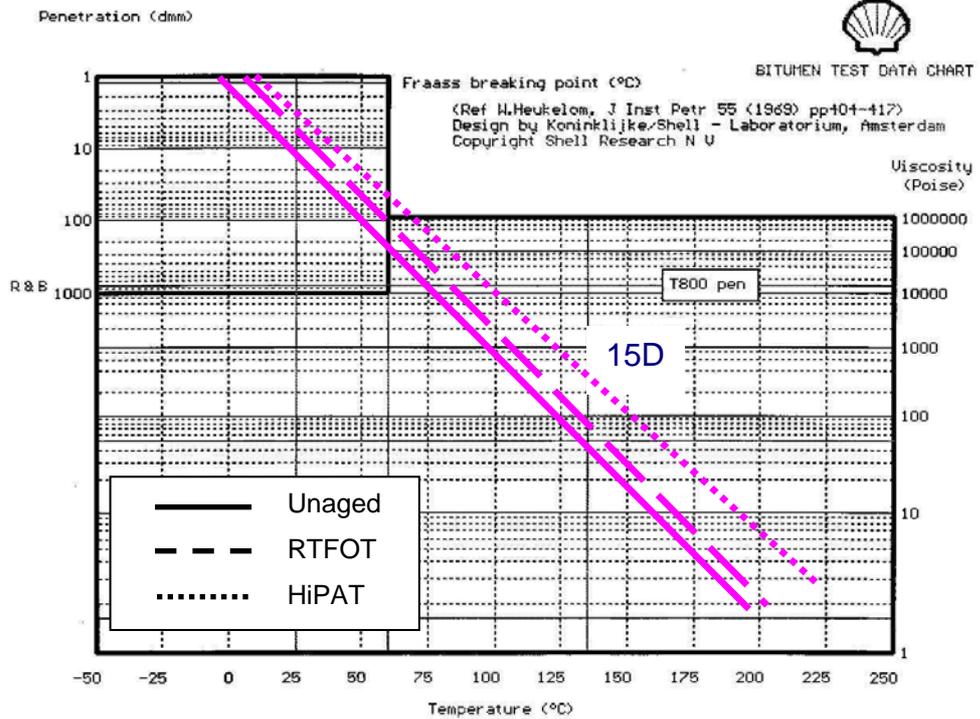


Figure C.2 – BTDC plot of 15D binder

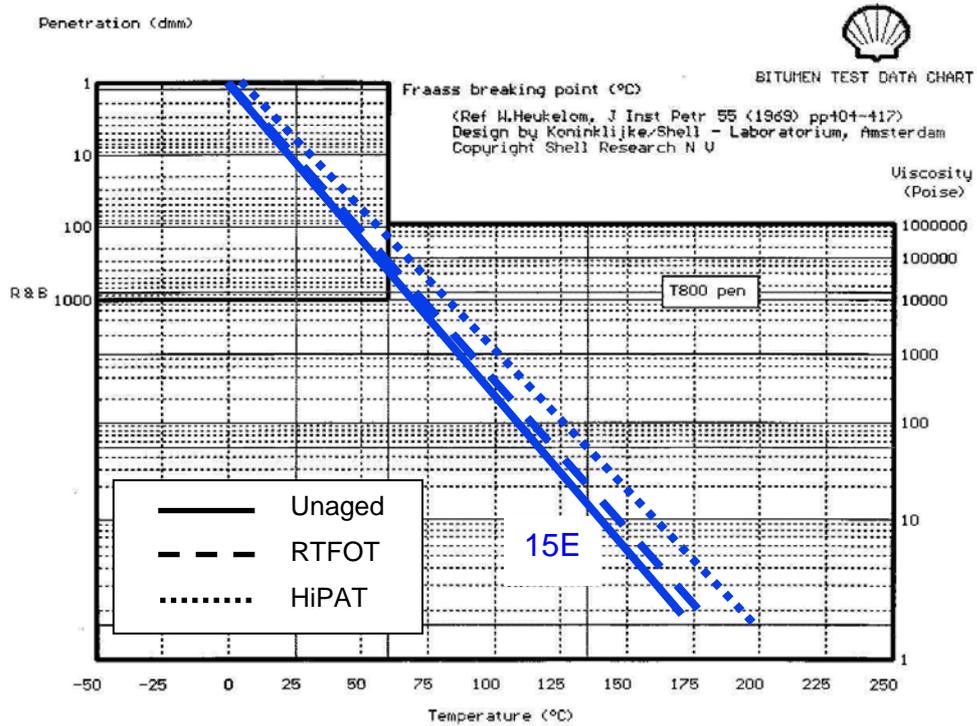


Figure C.3 – BTDC plot of 15E binder

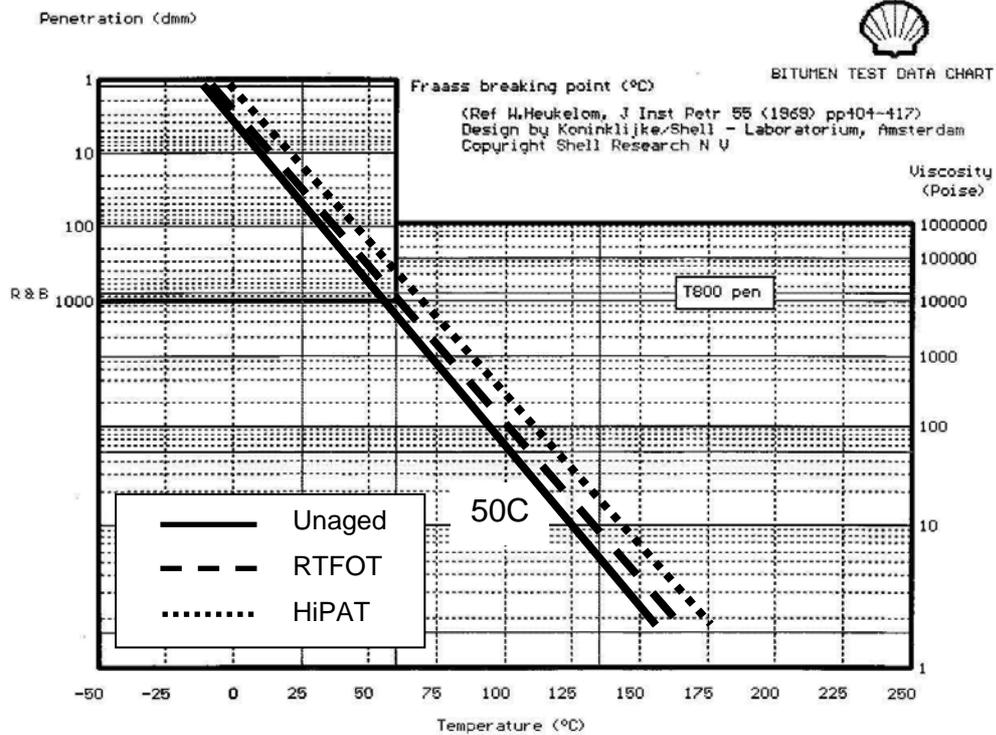


Figure C.4 – BTDC plot of 50C binder

C.6 Dynamic Shear Rheometer (DSR) Test

The rheological characteristics of the binders were measured using the DSR [AASHTO TP5] over an applied frequency range of 0.1 to 10 Hz and a temperature range of -5 to 80°C. Three different parallel plate geometries were used over the temperature range.

The standard 25 mm geometry was used between 40 to 80°C, the standard 8 mm geometry was used between 10 to 50°C and the 8 mm spindle with a 10 mm diameter thick shaft was used between -5 to 20°C. Strain sweeps were performed at a frequency of 1.0 Hz at each of the testing temperature, prior to the frequency sweep tests to ensure that the tests were conducted within the linear viscoelastic response of the binder (see Figure C.5). Results from each test were then merged to produce a master curve (see Figures C.6 and C.7).

Master curves of complex modulus and phase angle at a reference temperature of 25°C are presented in Figures C.8 to C.11.

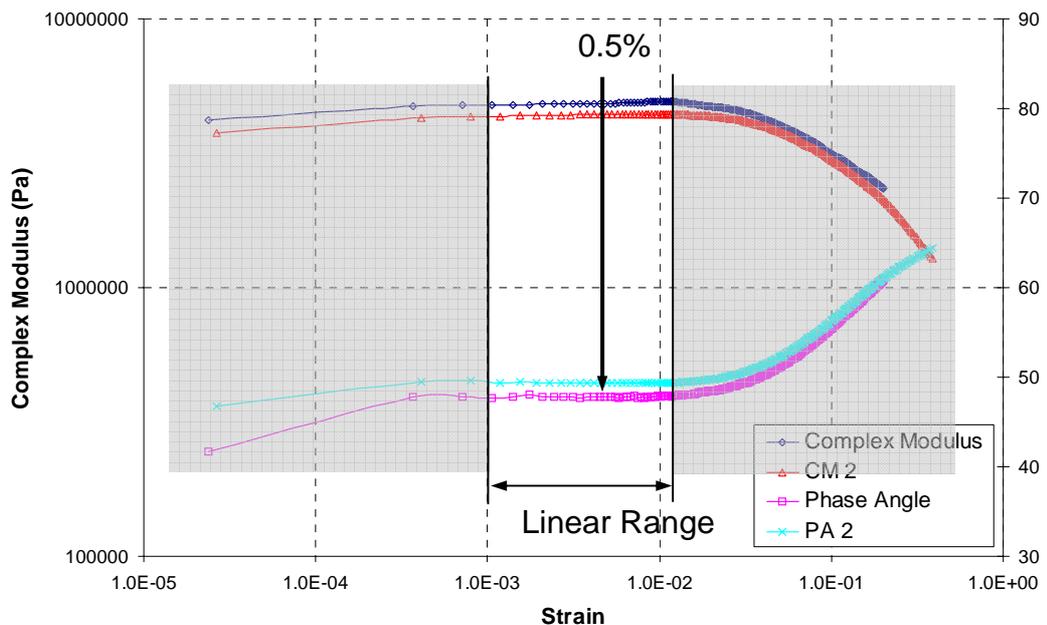


Figure C.5 – An example of strain sweep test.

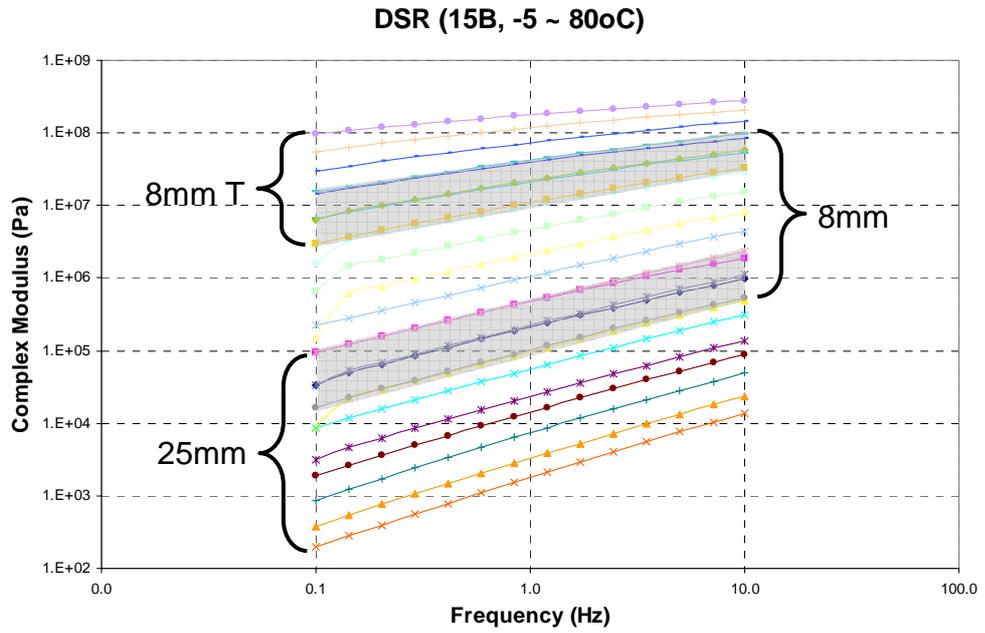


Figure C.6 – Typical complex modulus (G^*) results from a binder

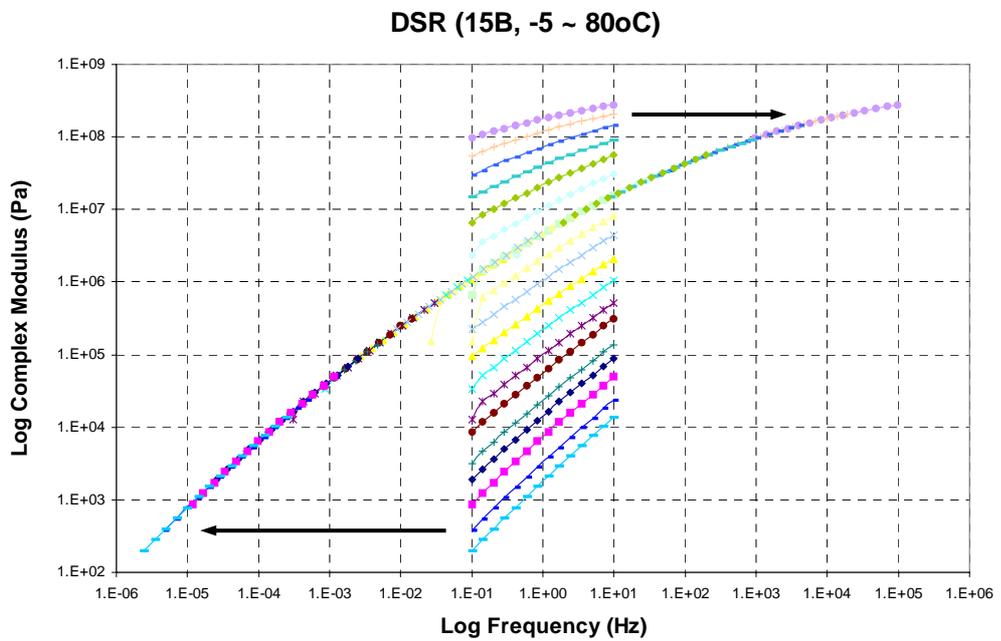


Figure C.7 – Typical complex modulus (G^*) master curve for a binder.

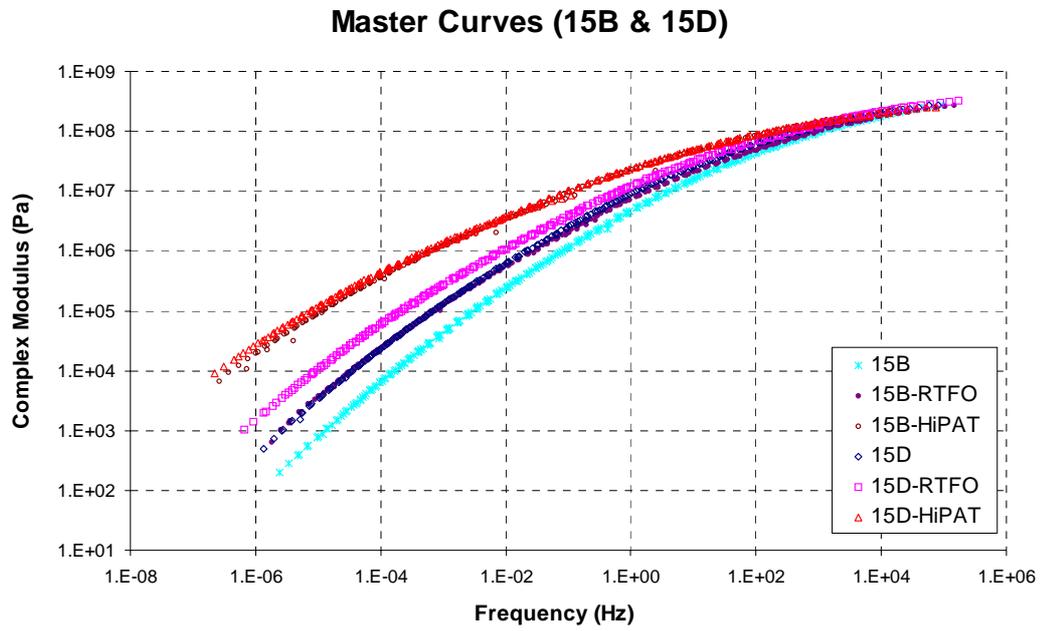


Figure C.8 – Complex Modulus (G^*) master curves of 15B and 15D binders.

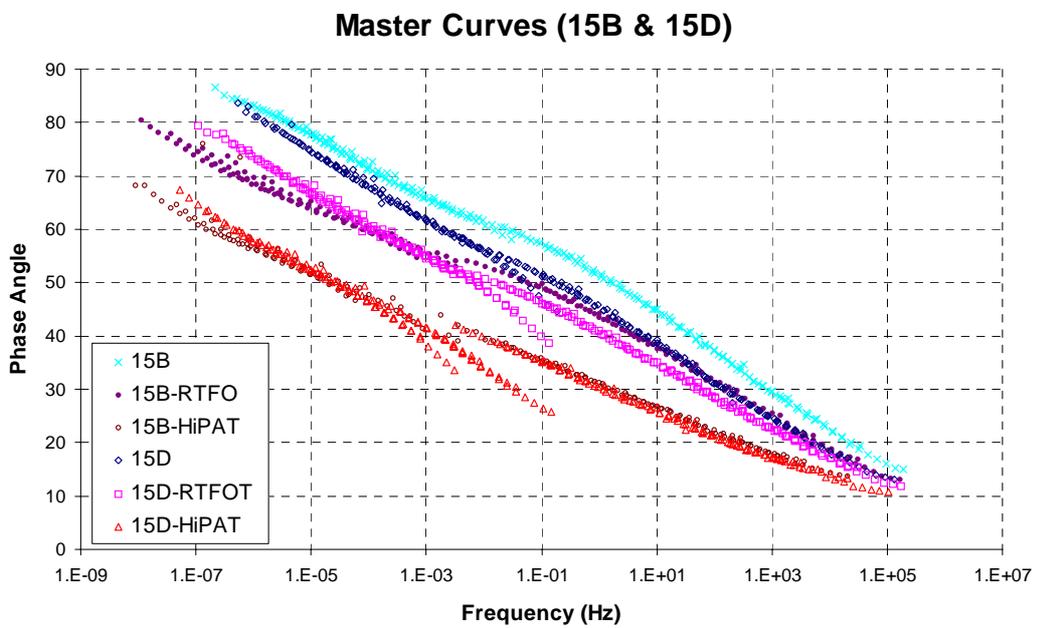


Figure C.9 – Phase Angle (δ) master curves of 15B and 15D binders.

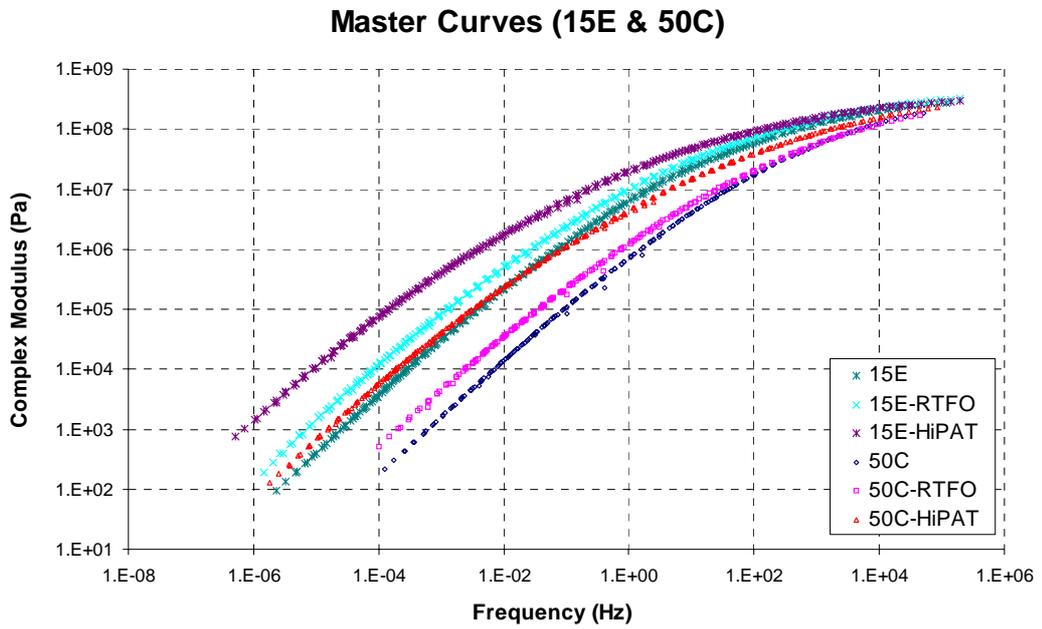


Figure C.10 – Complex Modulus (G^*) master curves of 15E and 50C binders.

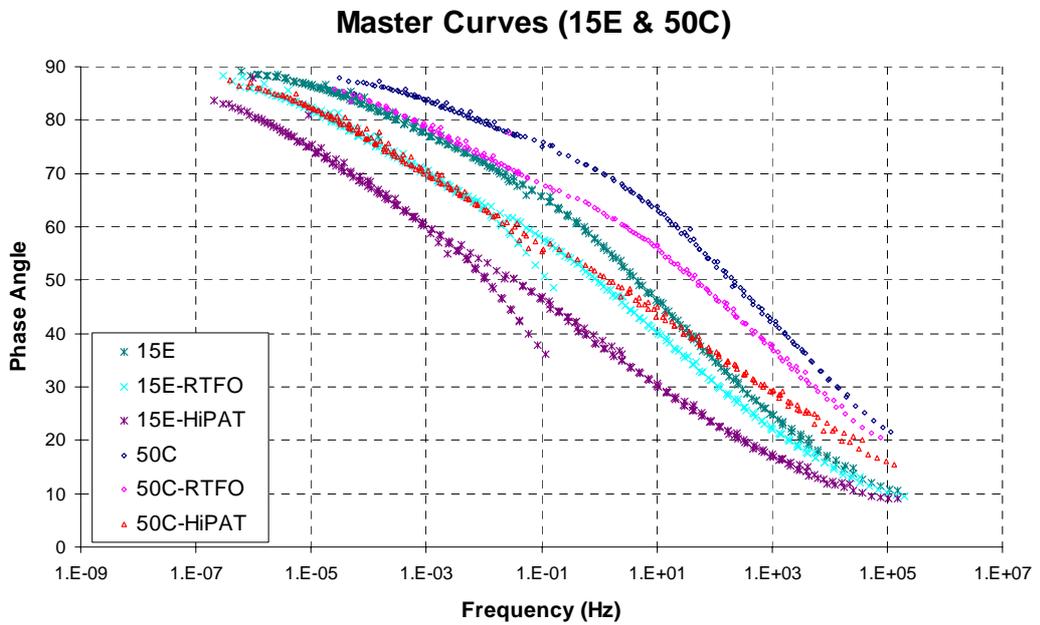


Figure C.11 – Phase Angle (δ) master curves of 15E and 50C binders.

C.6.1 Review of Dynamic Shear Rheometer Results

From Figures C.8 to C.11, binders 15B and 15E (unaged) have similar G^* values but 15E is more viscous (i.e. higher δ), particularly at low/intermediate frequencies. Among the 15 Pen grade binders, 15B showed the largest change in G^* values and phase angle (δ) due to ageing, while 15E showed the least. 15D showed the highest G^* value and lowest phase angle (δ) among the unaged 15 Pen binders, but showed almost identical characteristics after long-term ageing.

Long-term aged 50C binder showed similar G^* values to those of the 15 Pen unaged binders (particularly 15B and 15E), while the phase angles (δ) showed similar values to those of 15B and 15D.

Draft for Development

Method for the Assessment of Durability of Compacted Asphalt Mixtures using the Saturated Ageing Tensile Stiffness (SATS) Test

1. Scope

This Draft for Development specifies a test method to assess the sensitivity of asphalt pavement materials to a combined ageing moisture sensitivity procedure, by measuring stiffness before and after the test.

The test method is intended to simulate the effect of ageing and moisture on asphalt pavement materials in service. It is suitable for coated macadam base and binder course mixtures with a nominal air void content of 8% and a nominal binder content of 4%. The test may not be suitable for mixtures of high binder content or manufactured with binders of penetration higher than 50.

2. Normative References

This Draft for Development incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this Draft for Development only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

British Standard Draft for Development DD 213: 1993 *Method for determination of the indirect tensile stiffness modulus of bituminous mixtures.*

British Standard BS EN 12697-5: 2002 *Bituminous mixtures – Test Methods for hot mix asphalt Part 5: Determination of the maximum density.*

British Standard BS 598-100: 1987 (1996) *Sampling and examination of bituminous mixtures for roads and other paved areas Part 100: Methods for sampling for analysis.*

British Standard BS 598-104: 1989 *Sampling and examination of bituminous mixtures for roads and other paved areas Part 104: Method of test for the determination of density and compaction.*

British Standard BS 6100 (1984 on) *Glossary of building and civil engineering terms.*

AASHTO Designation PP1, Edition 1A: 1993 *Standard practice for accelerated ageing of asphalt binder using a pressurized ageing vessel (PAV).*

3. Terms and Definitions

For the purposes of this Draft for Development the following terms and definitions given in BS 6100 apply, together with the following.

3.1 Maximum Density

Mass per unit volume, without air voids, of a bituminous mixture at known test temperature.

3.2 Dry Bulk Density

Mass per unit volume, including the air voids, of a specimen at known test temperature.

3.3 Saturation Before Conditioning

The saturation of the mixture, determined as the calculated percentage of air voids filled with water after partial saturation, prior to conditioning as described in this Draft for Development.

3.4 Unconditioned Stiffness

The stiffness modulus of the mixture as determined in accordance with BS DD 213: 1993, prior to conditioning as described in this Draft for Development.

3.5 Conditioned Stiffness

The stiffness modulus of the mixtures as determined in accordance in accordance with BS DD 213: 1993, after conditioning as described in this Draft for Development.

3.6 Stiffness Ratio

The ratio of the conditioned stiffness to the unconditioned stiffness.

3.7 Saturation After Conditioning

The saturation of the mixture, determined as the calculated percentage of air voids filled with water after conditioning as described in this Draft for Development.

4. Principle of Test

Nominally identical test specimens are subjected to moisture saturation by using a vacuum system and then transferred into a pressurised ageing vessel partially filled with water, where they are subjected to a conditioning procedure at 85°C temperature and 2.1 MPa pressure for 65 hours. The stiffness of individual specimens after the conditioning procedure, expressed as a percentage of the stiffness before the conditioning procedure, is plotted against their percentage saturation to determine the sensitivity of the material to ageing and moisture. The whole process is referred to as the saturated ageing tensile stiffness (SATS) test.

5. Materials

- 5.1** De-aired (distilled) water, freshly de-aired and cooled
- 5.2** Self-adhesive aluminium foil: Capable of adhering to and covering the specimen with an impervious coating.
- 5.3** Absorbent paper towelling.

6. Apparatus

- 6.1** Vacuum desiccator and vacuum pump, including manometer or calibrated vacuum gauge as described in BS EN 12697-5.
- 6.2** Balance with sufficient capacity and accurate to at least 1g.
- 6.3** Pressure Vessel as described in AASHTO PP1, and of sufficient capacity to accommodate five test specimens for a full test. Dimensions of a suitable vessel are shown in Figure 1.
- 6.4** Nottingham Asphalt Tester (NAT) capable of performing the Indirect Tensile Stiffness Modulus (ITSM) test on compacted bituminous mixture specimens in accordance with BS DD213.
- 6.5** Specimen tray, to accommodate five test specimens for a full test. Dimensions of a suitable tray are shown in Figure 2.

7. Sample Preparation

Test specimens shall be cores of $100 + 0, -5$ mm diameter and 60 ± 5 mm thickness. The standard procedure involves specimens cored from a slab manufactured using a laboratory roller compactor. Five nominally identical specimens are required for the full test. Specimens for test may also be cores from an in-service pavement in accordance with BS 598-100.

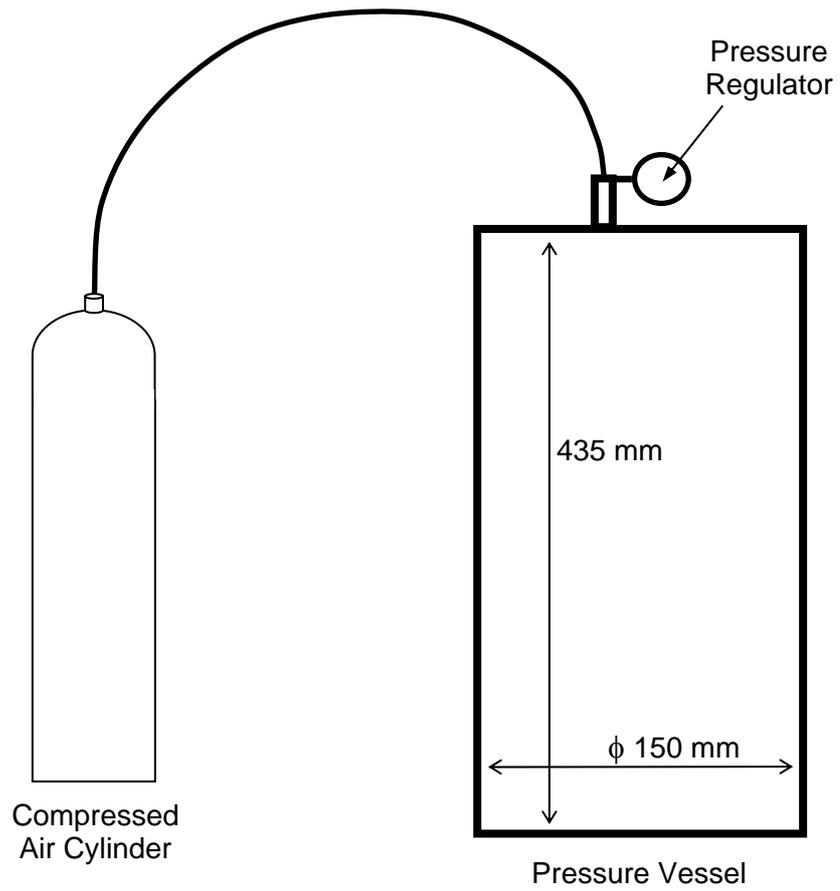


Figure 1: Schematic and Nominal Dimensions of Typical Pressure Vessel

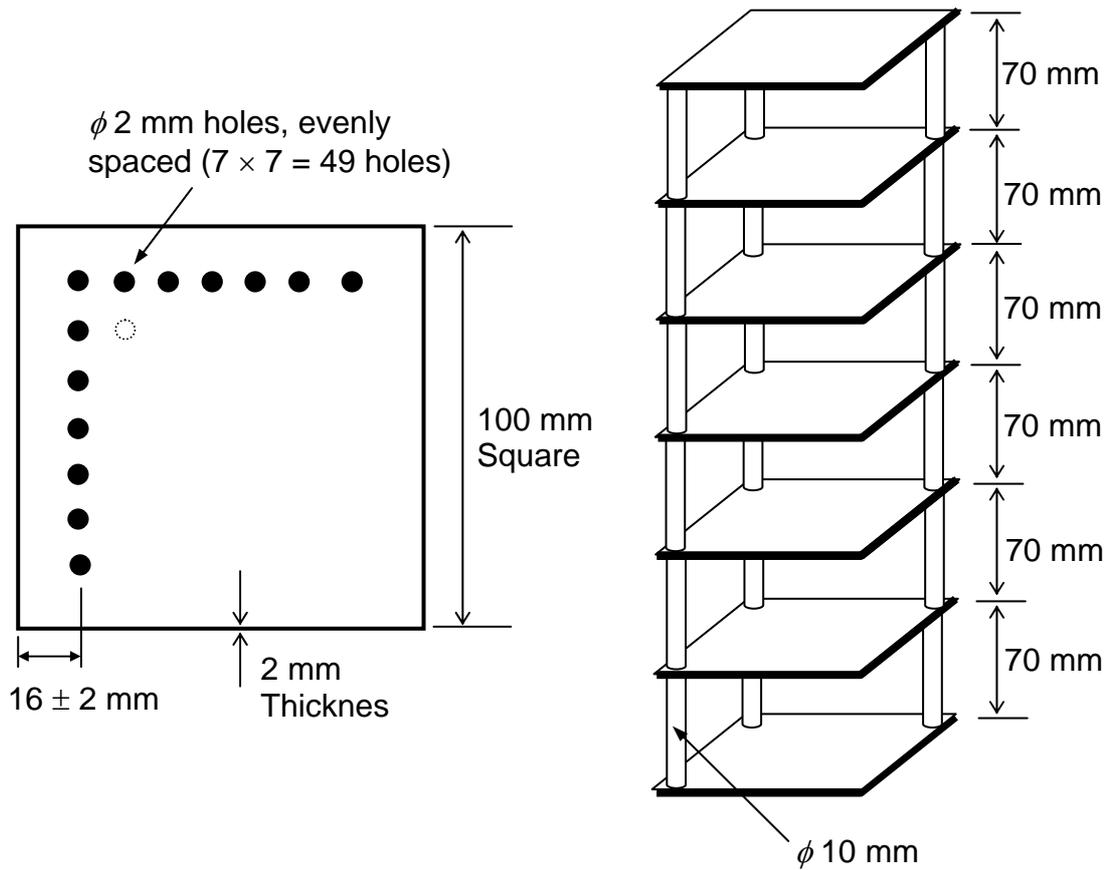


Figure 2: Schematic and Nominal Dimensions of Typical Specimen Tray

8. Procedure

- 8.1** The maximum density of the bituminous mixture under test shall be determined as described in BS EN 12697-5.
- 8.2** Determine the bulk density of each compacted specimen as described in BS598-104, except that self-adhesive aluminium foil shall be used to seal the specimen during immersion, in place of the wax recommended in BS 598. Remove the foil after the test.
- 8.3** Calculate the air voids of each specimen as described in Clause 9.1 below. Reject any specimen whose calculated air voids are outside the range $8 \pm 2\%$, and replace with further samples of suitable void content.

- 8.4** Determine the unconditioned (initial) stiffness modulus at $20 \pm 5^\circ\text{C}$ in the NAT. Designate this as ITSM_{U} .
- 8.5** Determine the dry mass of each specimen using the balance and designate this as M_{d} .
- 8.6** Place the specimens in the vacuum desiccator, cover them with distilled water at $20 \pm 1^\circ\text{C}$, seal the apparatus, and apply a partial vacuum pressure of 68 ± 3 kPa for 30 minutes.
- 8.7** Remove each specimen from the vacuum desiccator, remove any water on its surface using the absorbent paper towelling and determine its wet mass. Designate this as M_{w} . Determine the percent saturation (S) as described in Clause 9.2 below. Reject any specimens whose calculated saturation is $\geq 80\%$.
- 8.8** Partly fill the pressure vessel with water. The water level should be between the 4th and 5th specimen, as shown in the schematic in Figure 3. Use **freshly distilled water** only (**do not re-use**). The vessel and water should be controlled to the target temperature at least 2 hours before the conditioning procedure commences.
- 8.9** Place the specimens into the vessel, using the specimen tray, and perform the conditioning procedure, as described in AASHTO PP1, at 2.1 ± 0.1 MPa pressure and $85 \pm 1^\circ\text{C}$ for 65 ± 1 h.
- 8.10** After the test period of 65h has elapsed, **turn off the heater** and **release the pressure** from the vessel slowly. Adjust the pressure release mechanism so that the pressure returns to atmospheric pressure over a period of 20 to 30 minutes. **Do not open the cover at this stage.**
- 8.11** When the pressure vessel display temperature has reduced to 70°C (this may take up to 3 hours) open the cover and extract all the specimens, on the tray, from the vessel.

- 8.12** Surface dry each specimen using the absorbent paper towelling, and measure its wet mass as soon as possible (within 3 minutes of removing the specimens from the pressure vessel). Designate this mass as M_{w2} and calculate the saturation after the conditioning procedure (S_a) as described in Clause 9.3 below.
- 8.13** Observe the specimens for any visual cracks and unusual appearance (i.e. colour of binder or aggregates). Record any comments as appropriate.
- 8.14** Place the specimens into the NAT or other suitable conditioning environment at $20 \pm 0.5^\circ\text{C}$ in preparation for ITSM testing, as described in BS DD 213.
- 8.15** Determine the stiffness modulus as described in BS DD 213 within 5 hours after completing the conditioning procedure. Designate this as ITSM_C .

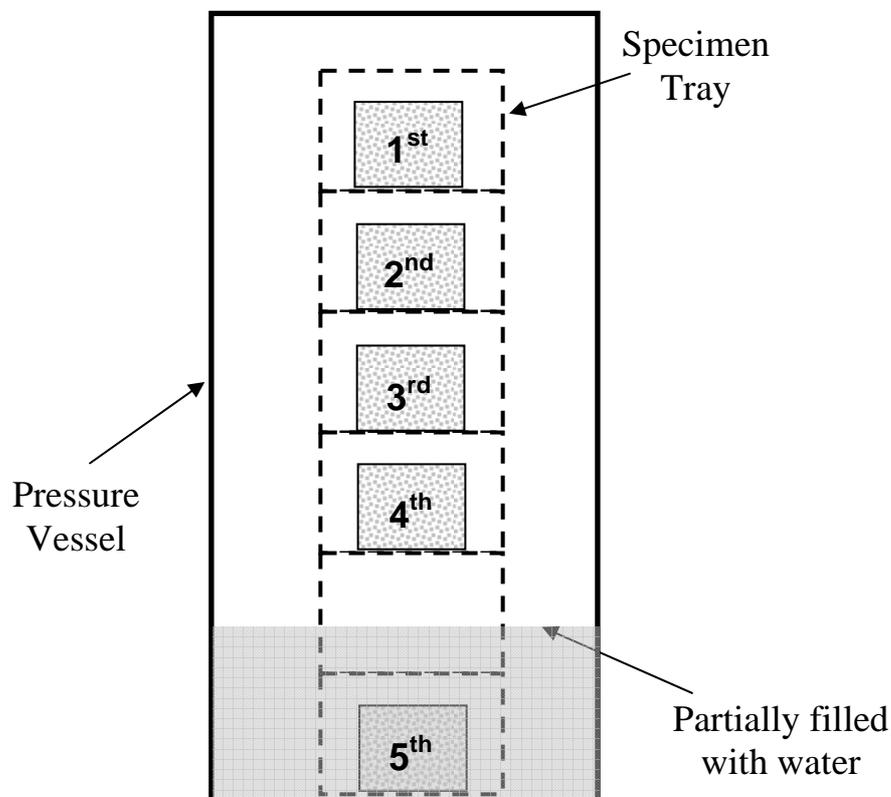


Figure 3: Schematic Diagram of the Specimen Configuration in the Pressure Vessel

9. Calculation

9.1 Dry bulk density and calculated air voids

Calculate the **dry bulk density** of each specimen (in Mg/m³) as follows:

$$\text{Dry Bulk Density } (G_{mb}) = \frac{A}{(B - C) - \left(\frac{B - A}{D}\right)}$$

where:

A = the mass of the specimen in air (in g)

B = the mass of the coated specimen in air (in g)

C = the mass of the coated specimen in water (in g)

D = the density* of the self-adhesive aluminium foil in Mg/m³

The material described in Clause 5.2 has been found to have a typical density of 1.65Mg/m³

Express the specimen bulk density to the nearest 0.001Mg/m³

Calculate the **air voids** of each specimen (in %) as follows:

$$\text{Air Voids} = \frac{G_{mm} - G_{mb}}{G_{mm}} \times 100$$

where

G_{mm} is the maximum density determined in accordance with BS EN 12697-5

G_{mb} is the bulk density determined in accordance with Clause 8.2 above

Express the calculated air voids to the nearest 0.1%.

9.2 Saturation before conditioning

Calculate the saturation before conditioning as follows:

$$S = \frac{\frac{M_w - M_d}{G_{mb}} - \frac{M_d}{G_{mm}}}{\frac{M_d}{G_{mb}} - \frac{M_d}{G_{mm}}} \times 100$$

where:

S = percent saturation

M_d = mass of dry specimen, g,

M_w = mass of wet specimen, g,

G_{mb} = bulk density and

G_{mm} = maximum density

Express the saturation to the nearest 1%.

9.3 Saturation after conditioning

Calculate the saturation after conditioning as follows:

$$S_a = \frac{M_{w2} - M_d}{\frac{M_d}{G_{mb}} - \frac{M_d}{G_{mm}}} \times 100$$

where:

S_a = saturation after conditioning

M_d = mass of dry specimen, g,

M_{w2} = mass of wet specimen (g) after conditioning

G_{mb} = bulk density and

G_{mm} = maximum density

9.4 Stiffness ratio

Calculate the stiffness ratio as follows:

$$ITSM_R = \frac{ITSM_C}{ITSM_U}$$

where:

$ITSM_R$ = stiffness ratio,

$ITSM_C$ = conditioned stiffness

$ITSM_U$ = unconditioned stiffness

Express the stiffness ratio to the nearest 0.01

10. Precision

The precision for this test method has not been established.

11. Report

With reference to this Draft for Development, the test report shall include the following information:

- a) Reference to this test method and test conditions
- b) Maximum density of the mixture tested, to the nearest 0.001 Mg/m³
- c) For each specimen tested, report
 - Bulk density (G_{mb}), to the nearest 0.001 Mg/m³
 - Percent air voids (V_v), to the nearest 0.1%
 - Percent saturation (S) before conditioning, to the nearest 1%
 - Percent saturation after conditioning, (S_a), to the nearest 1%
 - Unconditioned stiffness ($ITSM_U$) before conditioning, to the nearest 100 MPa
 - Conditioned stiffness ($ITSM_C$) after conditioning, to the nearest 100 MPa
 - Stiffness ratio ($ITSM_R$), to the nearest 0.01
 - Any cracks or other damage.
- d) For the mixture tested, the stiffness ratio of the individual specimens tested shall be plotted against the corresponding saturation value after saturation. A graphical representation of a typical set of data is shown in Figure 4.

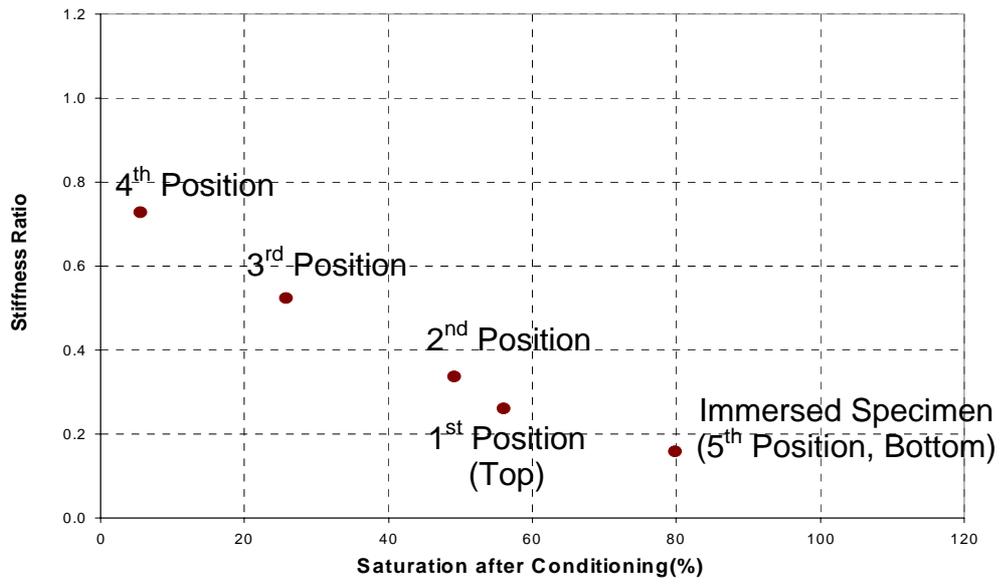


Figure 4: Graphical Representation of Typical Stiffness/Saturation Data

| | | | | | | | | | | |
|------|---------------------------------|------------|------|------|-------------|-------------|-------------|-------------|-------------|-------------|
| 5-1 | 01-215 | 15B Acidic | 7.37 | 88.0 | 10613 | 13012 | 9049 | 8230 | 7568 | 6999 |
| PAV | 01-346 | 15B Acidic | 8.33 | 73.5 | 10133 | 10056 | 8455 | 7969 | 7344 | 7055 |
| | Mean | | | | 10373 | 11534 | 8752 | 8099 | 7456 | 7027 |
| | Ratio to After Ageing Stiffness | | | | 0.90 | 1.00 | 0.76 | 0.70 | 0.65 | 0.61 |
| | Ratio to Initial Stiffness | | | | 1.00 | 1.11 | 0.84 | 0.78 | 0.72 | 0.68 |
| 5-2 | 01-213 | 15B Acidic | 6.43 | 92.5 | 10786 | 11066 | 9161 | 8338 | 7875 | 7389 |
| PAV | 01-351 | 15B Acidic | 9.64 | 69.3 | 9760 | 9777 | 7834 | 7004 | 6337 | 5906 |
| | Mean | | | | 10273 | 10422 | 8497 | 7671 | 7106 | 6648 |
| | Ratio to After Ageing Stiffness | | | | 0.99 | 1.00 | 0.82 | 0.74 | 0.68 | 0.64 |
| | Ratio to Initial Stiffness | | | | 1.00 | 1.01 | 0.83 | 0.75 | 0.69 | 0.65 |
| 6 | 01-352 | 15B Acidic | 8.68 | 75.8 | 9342 | 3435 | | | | |
| SATS | 01-353 | 15B Acidic | 9.08 | 68.0 | 9474 | 4092 | | | | |
| | Mean | | | | 9408 | 3764 | | | | |
| | Ratio to After Ageing Stiffness | | | | 2.50 | 1.00 | | | | |
| | Ratio to Initial Stiffness | | | | 1.00 | 0.40 | | | | |

| Chapter 4 - Individual Results (Section 4.2, Figure 4.2) | | | | | | | | | | |
|---|-----------------|--------------|---------------|------------------|-------------------|------------------|-----------------------------|---------------------------|--------------------|-------------------|
| Test No. | Specimen Number | Mixture Type | Air Voids (%) | Initial Sat. (%) | Retained Sat. (%) | Saturation Ratio | Initial Stiff. @ 20°C (MPa) | Final Stiff. @ 20°C (MPa) | Retained Stiffness | Specimen Position |
| 1 | 02-250a | 15B Acidic | 9.01 | 47.4 | | | 7807 | 1886 | 0.24 | 1 |
| | 02-250b | 15B Acidic | 8.14 | 50.6 | | | 8193 | 1879 | 0.23 | 2 |
| | 02-250c | 15B Acidic | 7.64 | 50.5 | | | 8391 | 2561 | 0.31 | 3 |
| | 02-251a | 15B Acidic | 7.61 | 52.2 | | | 8018 | 4727 | 0.59 | 4 |
| | 02-251b | 15B Acidic | 9.53 | 49.1 | | | 7232 | 4871 | 0.67 | 5 |
| | 02-251c | 15B Acidic | 8.70 | 51.5 | | | 8184 | 5932 | 0.72 | 6 |
| 2 | 02-663 | 15B Acidic | 7.74 | 50.4 | | | 7778 | 1676 | 0.22 | 1 |
| | 02-664 | 15B Acidic | 6.45 | 50.6 | | | 8026 | 4291 | 0.53 | 2 |
| | 02-665 | 15B Acidic | 6.47 | 49.1 | | | 8493 | 5649 | 0.67 | 3 |
| | 02-668 | 15B Acidic | 7.11 | 57.1 | | | 7368 | 6072 | 0.82 | 4 |
| | 02-670 | 15B Acidic | 7.08 | 54.5 | | | 8688 | 5858 | 0.67 | 5 |
| | 02-671 | 15B Acidic | 7.63 | 56.8 | | | 7868 | 6054 | 0.77 | 6 |
| 3 | 02-976 | 15B Acidic | 7.70 | 71.3 | | | 8999 | 2545 | 0.28 | 1 |
| | 02-969 | 15B Acidic | 7.60 | 74.3 | | | 9610 | 2863 | 0.30 | 2 |
| | 02-971 | 15B Acidic | 8.08 | 70.1 | | | 9546 | 3253 | 0.34 | 3 |
| | 02-970 | 15B Acidic | 7.16 | 70.5 | | | 9525 | 4809 | 0.50 | 4 |
| | 02-974 | 15B Acidic | 7.71 | 73.5 | | | 9984 | 5866 | 0.59 | 5 |
| | 02-975 | 15B Acidic | 7.49 | 75.7 | | | 8955 | 5192 | 0.58 | 6 |
| 4 | 02-1003 | 15B Acidic | 7.95 | 62.8 | | | 8561 | 1954 | 0.23 | 1 |
| | 02-1008 | 15B Acidic | 8.70 | 54.0 | | | 9738 | 2363 | 0.24 | 2 |
| | 02-1006 | 15B Acidic | 7.04 | 52.5 | | | 9061 | 3135 | 0.35 | 3 |
| | 02-1002 | 15B Acidic | 7.74 | 60.3 | | | 8644 | 2531 | 0.29 | 4 |
| | 02-1001 | 15B Acidic | 6.89 | 55.7 | | | 10920 | 5063 | 0.46 | 5 |
| | 02-1007 | 15B Acidic | 8.74 | 52.1 | | | 8625 | 6108 | 0.71 | 6 |

| | | | | | | | | | | |
|---|---------|------------|------|------|------|------|-------|------|-------------|---|
| 5 | 02-977 | 15B Acidic | 7.48 | 59.0 | 53.3 | 0.90 | 8211 | 2784 | 0.34 | 1 |
| | 02-972 | 15B Acidic | 7.35 | 57.1 | 41.7 | 0.73 | 8847 | 3331 | 0.38 | 2 |
| | 02-1009 | 15B Acidic | 7.66 | 55.0 | 24.6 | 0.45 | 7812 | 3075 | 0.39 | 3 |
| | 02-1012 | 15B Acidic | 7.66 | 58.9 | 15.7 | 0.27 | 7692 | 4939 | 0.64 | 4 |
| | 02-1013 | 15B Acidic | 7.86 | 49.8 | 13.7 | 0.28 | 7807 | 5822 | 0.75 | 5 |
| | 02-1014 | 15B Acidic | 9.33 | 50.3 | 1.5 | 0.03 | 8500 | 7515 | 0.88 | 6 |
| | | | | | | | | | | |
| 6 | 02-1323 | 15B Acidic | 5.74 | 52.8 | 52.5 | 0.99 | 9077 | 2743 | 0.30 | 1 |
| | 02-1324 | 15B Acidic | 6.45 | 50.4 | 11.9 | 0.24 | 10811 | 5488 | 0.51 | 2 |
| | 02-1326 | 15B Acidic | 5.17 | 50.5 | 14.6 | 0.29 | 11288 | 7350 | 0.65 | 3 |
| | 02-1327 | 15B Acidic | 5.05 | 45.2 | 11.9 | 0.26 | 9486 | 8141 | 0.86 | 4 |
| | 02-1328 | 15B Acidic | 6.04 | 50.5 | 5.6 | 0.11 | 9255 | 7810 | 0.84 | 5 |
| | 02-1336 | 15B Acidic | 5.11 | 49.6 | 13.4 | 0.27 | 10562 | 8041 | 0.76 | 6 |
| | | | | | | | | | | |
| | | | | | | | | | | |
| 7 | 02-416 | 15B Basic | 7.93 | 52.9 | | | 10067 | 6424 | 0.64 | 1 |
| | 02-417 | 15B Basic | 7.42 | 57.3 | | | 9637 | 7317 | 0.76 | 2 |
| | 02-418 | 15B Basic | 8.03 | 51.4 | | | 10551 | 9014 | 0.85 | 3 |
| | 02-421 | 15B Basic | 7.60 | 53.5 | | | 12203 | 9589 | 0.79 | 4 |
| | 02-422 | 15B Basic | 7.75 | 49.3 | | | 10268 | 8220 | 0.80 | 5 |
| | 02-424 | 15B Basic | 7.04 | 53.9 | | | 10811 | 8459 | 0.78 | 6 |

| | | | | | | | | | | | |
|---|-------|-----|-------|------|------|------|------|-------|------|-------------|---|
| 5 | 03-13 | 15B | Basic | 8.00 | 72.0 | 58.3 | 0.81 | 11634 | 6953 | 0.60 | 1 |
| | 03-14 | 15B | Basic | 7.15 | 75.5 | 66.2 | 0.88 | 11402 | 7913 | 0.69 | 2 |
| | 03-16 | 15B | Basic | 8.28 | 71.9 | 43.5 | 0.61 | 10242 | 6595 | 0.64 | 3 |
| | 03-17 | 15B | Basic | 9.07 | 73.5 | 32.0 | 0.44 | 9669 | 7337 | 0.76 | 4 |
| | 03-18 | 15B | Basic | 8.65 | 76.3 | 72.7 | 0.95 | 10255 | 8072 | 0.79 | 6 |
| 6 | 03-20 | 15B | Basic | 9.59 | 69.1 | 51.0 | 0.74 | 9400 | 6488 | 0.69 | 1 |
| | 03-21 | 15B | Basic | 8.56 | 69.6 | 39.2 | 0.56 | 9454 | 6735 | 0.71 | 2 |
| | 03-22 | 15B | Basic | 8.54 | 65.4 | 35.3 | 0.54 | 9825 | 6670 | 0.68 | 3 |
| | 03-23 | 15B | Basic | 9.12 | 67.5 | 31.1 | 0.46 | 11249 | 8452 | 0.75 | 4 |
| | 03-24 | 15B | Basic | 8.65 | 74.4 | 74.9 | 1.01 | 9799 | 8001 | 0.82 | 6 |
| 7 | 03-25 | 15B | Basic | 8.85 | 79.6 | 64.2 | 0.81 | 9649 | 6656 | 0.69 | 1 |
| | 03-27 | 15B | Basic | 9.24 | 72.7 | 52.5 | 0.72 | 10567 | 7413 | 0.70 | 2 |
| | 03-28 | 15B | Basic | 9.35 | 61.7 | 35.3 | 0.57 | 9554 | 6758 | 0.71 | 3 |
| | 03-29 | 15B | Basic | 9.28 | 70.7 | 35.5 | 0.50 | 9263 | 6662 | 0.72 | 4 |
| | 03-30 | 15B | Basic | 9.13 | 73.5 | 67.5 | 0.92 | 8655 | 5776 | 0.67 | 6 |
| 8 | 03-32 | 15B | Basic | 9.00 | 73.1 | 51.9 | 0.71 | 9151 | 6089 | 0.67 | 1 |
| | 03-33 | 15B | Basic | 7.57 | 79.8 | 54.4 | 0.68 | 9716 | 6749 | 0.69 | 2 |
| | 03-34 | 15B | Basic | 9.15 | 74.3 | 47.3 | 0.64 | 9732 | 7051 | 0.72 | 3 |
| | 03-35 | 15B | Basic | 7.82 | 76.9 | 32.7 | 0.42 | 10222 | 7872 | 0.77 | 4 |
| | 03-36 | 15B | Basic | 9.31 | 76.0 | 71.1 | 0.94 | 9929 | 7053 | 0.71 | 6 |

| | | | | | | | | | | | |
|---|---------|-----------|---------|------|------|-------|------|-------|-------|-------------|---|
| 5 | 02-3139 | 15B Basic | Low AV | 3.97 | 41.4 | 45.9 | 1.11 | 14894 | 7673 | 0.52 | 1 |
| | 02-3140 | 15B Basic | Low AV | 4.41 | 37.9 | 29.2 | 0.77 | 14205 | 7649 | 0.54 | 2 |
| | 02-3141 | 15B Basic | Low AV | 3.84 | 35.2 | 22.8 | 0.65 | 14216 | 7783 | 0.55 | 3 |
| | 02-3142 | 15B Basic | Low AV | 3.02 | 29.1 | 11.9 | 0.41 | 12922 | 8912 | 0.69 | 4 |
| | 02-3143 | 15B Basic | Low AV | 3.90 | 40.8 | 89.6 | 2.20 | 15351 | 10462 | 0.68 | 6 |
| 6 | 02-3144 | 15B Basic | Low AV | 4.07 | 36.0 | 40.7 | 1.13 | 14540 | 7628 | 0.52 | 1 |
| | 02-3145 | 15B Basic | Low AV | 3.58 | 24.2 | 20.2 | 0.83 | 13194 | 7637 | 0.58 | 2 |
| | 02-3146 | 15B Basic | Low AV | 4.05 | 56.3 | 24.9 | 0.44 | 14661 | 7507 | 0.51 | 3 |
| | 02-3147 | 15B Basic | Low AV | 3.82 | 29.9 | 14.5 | 0.48 | 12828 | 8656 | 0.67 | 4 |
| | 02-3148 | 15B Basic | Low AV | 4.42 | 28.6 | 73.0 | 2.55 | 13133 | 9819 | 0.75 | 6 |
| 7 | 02-3168 | 15B Basic | High BC | 7.90 | 55.1 | 47.7 | 0.87 | 10107 | 5200 | 0.51 | 1 |
| | 02-3169 | 15B Basic | High BC | 6.78 | 52.5 | 31.1 | 0.59 | 9475 | 5460 | 0.58 | 2 |
| | 02-3170 | 15B Basic | High BC | 7.66 | 49.1 | 27.0 | 0.55 | 8461 | 4851 | 0.57 | 3 |
| | 02-3173 | 15B Basic | High BC | 6.56 | 46.9 | 20.9 | 0.45 | 8245 | 5069 | 0.61 | 4 |
| | 02-3174 | 15B Basic | High BC | 7.78 | 46.9 | 74.6 | 1.59 | 8436 | 6064 | 0.72 | 6 |
| 8 | 02-3175 | 15B Basic | High BC | 7.28 | 53.8 | 52.0 | 0.97 | 8332 | 4435 | 0.53 | 1 |
| | 02-3176 | 15B Basic | High BC | 7.00 | 49.3 | 29.7 | 0.60 | 9323 | 5031 | 0.54 | 2 |
| | 02-3177 | 15B Basic | High BC | 8.69 | 50.2 | 28.1 | 0.56 | 8819 | 4446 | 0.50 | 3 |
| | 02-3178 | 15B Basic | High BC | 8.27 | 53.6 | 22.1 | 0.41 | 9858 | 6077 | 0.62 | 4 |
| | 02-3179 | 15B Basic | High BC | 7.29 | 51.4 | 77.5 | 1.51 | 8784 | 5822 | 0.66 | 6 |
| 9 | 02-3231 | 15E Basic | EME2 | 4.33 | 63.6 | 54.4 | 0.86 | 9123 | 4157 | 0.46 | 1 |
| | 02-3232 | 15E Basic | EME2 | 4.38 | 71.2 | 48.6 | 0.68 | 9320 | 4482 | 0.48 | 2 |
| | 02-3233 | 15E Basic | EME2 | 4.02 | 64.9 | 40.6 | 0.62 | 9197 | 5008 | 0.54 | 3 |
| | 02-3234 | 15E Basic | EME2 | 4.44 | 69.2 | 39.4 | 0.57 | 9435 | 4857 | 0.51 | 4 |
| | 02-3235 | 15E Basic | EME2 | 4.31 | 67.8 | 115.6 | 1.71 | 10085 | 6385 | 0.63 | 6 |

| | | | | | | | | | | | | |
|-------|---------|-----|--------|-------|------|-------|------|-------|------|------|---|---------------|
| 7.3.1 | 02-3597 | 15B | Acidic | 7.89 | 59.2 | 96.8 | 1.63 | 9496 | N/A | | 1 | Old Tray |
| | 02-3598 | 15B | Acidic | 9.19 | 48.3 | 68.5 | 1.42 | 9104 | N/A | | 2 | Half Pressure |
| | 02-3599 | 15B | Acidic | 7.36 | 59.8 | 72.5 | 1.21 | 9492 | 1252 | 0.13 | 3 | |
| | 02-3600 | 15B | Acidic | 7.31 | 61.7 | 39.5 | 0.64 | 9185 | 2401 | 0.26 | 4 | |
| | 02-3601 | 15B | Acidic | 6.97 | 63.7 | | 0.00 | 10103 | N/A | | 6 | |
| 7.3.1 | 03-2507 | 15B | Basic | 8.65 | 60.7 | 64.9 | 1.07 | 9573 | 5931 | 0.62 | 1 | New Tray |
| | 03-2508 | 15B | Basic | 9.35 | 65.3 | 62.4 | 0.96 | 9636 | 7508 | 0.78 | 2 | Half Pressure |
| | 03-2509 | 15B | Basic | 9.68 | 62.2 | 50.6 | 0.81 | 10071 | 8018 | 0.80 | 3 | |
| | 03-2510 | 15B | Basic | 7.30 | 65.7 | 53.7 | 0.82 | 9843 | 6892 | 0.70 | 4 | |
| | 03-2511 | 15B | Basic | 7.32 | 70.2 | 84.8 | 1.21 | 11240 | 8648 | 0.77 | 6 | |
| 7.3.1 | 03-2857 | 15B | Basic | 8.94 | 60.4 | 55.1 | 0.91 | 9857 | 7111 | 0.72 | 1 | Old Tray |
| | 03-2858 | 15B | Basic | 9.83 | 59.6 | 45.9 | 0.77 | 9079 | 6468 | 0.71 | 2 | Half Pressure |
| | 03-2859 | 15B | Basic | 8.89 | 61.8 | 47.8 | 0.77 | 10397 | 7413 | 0.71 | 3 | |
| | 03-2860 | 15B | Basic | 8.10 | 57.8 | 38.2 | 0.66 | 10579 | 7650 | 0.72 | 4 | |
| | 03-2861 | 15B | Basic | 9.32 | 58.0 | 68.2 | 1.18 | 9762 | 8275 | 0.85 | 6 | |
| 7.3.2 | 03-3056 | 15B | Acidic | 8.56 | 61.0 | 100.4 | 1.65 | 10235 | 1759 | 0.17 | 1 | New Tray |
| | 03-3057 | 15B | Acidic | 9.51 | 53.2 | 81.6 | 1.53 | 8254 | 1550 | 0.19 | 2 | Atmospheric |
| | 03-3058 | 15B | Acidic | 7.98 | 55.2 | 92.4 | 1.67 | 10177 | 1822 | 0.18 | 3 | |
| | 03-3061 | 15B | Acidic | 9.26 | 50.3 | 56.3 | 1.12 | 9811 | 1800 | 0.18 | 4 | |
| | 03-3062 | 15B | Acidic | 7.93 | 61.3 | 140.8 | 2.30 | 9876 | 1013 | 0.10 | 6 | |
| 7.3.2 | 03-2862 | 15B | Basic | 8.71 | 57.7 | 50.7 | 0.88 | 10612 | 9771 | 0.92 | 1 | New Tray |
| | 03-2863 | 15B | Basic | 8.50 | 59.3 | 56.1 | 0.95 | 10059 | 9384 | 0.93 | 2 | Atmospheric |
| | 03-2864 | 15B | Basic | 8.91 | 58.4 | 42.6 | 0.73 | 9665 | 8978 | 0.93 | 3 | |
| | 03-2865 | 15B | Basic | 10.04 | 52.9 | 32.0 | 0.60 | 9150 | 8912 | 0.97 | 4 | |
| | 03-2868 | 15B | Basic | 9.61 | 56.5 | 61.3 | 1.08 | 9341 | 9077 | 0.97 | 6 | |

| | | | | | | | | | | | | |
|-----|--------|-----|-------|------|------|------|------|-------|-------|------|---|----------|
| 7.5 | 04-530 | 15B | Basic | 9.17 | 52.0 | 40.3 | 0.78 | 11522 | 11502 | 1.00 | 1 | New Tray |
| | 04-532 | 15B | Basic | 8.47 | 62.5 | 48.6 | 0.78 | 12146 | 12160 | 1.00 | 2 | 30°C |
| | 04-533 | 15B | Basic | 8.38 | 60.0 | 47.0 | 0.78 | 10988 | 11217 | 1.02 | 3 | |
| | 04-534 | 15B | Basic | 8.28 | 59.3 | 46.5 | 0.78 | 11317 | 11326 | 1.00 | 4 | |
| | 04-535 | 15B | Basic | 8.18 | 59.2 | 80.8 | 1.36 | 11842 | 12845 | 1.08 | 6 | |

| Chapter 8 - Individual Results (Section 8.3.5, Figures 8.10 and 8.11) | | | | | | | | | | | | |
|--|-----------------|--------------|--------|---------------|------------------|-------------------|------------------|-----------------------------|---------------------------|--------------------|-------------------|--------------|
| Test No. | Specimen Number | Mixture Type | | Air Voids (%) | Initial Sat. (%) | Retained Sat. (%) | Saturation Ratio | Initial Stiff. @ 20°C (MPa) | Final Stiff. @ 20°C (MPa) | Retained Stiffness | Specimen Position | Note |
| 1 | 04-1384 | 15B | Acidic | 9.76 | | | | 8903 | 9907 | 1.11 | 1 | Dry HiPAT |
| | 04-1385 | 15B | Acidic | 7.02 | | | | 10232 | 11454 | 1.12 | 3 | 30°C Depress |
| | 04-1401 | 15B | Acidic | 7.16 | | | | 9611 | 11857 | 1.23 | 5 | |
| | | | | | | | | | | 1.16 | Mean | |
| | 04-1405 | 15B | Basic | 6.93 | | | | 12271 | 15441 | 1.26 | 2 | Dry HiPAT |
| | 04-1410 | 15B | Basic | 7.09 | | | | 12196 | 15563 | 1.28 | 4 | 30°C Depress |
| | 04-531 | 15B | Basic | 9.51 | | | | 10237 | 13884 | 1.36 | 6 | |
| | | | | | | | | | | 1.30 | Mean | |
| | | | | | | | | | | | | |
| | | | | | | | | | | | | |
| 2 | 02-1696 | 15B | Acidic | 5.93 | | | | 9805 | 10269 | 1.05 | 1 | Dry HiPAT |
| | 02-2125 | 15B | Acidic | 6.82 | | | | 9557 | 10083 | 1.06 | 2 | 85°C Depress |
| | | | | | | | | | | 1.05 | Mean | |
| | | | | | | | | | | | | |
| 3 | 02-2476 | 15B | Basic | 8.81 | | | | 11508 | 9582 | 0.83 | 1 | Dry HiPAT |
| | 02-2477 | 15B | Basic | 8.68 | | | | 11008 | 9438 | 0.86 | 2 | 85°C Depress |
| | | | | | | | | | 9510 | 0.85 | Mean | |

NG953 (08/04) Method for the Assessment of Durability of Compacted Asphalt Mixtures using the Saturation Ageing Tensile Stiffness (SATS) Test

1 The test method is intended to simulate the combined effect of ageing and moisture on asphalt pavement materials in service. It is suitable for coated macadam base and binder course mixtures with a nominal (water accessible) air void content of 8% and a nominal binder content of 4%. The test may not be suitable for mixtures of high binder content or manufactured with binders of penetration greater than 50.

2 Clause 953 incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed in Appendix F. For dated references, subsequent amendments to or revisions of any of these publications apply to this Clause only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

3 Recent evidence from an early demonstration trial of High Modulus Base (HMB) materials in the UK showed an unexpected drop in material stiffness modulus (up to 60%) only a few years after construction. Other investigations into HMB materials that have been used for in-service pavements generally showed better performance, although some also displayed unexpected decreases in the stiffness modulus of the HMB material. It is thought that the sensitivity of the material to ageing and moisture is of key importance in helping to explain this behaviour. The main purpose of the SATS test is to reproduce in the laboratory the decrease in stiffness modulus that had been measured from the HMB trial site, so that unsuitable aggregate/binder combinations are screened out at mixture design stage.

4 With reference to sub-Clause 953.4 (ii), 100 mm wide 'Foil-faced self-adhesive tape' supplied by IDENT, has been found to be suitable. The tape can be obtained from WR Thermal Contracts, 1 Ludlam Avenue, Giltbrook, Nottingham, NG16 2UL (Tel: 07775 646 526).

5 With reference to sub-Clause 953.5 (v), a 100 mm diameter, 5 mm thick rigid porous disc for use with a standard soils triaxial cell as described in Clause 3.2 (g) of BS 1377: Part 8: 1990, has been found to be suitable.

6 The form and dimensions of the pressure vessel and specimen tray shown in Figures 9/9 and 9/10 have been found to yield repeatable results in the SATS test when used with two different aggregate types. Other forms of pressure vessel and specimen tray may also be suitable, but have not been specifically investigated. However, early work carried out in a standard binder pressure ageing vessel to AASHTO PP1 yielded similar results to those generated using the apparatus described in Clause 953.

7 The standard SATS test involves specimens cored from a slab manufactured using a laboratory roller compactor. Specimens for test may also be cored from an in-service pavement in accordance with BS 598-100, but research data on the performance of such specimens in the SATS test is not currently available.

8 The measurement of density on sealed specimens is considered essential for material with an air void content of 8%. Wax is not suitable as a sealing material since it is practically impossible to remove from specimens that will be subjected to subsequent mechanical testing. The self-adhesive aluminium foil described in sub-Clause NG 953.4 above has been successfully used in a number of laboratories in the UK for over 10 years, and is easy to remove. For laboratory prepared specimens with a rough surface texture, the foil may cause observation of texture voids as internal specimen voids, which may cause an under-estimation of the specimen density and an over-estimation of the air voids level of the specimen. However, the risk of this is considered to be relatively small in the SATS test specimens, which are normally cored from a laboratory manufactured slab and then trimmed (sawn) to the specified thickness.

9 The test temperature of 85°C and test duration of 65 hours are the same as those adopted for the High Pressure Ageing Test (HiPAT) developed for BBA/HAPAS (British Board of Agrément/Highway Authorities Product Approval Scheme) Specialist Group (SG) 4 for Modified Binders. The reduced temperature compared

with the standard conditions described in AASHTO PP1 was used to reduce the difference between test temperature and service temperature, with the extended time incorporated in compensation.

10 With reference to sub-Clause 953.16, the additional cooling period is applied to reduce the pressure related damage during the depressurising process. Normally the target temperature (30°C) can be achieved within 24 hours. It should be noted that the pressure needs to be carefully maintained during this cooling process, as the reducing temperature will reduce the vessel pressure accordingly. This can be achieved by carefully setup the pressure input controlling valve to just below the target pressure (2.1 MPa), thus any gradual lose of pressure will be automatically compensated by the gradual input of the pressure from the compressed air cylinder.

11 It was found during the development of the HiPAT that it was necessary to reduce pressure slowly, to avoid excessive foaming of binder due to release of dissolved air. A similar slow release of pressure is recommended at the end of the SATS conditioning procedure, to reduce damage to the specimens as a result of sudden changes of pressure. The influence of the method of pressure release in the test is the subject of ongoing research.

12 With reference to sub-Clause 953.18, 3 minutes has been found to be a practical time period to complete the surface drying and weighing of all five specimens after removal from the pressure vessel. Any reduction in this time period (below 3 minutes) should help to improve the precision of the test.

13 Figure NG 9/1 shows a suitable format for the graphical representation of the stiffness ratio versus saturation value plot. The data shown in the figure are examples only. Acceptable values for stiffness ratio at a given saturation after conditioning, for a mixture to be considered not susceptible to the combined effect of ageing and moisture in service, are the subject of ongoing research.

14 The precision for the SATS test method has not been established.

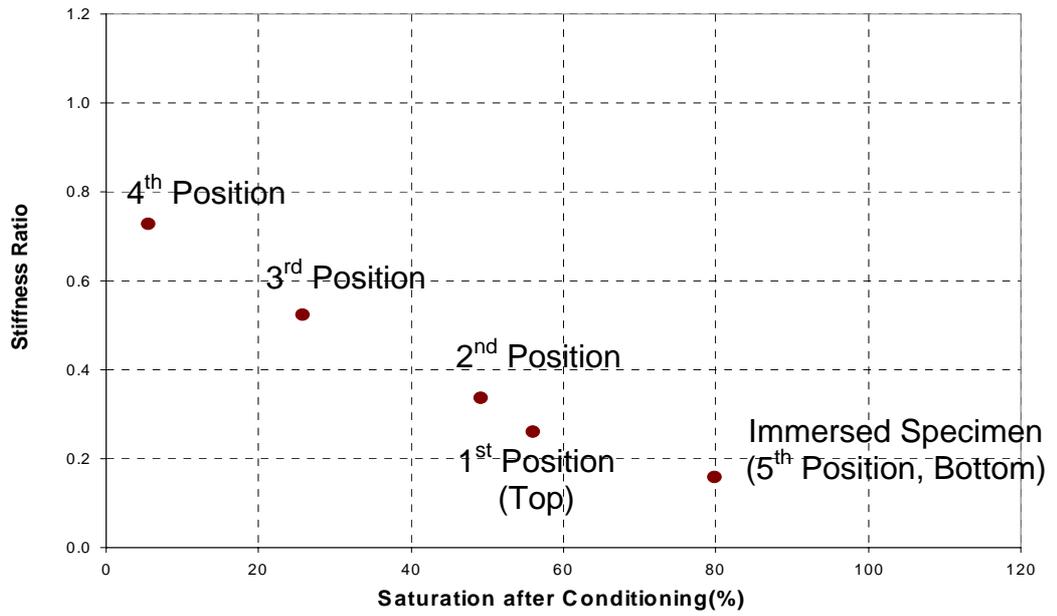


Figure NG 9/1: (08/04) Graphical Representation of Typical Stiffness/Saturation Data

953 (08/04) Method for the Assessment of Durability of Compacted Asphalt Mixtures using the Saturation Ageing Tensile Stiffness (SATS) Test

Scope

1 This Clause specifies a test method to assess the sensitivity of asphalt pavement materials to a combined ageing/moisture sensitivity procedure, by measuring stiffness before and after the test. The test is known as the Saturation Ageing Tensile Stiffness (SATS) test.

Terms and Definitions

2 For the purposes of this Clause the terms and definitions given in BS 6100 shall apply, together with the following.

(i) Maximum Density

Mass per unit volume, without air voids, of a bituminous mixture at known test temperature.

(ii) Dry Bulk Density

Mass per unit volume, including the air voids, of a specimen at known test temperature.

(iii) Saturation Before Conditioning

The saturation of the mixture, determined as the calculated percentage of air voids filled with water after partial vacuum saturation, prior to conditioning as described in this Clause.

(iv) Unconditioned Stiffness

The stiffness modulus of the mixture as determined in accordance with BS DD 213:1993, prior to conditioning as described in this Clause.

(v) Conditioned Stiffness

The stiffness modulus of the mixture as determined in accordance with BS DD 213:1993, after conditioning as described in this Clause.

(vi) Stiffness Ratio

The ratio of the conditioned stiffness to the unconditioned stiffness

(vii) Saturation After Conditioning

The saturation of the mixture, determined as the calculated percentage of air voids filled with water after conditioning as described in this Clause.

Principle of Test

3 Nominally identical test specimens shall be subjected to moisture saturation by using a vacuum system. They shall then be transferred into a pressurised vessel partially filled with water, where they shall be subjected to a conditioning procedure at 85°C temperature and 2.1 MPa pressure for 65 hours. The stiffness ratio of individual specimens shall be plotted against their percentage saturation after conditioning to determine the sensitivity of the material to ageing and moisture. The whole process is referred to as the Saturation Ageing Tensile Stiffness (SATS) test.

Materials

4 The following materials shall be used:

- (i) Distilled water, freshly de-aired and cooled.
- (ii) Self-adhesive aluminium foil: Capable of adhering to and covering the specimen with an impervious coating.
- (iii) Absorbent paper towelling.

Test Apparatus

5 The following test apparatus shall be used:

- (i) Vacuum desiccator and vacuum pump, including manometer or calibrated vacuum gauge as described in BS EN 12697-5.
- (ii) Balance with sufficient capacity and accurate to 1.0 g.
- (iii) Pressure Vessel having the form and dimensions specified in Figure 9/9, similar to that described in AASHTO PP1, and of sufficient capacity to accommodate 5 test specimens, of dimensions as described in sub-Clause 6 of this Clause, for a full test.
- (iv) Test equipment capable of performing the Indirect Tensile Stiffness Modulus (ITSM) test on compacted bituminous mixture specimens in accordance with BS DD 213.
- (v) Specimen tray, having the form and dimensions specified in Figure 9/10, to accommodate five test specimens for a full test. The tray shall sit in the pressure vessel on top of a porous disc as shown in Figure 9/11.

Sample Preparation

6 Test specimens shall be cores of $100 \pm 0, -5$ mm diameter and 60 ± 5 mm thickness. Specimens shall be cored from a slab manufactured using a laboratory roller compactor in accordance with prEN12697-33. Five nominally identical specimens are required for the full test.

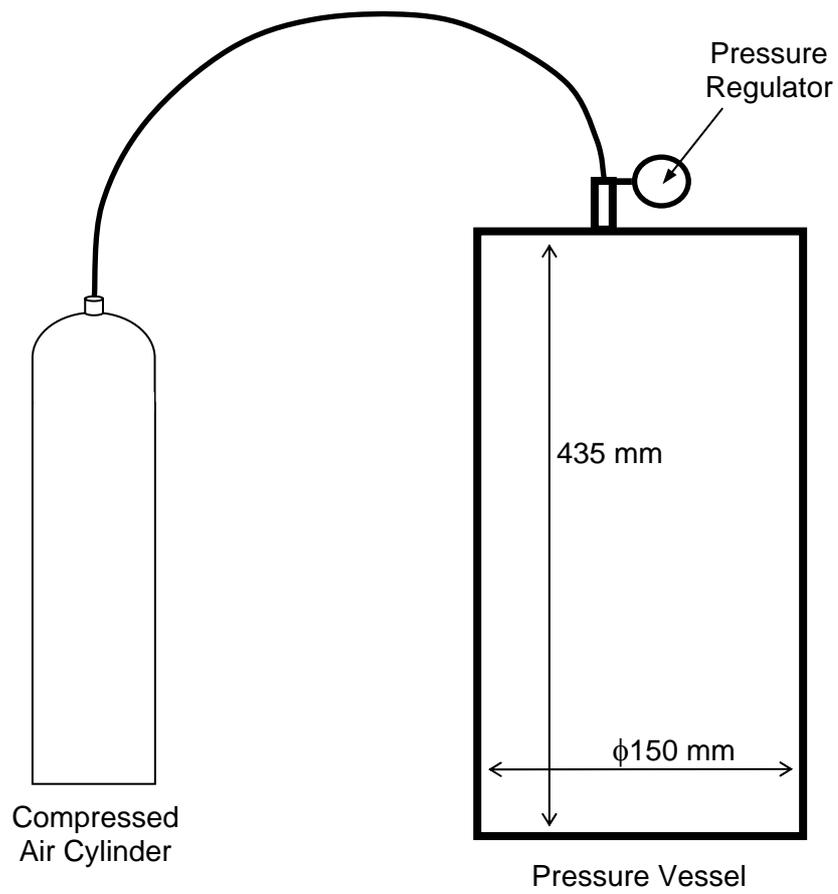


Figure 9/9: (08/04) Schematic and Dimensions of Typical Pressure Vessel

Note: All dimensions ± 2 mm

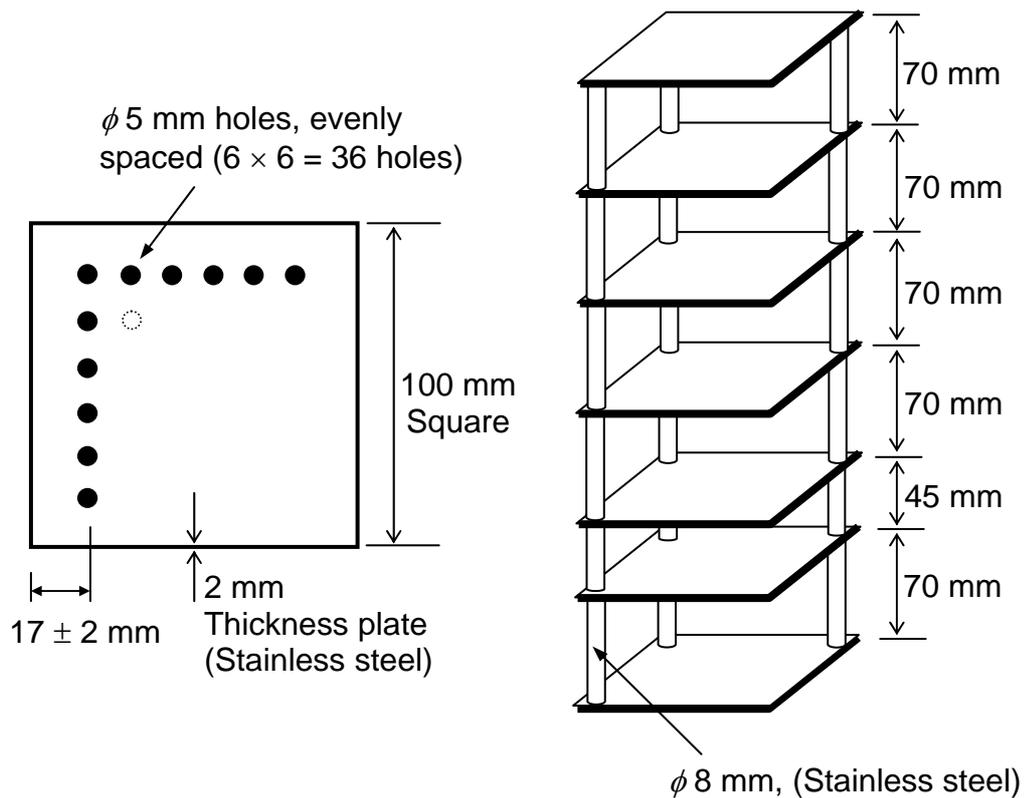


Figure 9/10: (08/04) Schematic and Dimensions of Typical Specimen Tray

Note: All dimensions ± 0.5 mm except where indicated otherwise.

Procedure

7 The maximum density of the bituminous mixture under test shall be determined as described in BS EN 12697-5.

8 The dry bulk density of each compacted specimen shall be determined as described in BS EN 12697-6, Procedure C; self-adhesive aluminium foil shall be used to seal the specimen during immersion. Remove the foil after the test.

9 The air voids of each specimen shall be calculated as described in sub-Clause 953.22 below. Any specimen whose calculated air voids are outside the range $8 \pm 2\%$ shall be rejected and replaced with further samples of suitable void content.

10 The unconditioned (initial) stiffness modulus shall be determined at $20 \pm 0.5^\circ\text{C}$ in the NAT. Designate this as ITSM_U .

11 Determine the dry mass of each specimen using the balance and designate this as M_d .

12 The specimens shall be partially saturated by placing them in the vacuum desiccator, covering them with distilled water at $20 \pm 1^\circ\text{C}$, sealing the apparatus, and applying a partial vacuum pressure of 68 ± 3 kPa for 30 minutes.

13 Remove each specimen from the vacuum desiccator, remove any water on its surface using the absorbent paper towelling and determine its wet mass. Designate this as M_w . The percent saturation (S) shall be determined as described in sub-Clause 953.23 below. Any specimens whose calculated saturation is $\geq 80\%$ shall be rejected.

14 Partly fill the pressure vessel with water. The water level shall be between the 4th and 5th specimen after loading the samples, as shown in Figure 9/11. Freshly distilled water only shall be used (do not re-use). The vessel and water shall be controlled to the target temperature for at least 2 hours before the conditioning procedure commences.

15 The specimens shall be placed into the vessel, using the specimen tray, the cover closed and the pressure gradually increased from atmospheric pressure to 2.1 MPa over a period of 20 minutes. The conditioning procedure shall then be performed, as described in AASHTO PP1, at 2.1 ± 0.1 MPa pressure and the target temperature of $85 \pm 1^\circ\text{C}$ for 65 ± 1 h.

16 After the test period of 65 hrs has elapsed, adjust the target temperature to 30°C . Leave the vessel for the natural cooling for another 24 hrs (Do not release the pressure yet).

17 When the pressure vessel display temperature has reduced to 30°C (after the additional 24 hrs cooling period), the pressure slowly released from the vessel. The pressure release mechanism shall be adjusted so that the pressure returns to atmospheric pressure over a period of 20 to 30 minutes (linear reduction). The cover shall be opened and all the specimens extracted, on the tray, from the vessel.

18 Each specimen shall be surface dried using the absorbent paper towelling, and its wet mass measured within 3 minutes of removing the specimens from the pressure vessel. Designate this mass as M_{w2} . The saturation after the conditioning procedure (S_a) shall be calculated as described in sub-Clause 953.24 below.

19 The specimens shall be observed for any visual cracks and unusual appearance (i.e. colour of binder or aggregates). Record any comments as appropriate.

20 The specimens shall be placed into the NAT or other suitable temperature controlled conditioning environment at $20 \pm 0.5^\circ\text{C}$ in preparation for ITSM testing, as described in BS DD 213.

21 The stiffness modulus shall be determined as described in BS DD 213 within 8 hrs after releasing the pressure from the vessel. Designate this as $ITSM_C$.

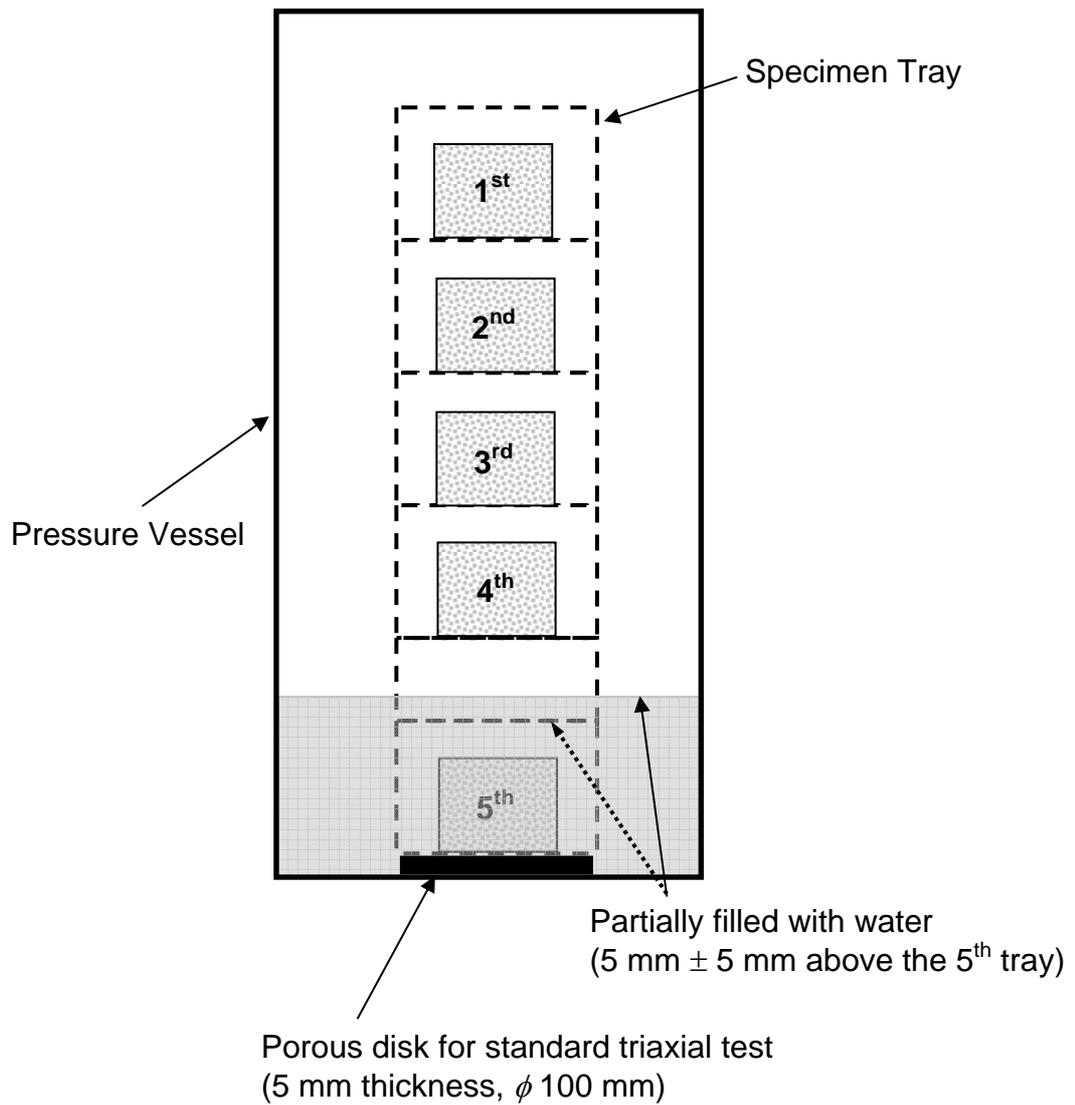


Figure 9/11: (08/04) Schematic Diagram of the Specimen Configuration in the Pressure Vessel

Calculation and Expression of Results

Dry bulk density and calculated air voids

22 Calculate the **dry bulk density** of each specimen (in Mg/m³) as follows:

$$\text{Dry Bulk Density } (G_{mb}) = \frac{A}{(B - C) - \left(\frac{B - A}{D}\right)}$$

where:

A = the mass of the dry specimen in air (in g)

B = the mass of the coated specimen in air (in g)

C = the mass of the coated specimen in water (in g)

D = the density* of the self-adhesive aluminium foil in Mg/m³

* The material described in sub-Clause 953.4 (ii) has been found to have a typical density of 1.650Mg/m³

The specimen dry bulk density shall be expressed to the nearest 0.001Mg/m³

The **air voids** of each specimen (in %) shall be calculated as follows:

$$\text{Air Voids} = \frac{G_{mm} - G_{mb}}{G_{mm}} \times 100$$

where:

G_{mm} is the maximum density determined in accordance with BS EN 12697-5

G_{mb} is the dry bulk density determined in accordance with BS EN 12697-6, Procedure C.

The calculated air voids shall be expressed to the nearest 0.1%.

Saturation before conditioning

23 Calculate the saturation before conditioning as follows:

$$S = \frac{\frac{M_w - M_d}{G_{mb}} - \frac{M_d}{G_{mm}}}{\frac{M_d}{G_{mb}} - \frac{M_d}{G_{mm}}} \times 100$$

where:

S = percent saturation before conditioning

M_d = mass of dry specimen, g

M_w = mass of wet specimen, g
 G_{mb} = dry bulk density and
 G_{mm} = maximum density

The saturation before conditioning shall be expressed to the nearest 1%.

Saturation after conditioning

24 Calculate the saturation after conditioning as follows:

$$S_a = \frac{M_{w2} - M_d}{\frac{M_d}{G_{mb}} - \frac{M_d}{G_{mm}}} \times 100$$

where:

S_a = percent saturation after conditioning

M_d = mass of dry specimen, g

M_{w2} = mass of wet specimen (g) after conditioning

G_{mb} = dry bulk density and

G_{mm} = maximum density

The saturation after conditioning shall be expressed to the nearest 1%.

Stiffness ratio

25 Calculate the stiffness ratio as follows:

$$ITSM_R = \frac{ITSM_C}{ITSM_U}$$

where:

$ITSM_R$ = stiffness ratio,

$ITSM_C$ = conditioned stiffness

$ITSM_U$ = unconditioned stiffness

The stiffness ratio shall be expressed to the nearest 0.01.

Test Report

26 The test report shall contain not less than the following information:

- a) A reference to this test method and test conditions.
- b) Maximum density of the mixture tested, to the nearest 0.001 Mg/m³.
- c) For each specimen tested, report:
 - Dry bulk density (G_{mb}), to the nearest 0.001 Mg/m³.
 - Percent air voids (V_v), to the nearest 0.1%.
 - Percent saturation (S) before conditioning, to the nearest 1%.
 - Percent saturation after conditioning, (S_a), to the nearest 1%.
 - Unconditioned stiffness ($ITSM_U$) before conditioning, to the nearest 100 MPa.
 - Conditioned stiffness ($ITSM_C$) after conditioning, to the nearest 100 MPa.
 - Stiffness ratio ($ITSM_R$), to the nearest 0.01.
 - Any cracks or unusual appearance.
- d) For the mixture tested, the stiffness ratio of the individual specimens tested shall be plotted against the corresponding saturation value after conditioning. The graph shall have linear axes for stiffness ratio (y axis) and saturation after conditioning (x axis). The scales of the graph shall cover a range of stiffness ratio of at least 0 - 1.0 and of saturation after conditioning of 0 - 100%.

27 The test report and graph, uniquely identified, shall be provided to the Overseeing Organisation with the Contractor's Proposal.