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Sub-Task 1: Assessment of Reclaimed Asphalts Containing Aged Polymer Modified Bitumen

Highways England, Mineral Products Association and Eurobitume UK

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Glossary

|G*|: Complex Shear Modulus

AASHTO: American Association of State Highway and Transportation Officials

C=O: Carbonyls

CI: Colloidal Index

DMA: Dynamic Mechanics Analysis

DSR: Dynamic Shear Rheometer

FTIR: Fourier Transform Infrared Spectroscopy

GEL: Gelatinous Structure

HE: Highways England

ITSM: Indirect Tensile Stiffness Test

Jnr: non-recoverable creep compliance

LTA: Long Term Ageing

LVE: Linear-Viscoelastic Region

MCHW: Manual of Contract Documents for Highway Works

MPA: Mineral Products Association

MRTFOT: Modified Rolling Thin Film Oven Test

MSCR: Multiple Stress Creep Recovery

NCHRP: National Cooperative Highway Research Program

PMB: Polymer Modified Bitumen

PSV: Polished Stone Value

RA: Reclaimed Asphalt

RA1: Laboratory aged RA

S=O: Sulfoxides

SARA: Saturates, Aromatics, Resins, and Asphaltenes

SOL: Solution Structure

SP: Softening Point

STA: Short Term Ageing

TTSP: Time-Temperature Superposition Principle

WLF: William, Landel and Ferry Equation

WTT: Wheel Track Testing

Executive Summary

This report presents the work undertaken under Sub-Task 1: Assessment of Potential Durability Impacts of Reclaimed Asphalt (RA) on Asphalt Mixtures Containing Aged Polymer Modified Bitumen (PMB). This work was a collaborative project between Highways England (HE), Mineral Products Association (MPA) and Eurobitume UK. The overall objective of this project was to improve the assessment of Reclaimed Asphalt (RA) materials used in surface courses and end of life assessment for mixtures containing RA, specifically RA containing PMB (PMBs within RA materials and mixtures containing RAs which also include virgin PMB).

Methodology:

The methodology developed for this project focused on making use of scientific protocols that provide the basis for the assessment of RA materials containing PMB. The testing methodology provided effective tools to distinguish between the rheological and chemical properties of different feedstocks of RA containing PMBs. A bitumen recovery protocol was developed for mixtures incorporating RA with PMB and is presented in Appendix A. The bitumen recovery process (extracting the bitumens from asphalt mixtures) used for this project was shown to have minimal impact on degrading the crosslinked polymer network of the bitumens. The Modified Rolling Thin Film Oven Test (MRTFOT) in accordance with MCHW Clause 955 provided a feasible protocol for blending the recovered bitumen from RA with virgin bitumens, as well as a means to accelerate ageing.

The laboratory analysis for this work comprised of four main stages as follows:

Stage 1 (Bitumen Testing): three different PMBs provided by different UK suppliers were subjected to short and long-term ageing. The PMBs were characterised in terms of their rheological and chemical properties after short and long-term ageing.

Stage 2 (Bitumen Blending): recovered bitumens from three different RA sources were characterised for their rheological and chemical properties. Following this, a single RA source was selected for the project. Stage 2 of the project focused on blending at 25% and 50% of the recovered bitumen from the selected RA with the virgin PMBs from Stage 1. This enabled an in-depth analysis and understanding of the interaction, mechanical and performance properties when adding virgin PMBs with aged RA bitumen.

Stage 3 (Asphalt Surface Course Mixtures): Following the key lessons from Stages 1 and 2, Stage 3 focused on selecting 1 RA source and 1 PMB source in addition to using various proportions of virgin aggregate materials to produce asphalt mixtures. The asphalt mixtures were produced at 0% RA with 100% virgin aggregate (Control) mixtures, 25% RA with 75% virgin aggregates and 50% RA with 50% virgin aggregates. The mechanical and performance properties of the asphalt mixtures were assessed. The recovered bitumens from the different mixtures were also subjected to rheological and chemical testing to provide an understanding of their interaction and performance.

Stage 4 (Recyclability Assessments): Stage 4 focused on carrying out an end-of-life and recyclability assessment of aged RA subjected to multiple recycling. The asphalt mixtures produced at Stage 3 from 50% RA were further processed to produce laboratory aged RA. The laboratory aged RA and one PMB plus virgin aggregate were used to produce asphalt mixtures at 50% (lab-aged RA). The asphalt mixtures and the recovered bitumens were evaluated for their mechanical, rheological and performance properties.

Guidance for recovered bitumen assessment of asphalt mixtures containing reclaimed PMB was developed.

Key findings:

The key findings from this project show that, following detailed laboratory investigation and analysis, asphalt mixtures produced using 50% RA have comparable mechanical and performance properties to the mixtures containing 100% virgin components (control). The recyclability assessment (Stage 4) indicated that the desired rheological properties of the PMB can be largely maintained after multiple recycling for asphalt mixtures with added highly polymer modified PMB. These findings require validation using full scale asphalt plant and road trials. The findings from this work show the feasibility of improving sustainability of material options by using high levels of RA up to 50% RA. It is strongly recommended that road trial sections are installed (with 0% RA control sections) using 25% and 50% RA for long-term assessment, monitoring and testing. The findings from road trials should provide important additional validation for the current laboratory work, provide a foundation for the development of guidance documents and specifications for the design, testing and use of asphalt mixtures incorporating up to 50% RA with PMB.

1. Introduction

1.1 Overview

This work was a collaborative project between Highways England (HE), Mineral Products Association (MPA) and Eurobitume UK. This project was tasked with undertaking an independent review of processes to improve the assessment of Reclaimed Asphalt (RA) materials. This included the end-of-life assessment for mixtures containing RA. The specific objective was to review the feasibility of using RA containing Polymer Modified Bitumen (PMB) in asphalt mixtures (primarily in surface course). The outputs from this work will enable HE and the Collaborative Project Team to further support Sustainable Development Strategies in delivering both economic and environmental benefits. This work aims to provide an improved understanding of the behaviour of PMBs within RA materials (and mixtures containing RAs which include virgin PMB) prior to developing specifications and methods for design, application and assessment.

To achieve the above aims, the following objectives were identified:

- Identify the ‘residual’ properties of the PMB in RA; taking into consideration the age, polymer content and rheological properties, and RA composition.
- Develop a method for PMB extraction from RA and a protocol for blending extracted bitumen with added new bitumens.
- Assess the physical and chemical properties of the blend, taking into consideration the RA content, type/grade of a new PMB and the long-term ageing potential.
- Evaluate the impact of using a high content of RA to asphalt mixtures, taking into consideration the effect of ageing and moisture-induced damage.
- Assess the effect of multiple recycling on the mechanical properties of asphalt mixtures containing RA including the effect on the rheological properties of the recovered bitumens.

The methodology of this work comprises four main elements to cover the objectives. Figure 1 shows a flow chart illustrating the main elements of the research methodology. The methodology involves an extensive laboratory investigation into PMBs and the recovered RA bitumens in addition to their ‘parent’ asphalt mixtures. The details of the testing methodology are presented in Section 2.

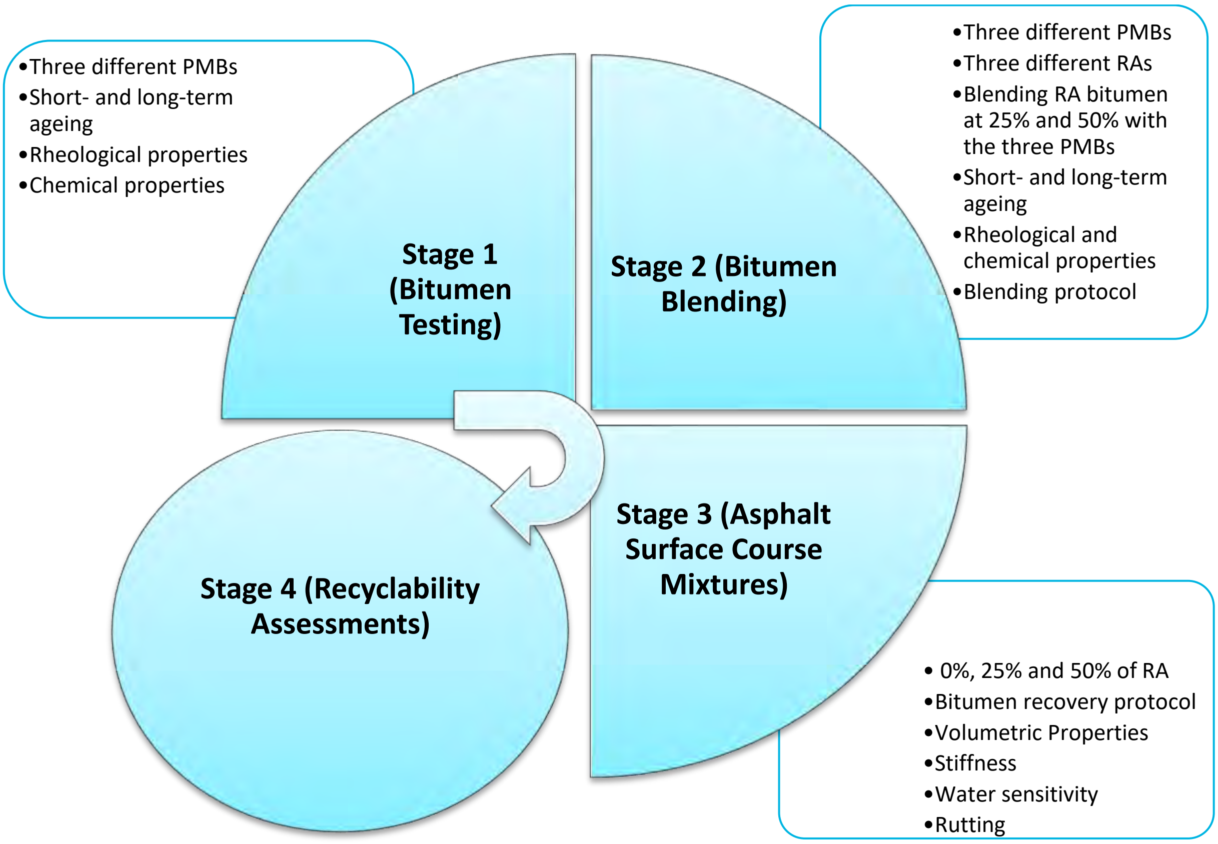


Figure 1. Project methodology

1.2 Background

Increasing the use of Reclaimed Asphalt (RA) in flexible pavements helps minimise the use of virgin aggregates and bitumens and can reduce the carbon footprint associated with the construction of roads. The increased use of RA to be incorporated into flexible pavements supports the 'Construction 2025' report [1] with aspirations of 50% carbon reduction by 2025 and ultimately the United Kingdom's target for net-zero carbon by 2050.

In the UK, Polymer Modified Bitumen (PMB) has been widely used for decades. PMBs have desirable rheological properties that contribute to enhanced resistance to rutting and fatigue cracking [2–4]. Many PMB asphalt surfaces are now approaching an end of first life, potentially offering a valuable source of RA materials containing bitumens with high quality rheological properties. Research has shown that the inclusion of RA with PMB has generally resulted in improved mechanical and performance properties [5,6]. The main concern with replacing a larger volumetric portion of new mixtures with RA is that the performance requirements may be compromised. This is due to the probable inconsistency of the properties of RA that may vary with, for example: recovery techniques, and the maintenance and in-service history of asphalt pavements [5]. Thus, most highway authorities have restrictions to limit the total amount of RA materials used.

Despite these limitations, researchers, highway authorities and the asphalt industry are collaborating to investigate and develop innovative techniques and designs that enable higher amounts of RA to be incorporated without compromising the mechanical and performance properties of the materials and ultimately, the pavement durability [5,7,8]. The use of high RA content in asphalt mixtures has been shown to provide the potential benefits, particularly when carefully controlled and monitored [7–13].

One important issue that needs to be addressed when designing the asphalt mixtures incorporating RA, is the degrees of blending between the aged bitumen coating the aggregate in the RA and the virgin bitumen. There are typically three assumptions considered for the availability of RA bitumen:

(i) a no-blending ‘‘black rock’’- 0% availability;

(ii) the RA bitumen is fully available and active to blend with the virgin bitumen - 100% availability;

(iii) only a portion of the RA bitumen is available to blend with the virgin bitumen - partial availability [14].

Some laboratory based studies indicated that the RA bitumen becomes partially available (to varying degrees) when mixed at elevated temperatures with the virgin aggregates and virgin bitumen [14–16]. On the other hand, a survey conducted by National Cooperative Highway Research Program (NCHRP) Synthesis of Highway Practice 495 [17] reported that 77% of the state highway agencies considered 100% RA bitumen availability; 17% of the state highway agencies considered around 75% of the RA bitumen to be available (partial availability); while only 6% of the respondents of the state highway agencies considered 0% RA bitumen availability [14,17].

Asphalt mixtures designed based on the assumption of 100% availability could lead to dry mixtures with less bitumen available to effectively coat the aggregates contributing to high air void content. These mixtures would be more susceptible to cracking, ravelling and moisture damage. Asphalt mixtures with the assumption of 0% RA bitumen availability could however result in excessive bitumen content, leading to bitumen bleeding and softer asphalt that is susceptible to rutting. Research studies have shown that when less than 30% of RA is mixed at elevated temperatures, the RA bitumen availability approaches 100%; while the assumption of partial availability becomes a greater consideration when more than 30% RA is used [18]. The former is also consistent with some UK practices for incorporating RA in asphalt surface course (such as DMRB HD 31/94 [19] and Road Note 43 [28]), which considers 100% RA bitumen availability when the RA is considered having residual penetration of not less than 15 dmm and added at not more than 10% of the mixture. It is noted that HD 31/94 has been withdrawn with no replacement clause on the use of RA. Nonetheless, the current industry practice continues considering 100% RA bitumen availability even when designing mixtures with high RA contents.

The range and complexity of considerations potentially necessary to be considered when making design assumptions for bitumen availability. This means that the design process and outcome needs to reflect the necessary end performance requirements for the asphalt mixture.

In this project, RA bitumen availability was assumed as 90% during the design of the asphalt mixtures incorporating RA. The next section will cover the materials and experimental programme used in this project.

2. Materials and Experimental Programme

2.1 Materials

2.1.1 Virgin Materials

Virgin aggregates, fillers and bitumens used in this project were as follows:

Aggregates and Fillers

The virgin aggregate materials used in this project were granite aggregates and limestone filler.

Polymer Modified Bitumens

In this project, four different polymer modified bitumens (PMBs) were provided by three different bitumen suppliers. The supplier names and product details were anonymised for this project. PMB codes and classifications are reported in Table 1. PMBs ‘A’, ‘B’ and ‘C’ were selected for testing to cover a wide range of properties.

Table 1. Classification of PMB

Supplier	Sample Code	EN 14023 Classification	Proposed Selection for Testing
1	A	65/105-50	✓
2	B	75/130-75	✓
3	C	65/105-75	✓
	D	65/105-70	✗

2.1.2 Site Won and Reclaimed Asphalt

Asphalt planings (site-won asphalt) were collected from M1 J23A to 24 (NB), M42 J9-8 (SB) and A5 WB Dordon Roundabout (RAB) to M42 J10. Information associated with these materials is presented below in Table 2. The composition analysis of the planed materials was assessed in accordance with BS EN 12697 parts 1 and 2; a summary of the results is presented in Figure 2. These asphalt planings were assumed to have contained PMBs. In Stage 2, detailed bitumen testing and analysis were also undertaken on the bitumen recovered from these planings to prove or disprove this assumption.

Table 2. Site Won Asphalt Materials

Site	Location	Material	Bitumen Content
1	M1 J23A to 24 (NB)	Clause 942 surface course planings	5.2 %
2	M42 J9-8 (SB)		5.2 %
3	A5 WB Dordon RAB to M42 J10		4.8 %

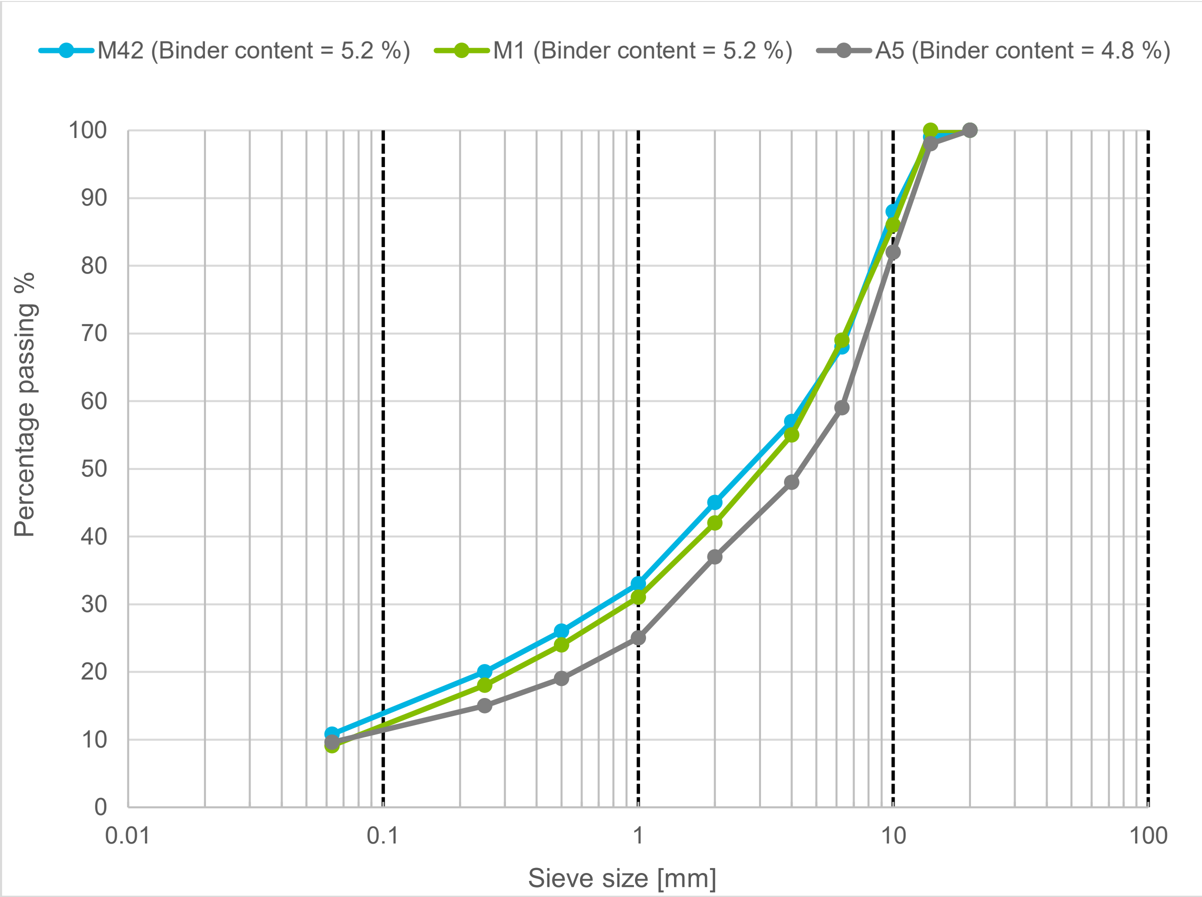


Figure 2. Site-won asphalt Compositional Analysis

The "As planed" asphalt was used as the Reclaimed Asphalt (RA) component without further processing e.g. crushing/screening to individual feedstock size fractions.

2.2 Methodology and Experimental Programme

2.2.1 Methodology

The methodology comprised of four main stages as shown in Figure 3.

Stage 1 includes extensive laboratory bitumen testing. The rheological and chemical properties of the three PMBs, 'A', 'B' and 'C', were evaluated under different ageing conditions, i.e. NA (Non-Aged), STA (Short Term Aged) and LTA (Long Term Aged). The Modified Rolling Thin Film Oven Test (MRTFOT) in accordance with MCHW Clause 955, was used to age the PMBs. The PMBs with different ageing conditions were characterised using empirical tests for penetration and softening point; rheological properties using Dynamic Mechanical Analysis (DMA) and Multiple Stress Creep Recovery (MSCR) tests using the Dynamic Shear Rheometer (DSR); chemical properties using Fourier Transform Infrared Spectroscopy (FTIR) and derivation of the basic chemical components of bitumen, i.e. the fractions of Saturates, Aromatics, Resins, and Asphaltenes (SARA).

Stage 2 used the bitumens recovered from three different site won asphalt materials and the PMBs from Stage 1. The different asphalts were typical Clause 942 surface course planings. The recovered bitumens from the asphalt materials were characterised for their physical and chemical properties using the same suite of bitumen testing as in Stage 1. The recovered bitumen from the M42 RA was selected to be blended at 25% and 50% with the three PMBs using the MRTFOT under short- and long-term ageing. In this stage, the virgin PMBs and the recovered bitumens from the RAs are directly blended in the MRTFOT cans, i.e. for 50% blending about 9.5g of the virgin PMB plus 9.5g of recovered bitumen from the RA are added in a MRTFOT sample can and blended. The same suite of bitumen testing as in Stage 1 was also used to characterise the blended bitumens.

Stage 3 involves asphalt mixture production using the M42 RA and PMB ‘B’ bitumen plus virgin aggregate. The “As planed” asphalt was used as the Reclaimed Asphalt (RA) component without further processing e.g. crushing/screening to individual feedstock size fractions. Therefore, the target grading of the asphalt mixture is achieved by adding specific aggregate size fraction contents. The asphalt mixtures were produced at 0% M42 RA (Control) mixtures, 25% M42 RA and 50% M42 RA. The laboratory manufactured asphalt mixtures were evaluated using the Indirect Tensile Stiffness Test (ITSM), Water Sensitivity, Wheel Track Testing (WTT) in addition to compositional analysis and air voids. The recovered bitumens for the different mixes were also subjected to the same suite of bitumen testing as in Stage 1.

Stage 4 made use of the asphalt mixtures produced at Stage 3 from 50% M42 RA. The asphalt mixtures were produced using 50% M42 RA and were further processed (crushed and long-term aged in an oven at 85°C and for 120 hours) to produce laboratory aged RA (denoted as RA1). The RA1 and one PMB bitumen plus virgin aggregate were used to produce asphalt mixtures at 50% RA1. The asphalt mixtures and the recovered bitumens (from the RA1 and the 50% RA1) were evaluated using the same suite of testing as in Stage 3 and Stage 1 (apart from the FTIR and SARA).

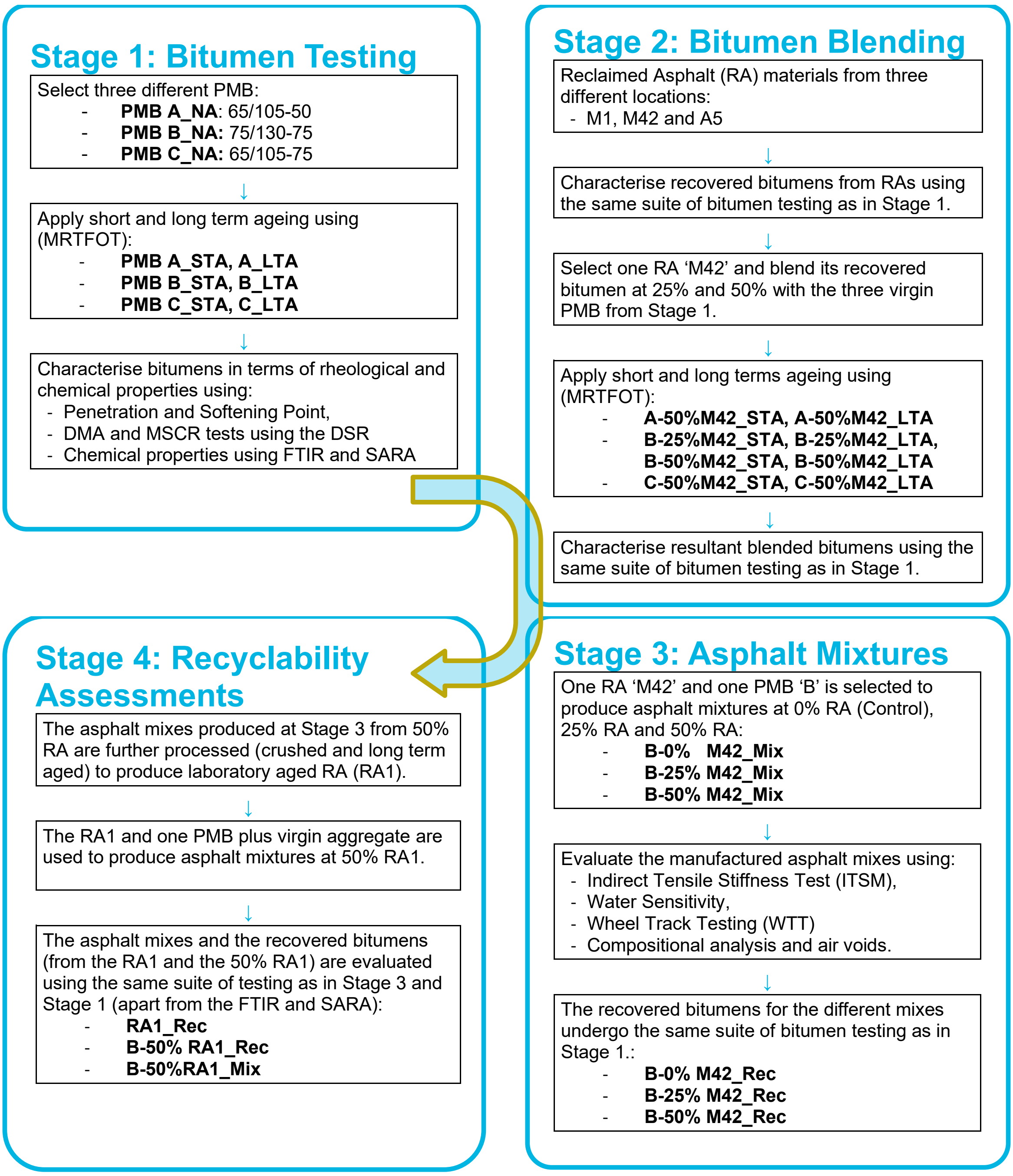


Figure 3. The main elements of the methodology and experimental design

The key components of the project including the main tests developed for this project are summarised below:

2.2.2 Modified Rolling Thin Film Oven Test (MRTFOT)

The test protocol to simulate the ageing of a PMB as detailed in MCHW Clause 955 has been adopted in this study. This protocol was also used for the blending of virgin PMB and recovered RA bitumen. For short-term ageing (STA), the samples contained in the modified cans, shown in Figure 4, were conditioned in the oven for 45 mins ± 1 min at a temperature of 163 ± 1°C.

For long term ageing (LTA), the samples were conditioned firstly for 60 mins ± 1 min at a temperature of 163 ± 1°C. This was followed by 6 hours ± 10 minutes at 135 ± 1°C in addition to the time for the MRTFOT to reach 135°C.

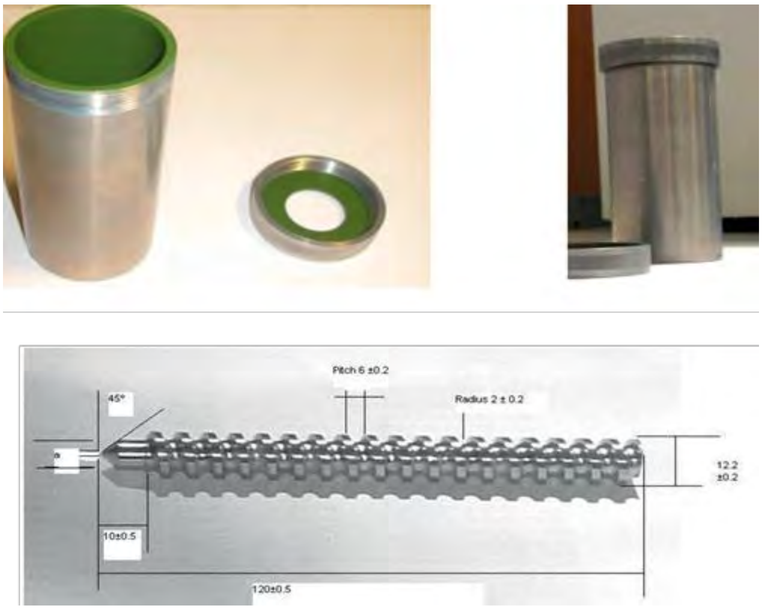


Figure 4. Test Apparatus for the MRTFOT.

2.2.3 Bitumen Recovery from Reclaimed Asphalt

The protocol used for bitumen recovery of the RA was carried out in accordance with BS EN 12697-1 and BS EN 12697-3. The bitumen extraction protocol was modified. The modified protocol makes use of Dichloromethane extraction followed by centrifugation to remove any suspended solids. This is then followed by rotary evaporation to remove the solvents. In this work, a modification has been added, i.e. washing the aggregate with extra solvent and repeating that for 3 to 4 cycles until no discolouration of the solvent is visible, to make sure that the bitumen is fully removed. Details of the modified bitumen extraction protocol are presented in Appendix A.

2.2.4 Penetration and Softening Point

The penetration and softening point for the virgin PMBs, blended bitumens and recovered bitumens were evaluated in accordance with BS EN 1426:2015 (BS 2000-49:2015) and BS EN 1427:2015 (BS 2000-58:2015), respectively. The virgin PMBs and the blended bitumens were tested under different ageing conditions; as-received (NA), short term ageing (STA) and long-term ageing (LTA).

2.2.5 Dynamic Mechanical Analysis (DMA)

The Dynamic Mechanical Analysis (DMA) of the bitumens was undertaken using a Kinexus DSR+ Model Dynamic Shear Rheometer (DSR) supplied by Malvern Instruments Ltd. The viscoelastic properties of the bitumens were evaluated using DMA to define the stress-strain-time-temperature response of the bitumens. Master curves of complex modulus |G*| at a reference temperature of 25 °C were produced for the bitumens using the Time-Temperature Superposition Principle (TTSP) and William, Landel and Ferry (WLF) equation [20].

Black diagrams were also used to show the relationship between stiffness and the viscoelasticity of the materials without the need to apply shift factors to the raw data as required for master curves. They are also useful in distinguishing the thermo-rheological properties of bitumens in one chart.

The bitumen samples were tested under the following settings:

- Oscillatory sweep frequency (0.1, 0.4, 1.0, 1.59, 4.0 and 10.0 Hz)
- Strain control mode within the Linear Viscoelastic (LVE) region (less than 1% strain)
- Parallel plate geometry of 25 mm diameter and 1 mm gap for temperatures (25°C, 35°C, 45°C, 55°C, 60°C, 65°C and 75°C).
- Parallel plate geometry of 8 mm diameter and 2 mm gap for temperatures (35°C, 25°C, 15°C, 5°C and 0 °C)

The Glover-Rowe parameter has been developed as an indication of the behaviour of bitumens at low temperatures. The parameter is computed from the complex modulus and the phase angle as shown below:

$$G - R = \frac{G^* \, (\cos\delta)^2}{\sin\delta}$$

In this project, the complex modulus (G*) and the phase angle δ were taken at 0°C and 1.59 Hz. Research has shown that this parameter can effectively capture the resistance of bitumens to thermal and traffic-induced stresses [21]. The lower the G-R value the better the bitumen is at resisting damage from thermal and traffic-induced cracking, without developing large stresses and regaining its original shape with minimum dissipated energy [21].

The Multiple Stress Creep Recovery (MSCR) test was conducted by applying repeated creep-recovery cycles with 1-second applied to creep shear stress followed by a 9-seconds recovery period in accordance with AASHTO TP 70-13. The main parameters retrieved from MSCR are the non-recoverable creep compliance (Jnr) and the % Recovery. Bitumens that have low Jnr values and high recoveries are less susceptible to rutting at high pavement temperatures. The following test parameters were used for the MSCR test:

- An isothermal temperature of 60˚C.
- Two stress levels (0.1 and 3.2 KPa).
- no rest periods when changing stress level.
- Application of 10 cycles at each stress level.
- Parallel plate geometry of 25 mm diameter and 1 mm gap.

2.2.6 Fourier Transform Infrared Spectroscopy (FTIR)

FTIR spectroscopy was used to evaluate the functional characteristics of bitumens under different ageing conditions. The main functional groups of bitumen that are affected by oxidative ageing are Carbonyls C=O (centred around 1700 cm-1) and Sulfoxides S=O (centred around 1030 cm-1) as detailed in [22,23]. Monitoring the formation of the Carbonyls and Sulfoxides can indicate the amount of oxygen intake for the different bitumens due to oxidative ageing. Bitumens that exhibit less oxygen intake during ageing and ultimately less hardening is preferred.

2.2.7 Saturates, Aromatics, Resins, and Asphaltenes (SARA)

Saturates, Aromatics, Resins, and Asphaltenes (SARA) analysis were carried out to monitor the evolution of the bitumen samples, as it relates to ageing. The hardening of bitumens during ageing is generally related to volatilization and oxidation of the light components (mainly aromatics) which convert into more chemically polar molecules or asphaltenes. The Colloidal Index (CI), calculated as the ratio between combined contents of asphaltenes and saturates (flocculent agents) and resins and aromatics (peptizing agents), was used to evaluate the stability of the colloidal structure.

$$Colloidal\ index\ (CI) = \frac{(asphaltenes + saturates)}{(resins + aromatics\ )}$$

The proportion of the peptising agents to asphaltenes determines whether the bitumens have sol-type behaviour or gel-type behaviour. If there are adequate peptising agents a solution (SOL) structure bitumen occurs, but a reduction forms a gelatinous (GEL) structure in the bitumen. Bitumens having a CI below 0.7 exhibits typical sol-type behaviour. Bitumens with a CI >1.2 exhibits a gel-type behaviour and bitumens with a CI between 0.7 to 1.2 are considered a sol/gel type structure. Bitumens with sol-type behaviour are considered better able to resist thermal and traffic-induced stresses [24].

2.2.8 Asphalt Mixture Testing

Stone Mastic Asphalt (SMA) is one of the primary asphalt mixture types that is used as a surface course in the strategic road network (SRN). There are many variants of SMA mixture design, and a generic SMA incorporating 10mm nominal aggregate size (SMA 10) following British Standard BS EN 13108-5 and PD 6691:2015+A1:2016 was selected for this study. The target bitumen content was specified as 6.2% by mass of the total mixture. For asphalt mixtures incorporating RA, the active bitumen availability from the RA was assumed as 90%, based on the properties of the recovered bitumen from RA 'M42' (the PGH of RA 'M42' was <75 °C, G*/sinδ values of RA 'M42' recovered bitumen were <1 kPa and <2.2 kPa at 75 °C for non-aged and STA, respectively). This assumption aligns with previous literature, as shown in Figure 5 [14,17]. Three different asphalt mixtures were produced using different RA content of 'M42', i.e. 0% RA, 25% RA and 50% RA with the PMB 'B'. The asphalt mixtures design and composition of each mixture are illustrated in Table 3.

Table 3. Asphalt mixtures design details

Composition	0% RA	25% RA	50% RA
Virgin aggregate content	100%	75%	50%
RA 'M42' content	-	25%	50%
Virgin PMB 'B' content	6.20%	5.05%	3.86%
Assumed available bitumen from RA [Note 1]	-	1.15%	2.34%
Target air voids	4%	4%	4%

Note 1: the available bitumen from the RA 'M42' was subtracted from the design bitumen content (6.2%) as follows:

- Assumed bitumen availability from RA = 90%; RA 'M42' bitumen content = 5.2%
- For 25% RA: the available bitumen from RA 'M42' = 90% x 5.2% x 25% = 1.15%
- For 50% RA: the available bitumen from RA 'M42' = 90% x 5.2% x 50% = 2.34%

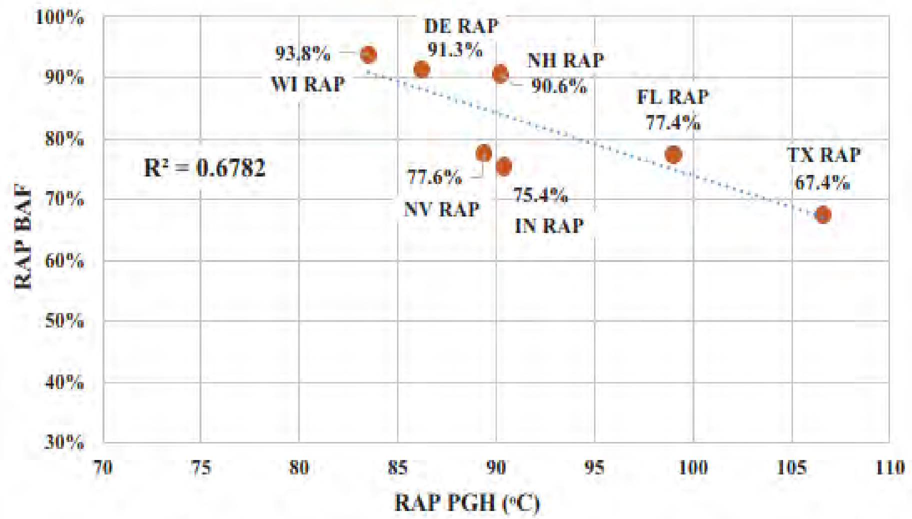


Figure 5. The active bitumen availability of RA (RAP BAF) versus the high temperature performance grade (PGH) [14]

The following protocol was used to manufacture asphalt mixtures incorporating RA:

- The virgin aggregate was preheated for more than 4 hours between 180°C and 200°C.
- The virgin bitumen was also preheated at the recommended mixing temperature of 190°C for 3 hours. The bitumen was stirred thoroughly before mixing.
- The M42 RA was first added to the mixer (preheated at 100 °C) and dry mixed for 5 minutes. The mixer temperature was then raised to 170 °C before adding the preheated virgin aggregate. The RA and the virgin aggregate were mixed for an additional 2 minutes at 170 °C.
- Finally, the preheated virgin bitumen was then added, and the materials were mixed for a further 2 minutes or until the virgin aggregate was completely coated.
- The asphalt materials placed in a slab mould (305 x 400mm) were then compacted at temperatures of (140°C to 150°C) using a laboratory steel roller.

Table 4 presents the summary of asphalt mixture testing carried out in Stage 3 and Stage 4.

Table 4. Asphalt mixtures testing summary

Phase	Asphalt Mixture ID	Description	Asphalt Mixture Tests
Stage 3	0% RA	Asphalt mixture produced using 100% of virgin aggregate and PMB 'B'	▪ Compositional Analysis in accordance with BS EN 12697-1 and 2. ▪ Maximum density in accordance with BS EN 12697-5. ▪ Bulk densities in accordance with BS EN 12697-6. ▪ ITSM in accordance with BS EN 12697-26 ▪ WTT in accordance with BS EN 12697-22 using the small device to 10,000 cycles. ▪ Water Sensitivity Tests in accordance with BS EN 12697-12
	25% RA	Asphalt mixture produced using 25% of RA 'M42' and 75% of virgin aggregate and PMB 'B'	
	50% RA	Asphalt mixture produced using 50% of RA 'M42' and 50% of virgin aggregate and PMB 'B'	
Stage 4	50% RA1	Asphalt mixture produced using 50% of RA1 and 50% of virgin aggregate and PMB 'B'. RA1 is produced by ageing the Stage 3 – 50% RA (i.e. Asphalt mixture produced using 50% of RA 'M42' and 50% of virgin aggregate and PMB 'B') in an oven at 85°C and for 120 hours	

3. Stage 1: Characterisation of the Bitumens and the Effect of Ageing

3.1 Introduction

This section presents the results of testing conducted on three different virgin PMBs, labelled as ‘A’, ‘B’ and ‘C’. This part of the study aims to understand the effects of ageing conditions on the physical and chemical properties of those PMBs. Physical properties included empirical tests for penetration and softening point and fundamental analysis using DMA and MSCR. Chemical properties were evaluated using FTIR and SARA analysis. This section provides a detailed analysis and highlights the key differences between the PMBs. The key findings from this exercise are also summarised and presented at the end of this section.

3.2 Test Results

3.2.1 Penetration and Softening Point

Figure 6 shows the results of the penetration and softening point test results for the three different PMBs and after the different ageing conditions.

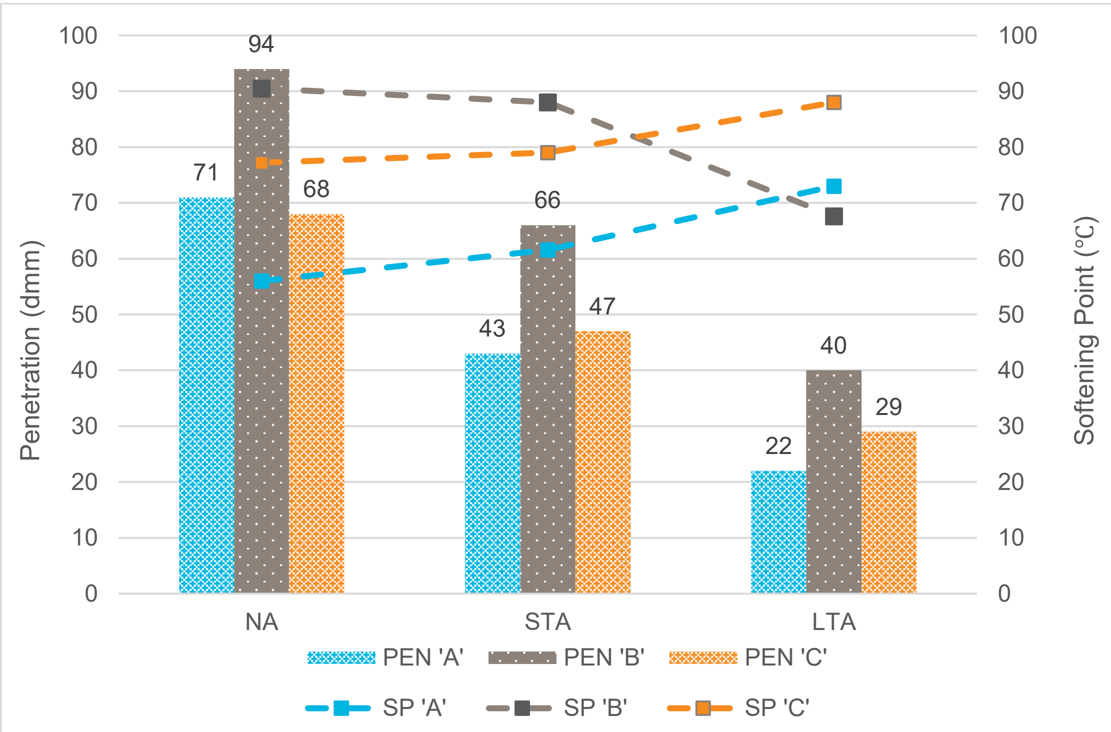


Figure 6. Penetration and softening point results

Figure 6 shows that PMB ‘B’ had higher penetration values following the different ageing conditions. PMB ‘B’ was the softest bitumen in comparison to the other PMBs ‘A’ and ‘C’.

The penetration values for all the PMBs decreased as expected with ageing. This is attributed to hardening due to ageing. The softening point increased with ageing for PMBs ‘A and C’.

The softening point decreased with ageing for PMB ‘B’. PMB ‘B’ is a highly polymer modified bitumen (as indicated by its higher softening point with a relatively higher penetration value). The polymer for PMB ‘B’ could have been degraded into lower molecular weight due to the ageing conditions leading to a lowering of the softening point.

3.2.2 Dynamic Mechanical Analysis (DMA)

Figure 7 to Figure 9 show the master curves and the black diagrams for the three PMBs under different ageing conditions. Figure 7 shows that the effect of ageing on the G* master curves is associated with an increase in G* and a shift for the black diagrams towards lower phase angles, indicating a higher elastic response. The smooth shape of the black diagram in Figure 7 resembled the general behaviour of unmodified bitumen [25] thus PMB ‘A’ could be characterised as a low polymer modified bitumen.

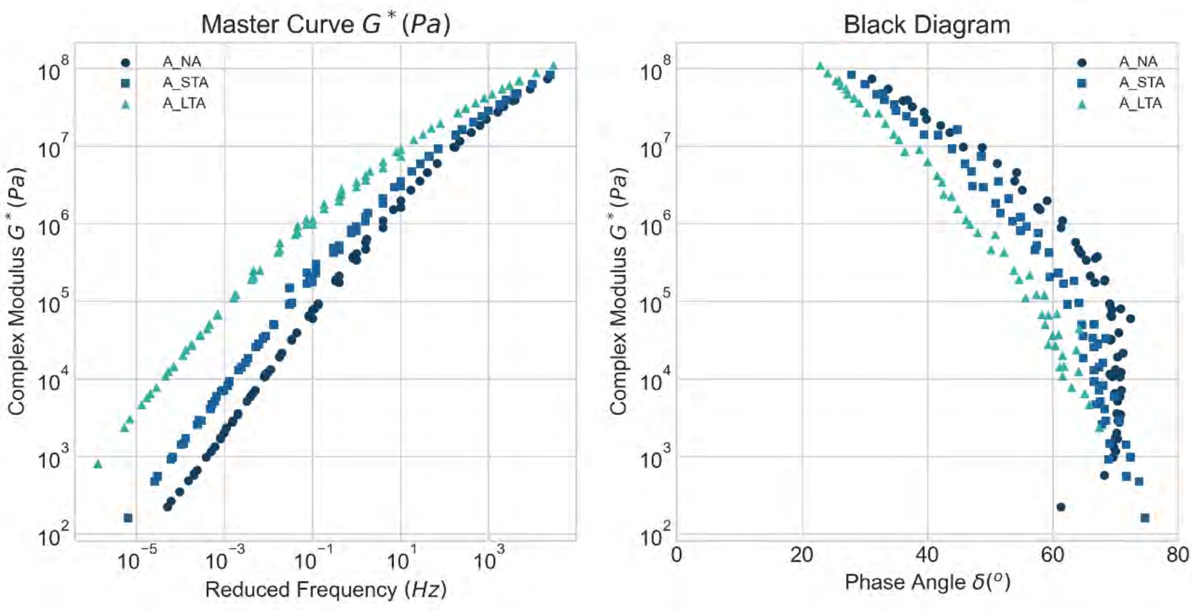


Figure 7. Master Curve at 25 °C reference temperature and the black diagram for the PMB ‘A’ at different ageing conditions

The master curves in Figure 8 shows a similar trend to that found in the PMB ‘A’ except that the relative increase in G* after ageing is significantly reduced over the frequency domain and there is a slight decrease in the G* master curve at low frequencies after STA ageing. The black diagrams, in Figure 8, show a considerable difference from that found in the PMB ‘A’. A typical 3-shape curve is formed for the black diagrams in Figure 8 indicating PMB ‘B’ is a highly polymer modified bitumen [25]. The effect of ageing on the black diagrams is associated with a shift towards lower phase angles for G* values greater than 10^5 Pa but there is a clear shift towards higher phase angles for G* values lower than 10^4 Pa. The increase in higher phase angles for G* values lower than 10^4 can be attributed to the degradation of the polymer network (the polymer dominant areas are at high temperatures and low frequencies) because of ageing conditions. This behaviour of PMB ‘B’ was also evident in the softening point section, as the softening point of PMB ‘B’ decreased with ageing.

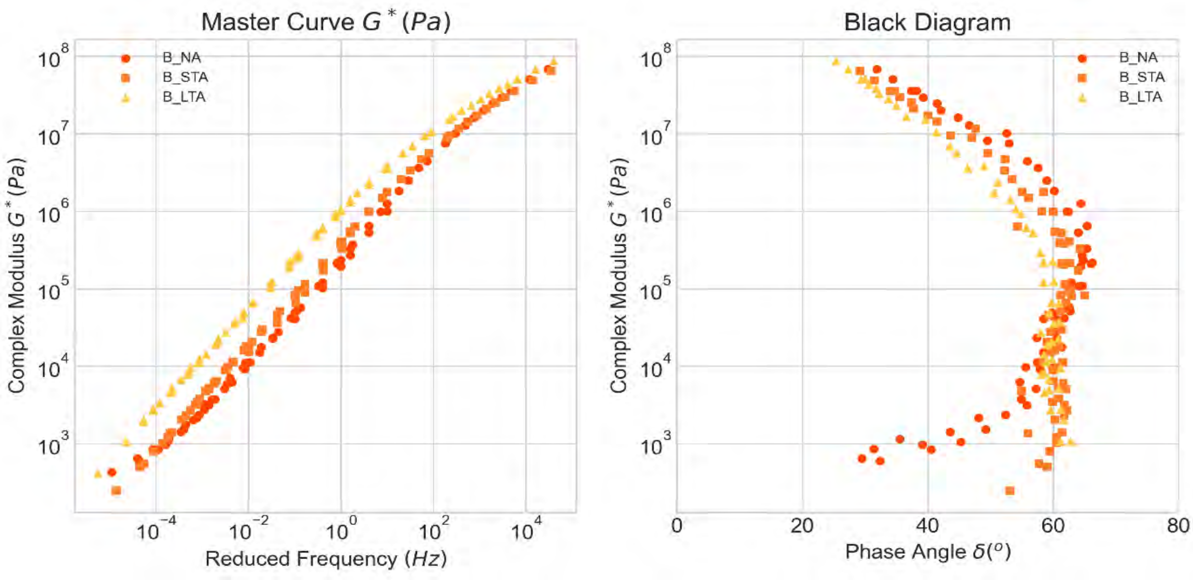


Figure 8. Master Curve at 25 °C reference temperature and a black diagram for PMB 'B' at different ageing conditions

The G* master curves for the PMB 'C' showed a significant increase in the G* after ageing. Unlike PMBs 'A and B', the increase is very clear at high frequencies and low temperatures. The black diagrams in Figure 9 also resemble a typical 3-shape indicating a clear crosslinked polymer structure. The effect of ageing led to a shift for the black diagrams towards lower phase angles for G* values higher than 10^4.

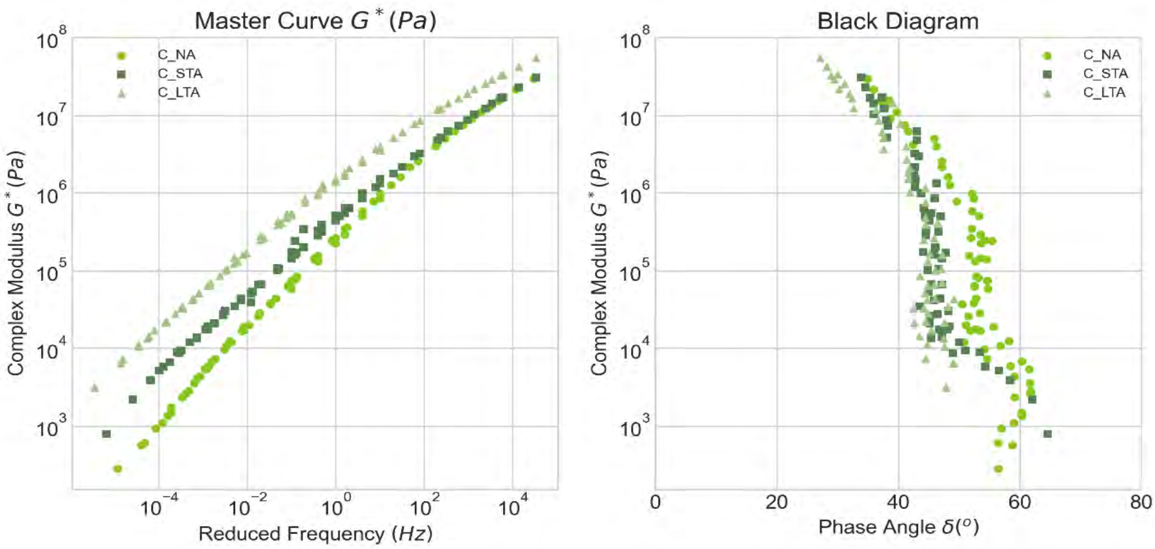


Figure 9. Master Curve at 25 °C reference temperature and a black diagram for PMB 'C' at different ageing conditions

Figure 10 shows the master curves and black diagrams of the unaged PMBs. The master curves for PMBs 'C and B' showed higher G* within the polymer-rich phase (low frequencies and high temperatures) compared to PMB 'A'. The G* master curve for PMB 'C' exhibited desirable thermo-rheological properties, i.e. high G* at low frequencies and/or high temperatures (desirable for rutting resistance); and low G* at high frequencies and/or low temperatures (desirable for fatigue and thermal cracking resistance).

The black diagrams showed a smooth curve for PMB 'A' indicating a lower polymer modification compared to the typical 3-shape for PMBs 'B and C' indicating a high polymer modification.

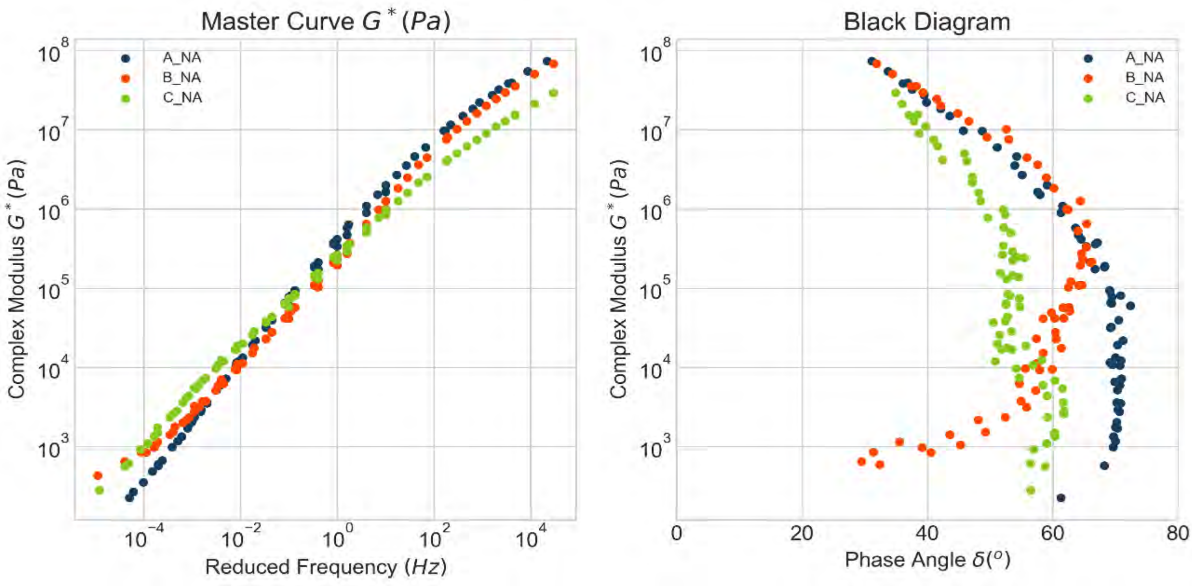


Figure 10. Master Curve at 25 °C reference temperature and a black diagram for all three PMBs without ageing

Figure 11 shows the results of the G-R parameter. The results showed that the G-R parameter increased with ageing indicating that the PMBs would become more susceptible to thermal and traffic-induced cracking with ageing. Figure 11 show that PMB 'C' exhibited the lowest G-R values followed by PMB 'B' and then PMB 'A'. For comparison, the G-R parameters for a 40/60 paving grade bitumen (LTA) were around 300 MPa, a 70/100 paving grade bitumen (LTA) was around 200 MPa, whilst for a 160/220 paving grade bitumen (LTA) was around 50 MPa [26]. This suggests that, after LTA, the low temperature parameter (related to thermal and traffic-induced cracking) of PMBs 'B' and 'C' was found to be closer to that of a 160/220 paving grade bitumen.

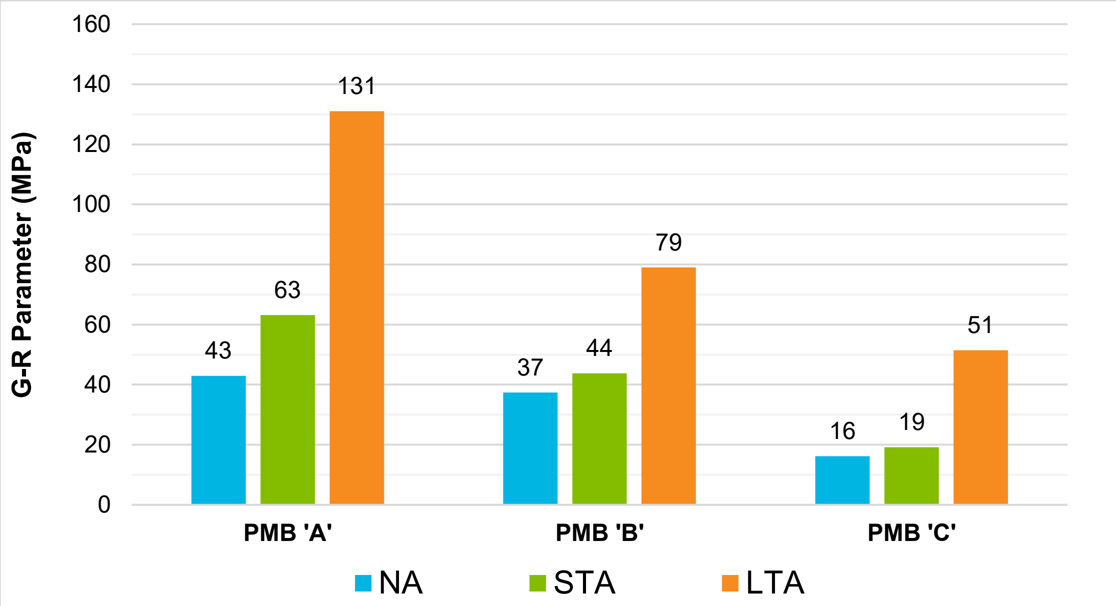


Figure 11. G-R parameter computed at 0 °C and 1.59 Hz for all three PMBs under different ageing conditions

3.2.3 Multiple Stress Creep and Recovery (MSCR)

The previous DMA analyses have dealt with the properties of materials within a linear viscoelastic region. The MSCR test was also conducted to understand the behaviour of PMBs at high stresses that are outside the linear viscoelastic region.

Figure 12 shows the accumulated strains consisting of 10 cycles of creep and recovery over 0.1 kPa and 3.2 kPa stress levels for a total of 200 seconds test duration. The results indicate that PMB ‘A’ (unaged and STA) exhibited the highest accumulated strain in comparison to PMB ‘B’ and ‘C’. PMBs ‘A and C’ followed the anticipated trend with ageing, i.e. increase in stiffness with ageing and, thus, accumulated lower strains. PMB ‘B’ experienced the opposite trend by accumulating higher strains with ageing. This confirmed the behaviour shown in the softening point and DMA section that the crosslinked polymer network could have been degraded due to the ageing conditions.

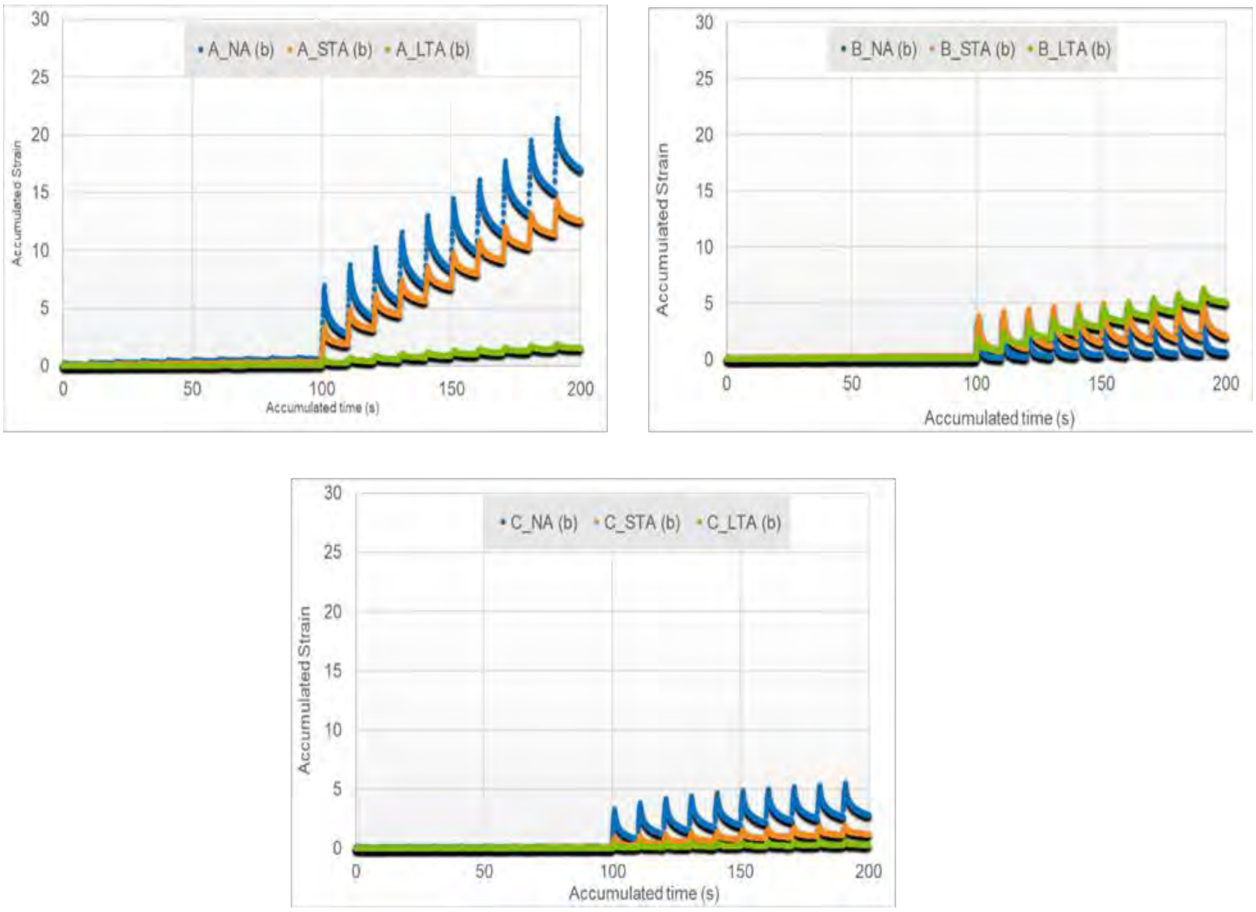


Figure 12. The total accumulated strain of the three PMBs under different ageing conditions

Figure 13 shows the results of non-recoverable compliance (Jnr) (average value for the 10 creep and recovery cycles) over 0.1 kPa and 3.2 kPa stress levels at a test temperature of 60 °C. The range bars represent the maximum and minimum values of the 10 cycles. Figure 13 shows that the PMB ‘A’ has the greatest Jnr values before LTA ageing compared to PMBs ‘B and C’. The effect of LTA ageing has resulted in the hardening of PMB ‘A’. The results in Figure 13 showed an opposite trend for PMB ‘B’ with Jnr values increased with ageing. This also confirmed previous tests and analysis with regards to the behaviour of PMB ‘B’. Partial polymer degradation can, therefore, contribute to compensate for the hardening in the rheological properties due to oxidative ageing, i.e. reduce the hardening susceptibility of PMBs after ageing.

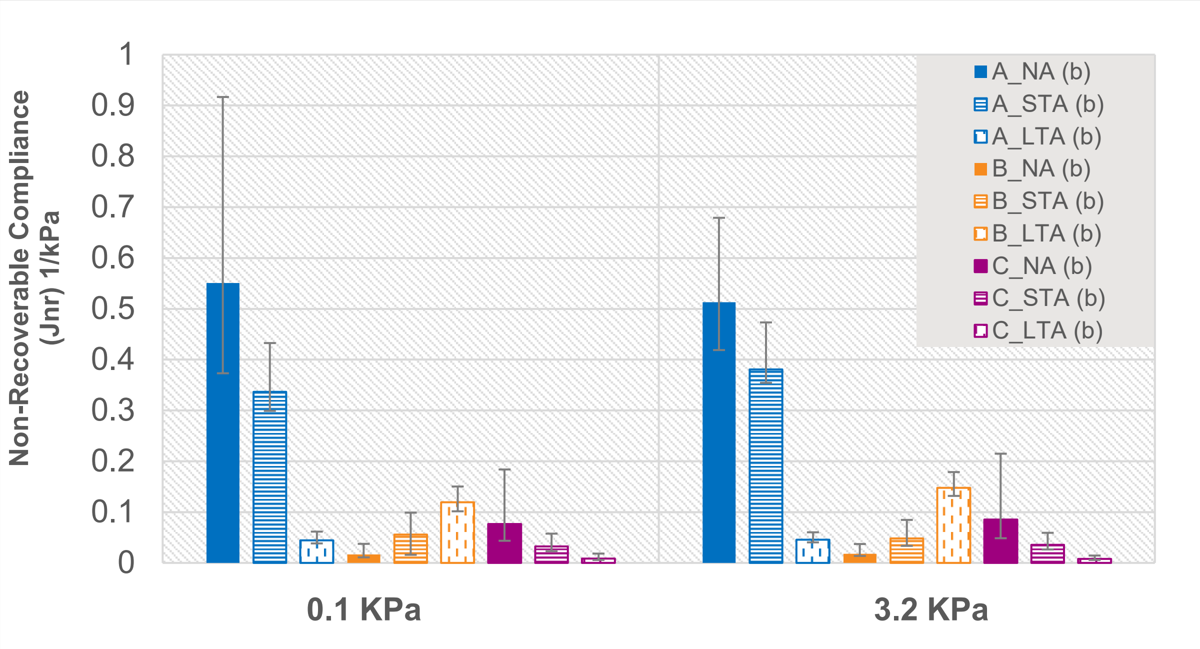


Figure 13. Jnr of the PMBs under different ageing conditions.

Figure 14 shows the average percentage recovery of 10 cycles at stress levels 0.1kPa and 3.2 kPa). The range bars represent the maximum and minimum values of the 10 cycles. PMBs 'B and C' showed higher recoveries than PMB 'A'. The effect of STA and LTA ageing were similar for PMBs 'A and C'. The recoveries decreased after STA and increased after LTA. For PMB 'B', the decrease in recoveries maintained after both STA and LTA. The different trends are attributed to the combined effects of the ageing of the base bitumen and the polymer. For STA and LTA of PMB 'B' and STA for PMB 'A and C', the polymer degradation (softening effect) due to ageing was dominant over the base bitumen (hardening effect); while for the LTA of PMBs 'A and C', the hardening of base bitumen was dominant over the polymer degradation.

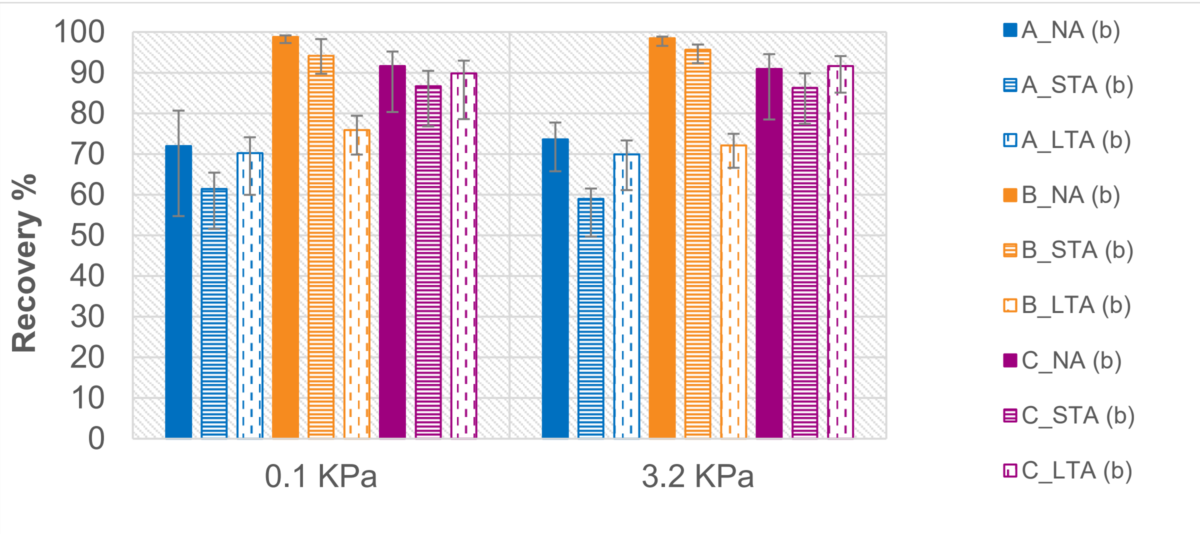


Figure 14. Recovery of the PMBs under different ageing conditions

Figure 15 is used by AASHTO TP 70-13 as an indicator of the presence of an acceptable elastomeric polymer. The average percentage recovery at 3.2 kPa, versus the average non-recoverable creep compliance at 3.2 kPa was plotted on the graph. All the PMBs after the different ageing conditions were above the line indicating an "acceptable" elastomeric polymer behaviour.

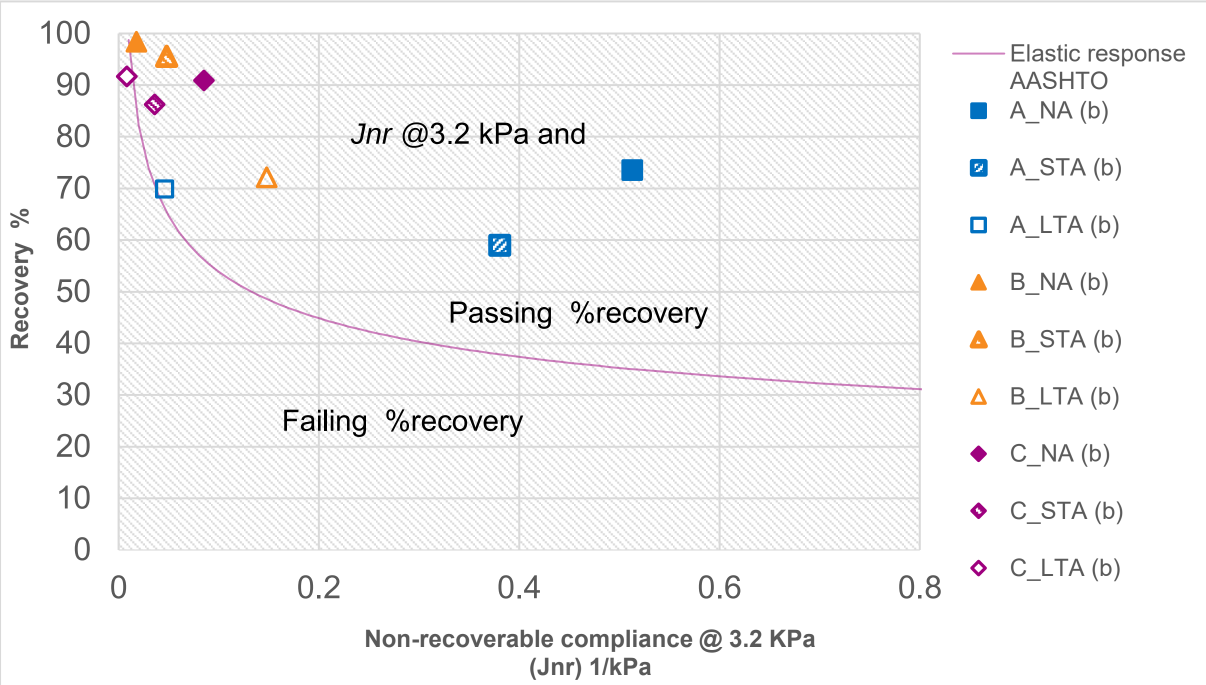


Figure 15. The elastic response (AASHTO TP 70-13)

3.2.4 Fourier Transform Infrared Spectroscopy (FTIR)

Figure 16 shows typical results of FTIR scan spectra (bandwidth vs absorbances) for the PMB ‘B’ under different oxidative conditions. The S=O peaks showed a consistent trend throughout the FTIR testing. The average peaks calculated are shown in Figure 16. These presented a quantitative indication for the effect of ageing. The results in Figure 17 show qualitatively the effect of ageing on the Sulfoxides S=O with peaks visible at approximately 1027 cm^{-1}. Figure 17 shows that the peaks for S=O for all the PMBs, as expected, increased with ageing. There was only one exception, with PMB ‘C’ showing a decrease after STA.

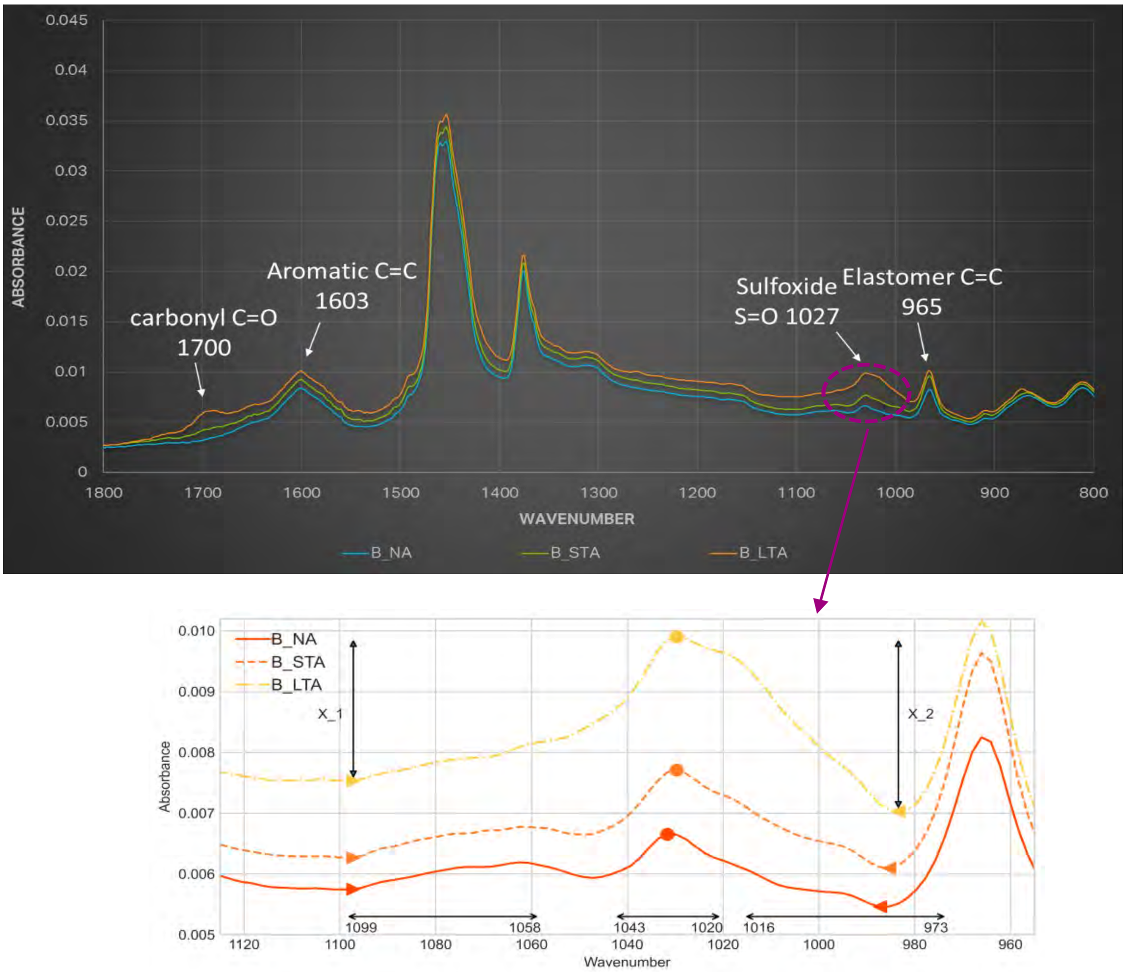


Figure 16. Typical FTIR scan for bitumen PMB 'B' and the procedure for calculating the S=O

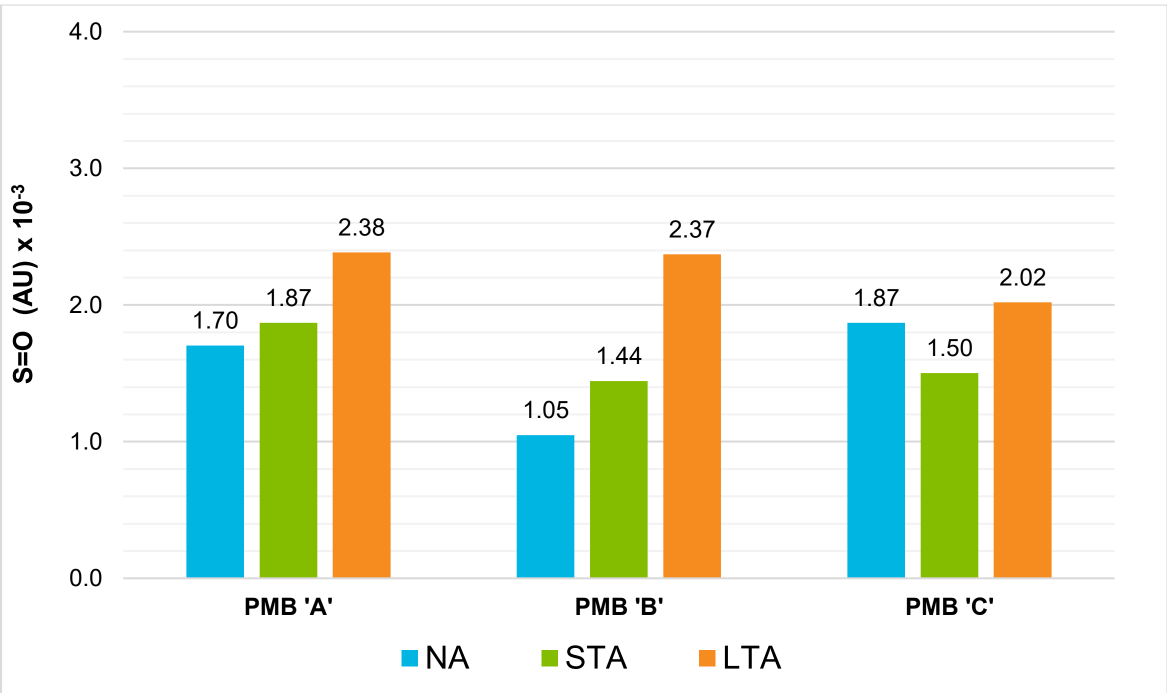


Figure 17. The effect of ageing on sulfoxide.

3.2.5 Saturates, Aromatics, Resins, and Asphaltenes (SARA)

Figure 18 shows the changes of the four SARA fractions with ageing. For PMBs ‘A’ and ‘C’, the trend is consistent for the aromatics and asphaltenes. The aromatics decreased and the asphaltenes increased with ageing.

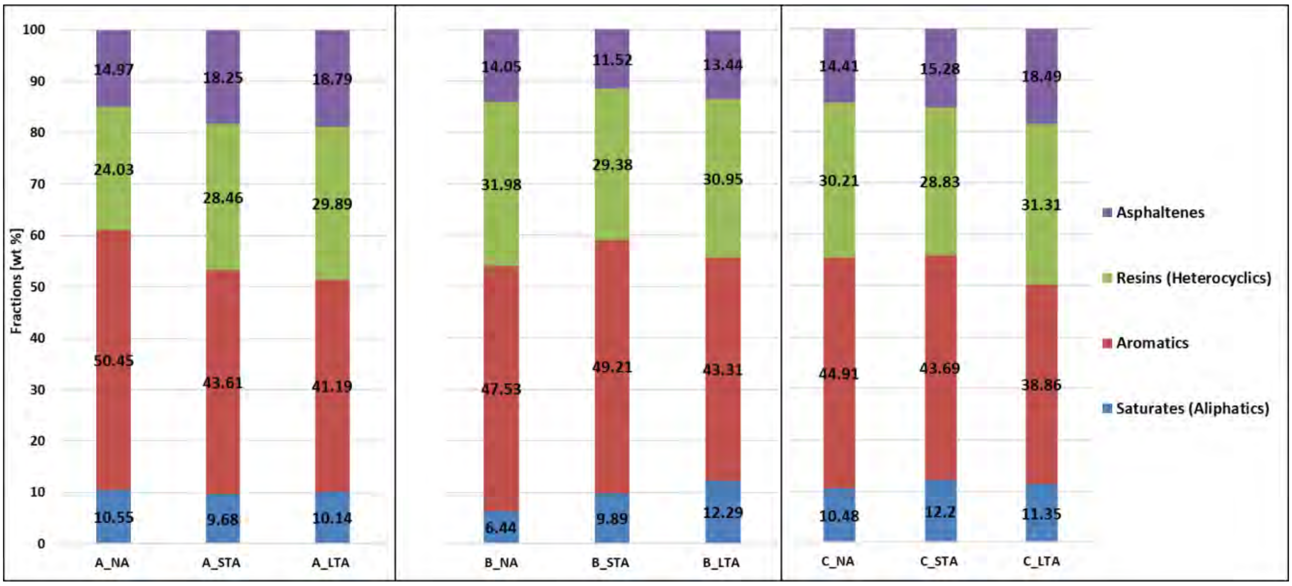


Figure 18. The SARA (saturate, aromatic, resins and asphaltenes) compositions of the three PMBs

Figure 19 shows that all the PMBs exhibited a sol-type behaviour with CI values <0.7. This was evident even after LTA. The PMBs changed towards a more gel-like colloidal structure with ageing. PMB ‘B’ showed the most sol-type behaviour (lowest CI values) in comparison to other bitumens.

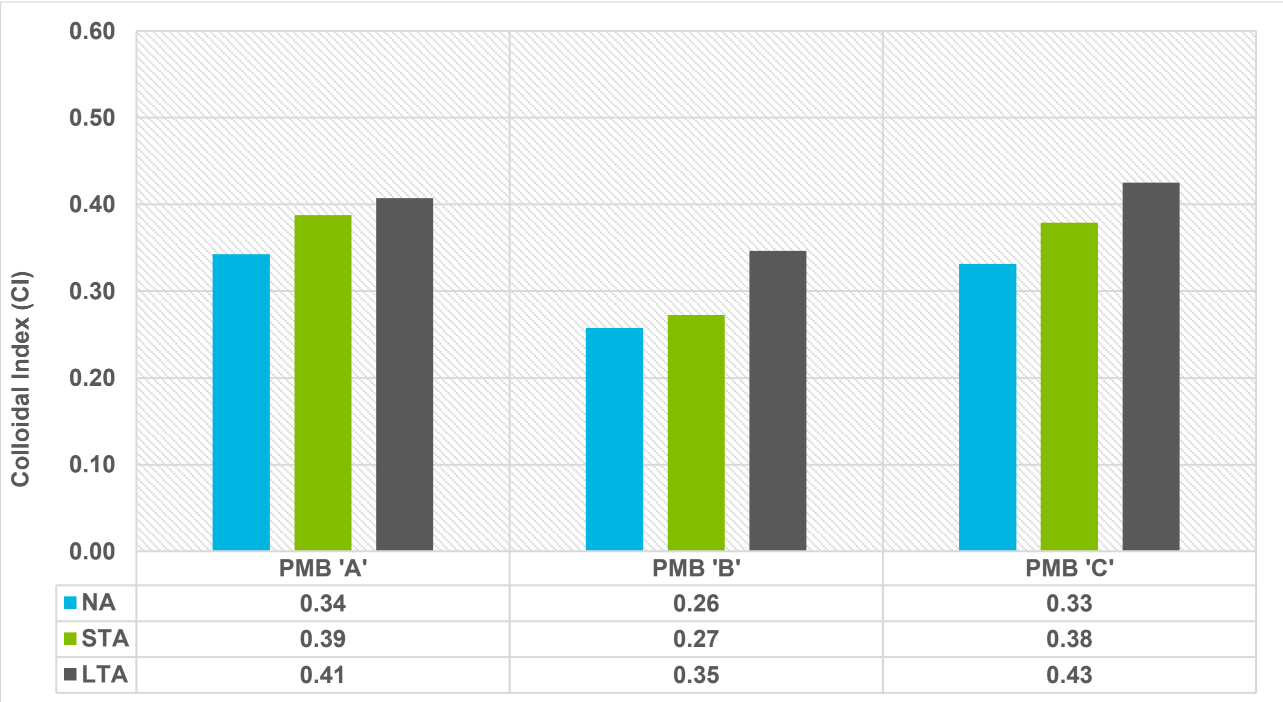


Figure 19. Colloidal index (CI) of the three PMBs under different ageing conditions

3.3 Key Findings

Comprehensive physical and chemical evaluations were carried out on the three selected virgin PMBs. The effect of oxidative ageing using MRTFOT on the properties of the PMBs was assessed. From the results presented in this section, the following summary and conclusions are drawn:

1. The analysis of softening point, DMA and MSCR showed that oxidative ageing can partly disintegrate the polymer network, resulting in softening the PMB. This was clear for PMB ‘B’. The presence of polymer can, therefore, contribute to reducing the hardening susceptibility of PMBs from ageing.

2. The DMA and MSCR analysis provided effective tools to distinguish between the properties of different PMBs. In particular, the black diagram and the relationship between non-recoverable compliance (Jnr) and the recovery from the MSCR found to be sensitive to the level of a crosslinked polymer structure. The DMA and MSCR showed that PMBs ‘B and C’ were highly polymer modified PMBs while PMB ‘A’ was a relatively lower polymer modified PMB. All the PMBs under different ageing conditions maintained an “acceptable” level of elastomeric polymer response in accordance with AASHTO TP 70-13.

3. The G-R parameter derived from the DMA testing at low temperature (0°C) was able to capture the effect of ageing on the cracking resistance of PMBs. The G-R results showed that PMB ‘C’ had the lowest G-R values indicating superior cracking resistance followed by PMB ‘B’ and then PMB ‘A’. These results highlight the importance of characterisation of the PMBs using fundamental testing such as DMA and MSCR. Using only empirical testing, e.g. penetration, indicated that PMB ‘C’ was the hardest (lowest penetration) and consequently would have been perceived as being inferior in cracking properties.

4. The chemical analysis using the FTIR and SARA showed that the PMBs had consistent behaviour with regards to the effect of ageing. The SARA analysis indicated all the PMBs under different ageing conditions exhibited sol-type behaviour with CI values <0.7.

4. Stage 2: Characterisation of RA Blended Bitumens

4.1 Introduction

The MRTFOT was used to directly blend the virgin PMBs (from Stage 1) with bitumens recovered from three different RA sources. Using the MRTFOT enabled mechanical interaction between the virgin and recovered RA bitumens and allowed evaluation of the short-term and long-term ageing of the blends at the same time. The rheological and chemical properties of the RAs are presented in this section. The rheological and chemical properties of the resultant blends (containing 25% or 50% of recovered bitumens from RA) under different ageing condition are also introduced. For the 25% and 50%, about 4.75g and 9.5g of the recovered bitumen from the RA plus 14.25g and 9.5g of the virgin PMB were blended under different ageing conditions in the MRTFOT sample can, respectively. This section provided a detailed analysis and understanding of the level of interaction between virgin PMBs and recovered bitumens from the RAs.

4.2 Test Results

4.2.1 Penetration and Softening Point

Table 5 shows the results of the penetration and softening point tests on the bitumens recovered from the different RA. The penetration and softening point results of the three RA ‘M1, M42 and A5’ showed that the bitumens had penetration > 45 dmm and softening point <54 °C.

Table 5. Penetration and softening point results of the RAs

Testing	M1	M42	A5
Penetration (dmm)	56	64	45
Softening Point (°C)	52.4	51.0	53.4

The composition analysis of the M1 and M42 site-won planings (Figure 2) and the recovered bitumen properties of these RAs (Table 5) appear to be comparable, and only one source of RA was to be considered for the next analysis. The RA from the M42 was, therefore, selected to be blended with the virgin PMBs for additional tests. Table 6 presents the penetration and softening point results for the blends of RA ‘M42’ recovered bitumens with the virgin PMBs under STA and LTA using the MRTFOT. The effect of STA and LTA on the 50% blends (M42 RA with the PMBs) led to a considerable decrease in the penetration values suggesting a hardening of the materials. The M42 RA seemed to be very susceptible to oxidative ageing via MRTFOT resulting in a considerable decrease in the penetration result of the blended bitumens. To illustrate, in Stage 1 the penetration of PMB ‘A’ after STA and LTA were 43 dmm and 22 dmm, respectively. However, the 50% blends for PMB ‘A’ with the recovered bitumen from M42 RA (64 dmm) exhibited a reduction in penetration to 25 dmm and 12 dmm after STA and LTA, respectively.

The softening point increased with ageing for the 50% blends, however, for the 25% blends, softening point decreased with ageing, due to the degradation of polymers (contains 75% of PMB 'B').

Table 6. Penetration and softening point results

Testing	M42								
	100% RA	50% RA/50% PMB						25% RA/75% PMB	
		A _STA	A _LTA	B _STA	B _LTA	C _STA	C _LTA	B _STA	B _LTA
Penetration (dmm)	64	25	12	36	25	27	17	52	33
Softening Point (°C)	51.0	60.8	70.6	61.6	66.0	68.8	74.6	73.6	65.6

4.2.2 Dynamic Mechanical Analysis (DMA)

Figure 20 shows the master curves and the black diagrams for the three bitumens recovered from the supplied RA (M45, M1 and A5). A 40/60 penetration paving grade bitumen was used as a reference bitumen for comparison in the DMA analysis as shown below in Figure 20. The master curves did not show a significant difference between the different RAs and highlighted that they were softer than the 40/60 paving grade bitumen after LTA. The master curves agree with the penetration and softening point results in Table 5 with the RA 'A5' as the hardest followed by 'M1' and then 'M42'. The black diagrams for the RAs are generally similar in shape (smooth shape) to the unmodified bitumens with slightly lower phase angles (more elastic) than the paving grade bitumen 40/60 at high temperatures. These results provide weak evidence of the presence of polymer modification.

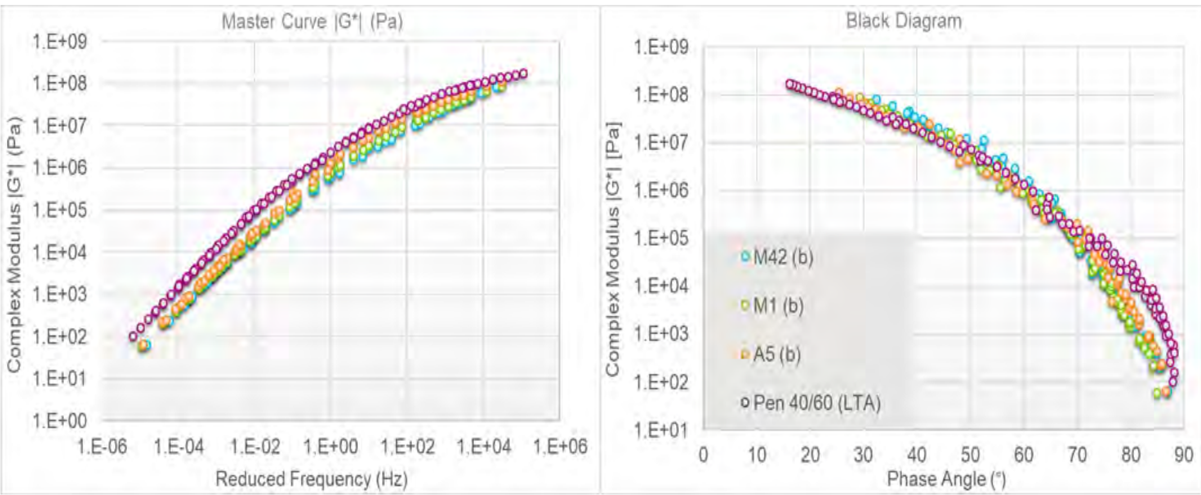


Figure 20. Master Curve at 25 °C and a black diagram for the bitumens recovered from the three RAs

Figure 21 to Figure 23 show the master curves and black diagrams for the RA 'M42' recovered bitumen and its blends with other virgin PMBs using MRTFOT ageing protocols after STA and LTA. The G* master curves, in Figure 21, for the 50/50 M42/A blends (after STA and LTA) exhibited higher values over the frequency domain than the 100% PMB 'A' or 100% RA 'M42' recovered bitumen. The master curves and penetration results suggested that oxidative ageing has a larger effect when the PMBs are blended with the RA.

The black diagrams for the 50/50 blends in Figure 21 fell between the virgin PMBs (more elastic) and the virgin RA (more viscous) performances. The effect of ageing on the blends was similar to the virgin PMBs, i.e. the black diagrams shifted towards lower phase angles with ageing.

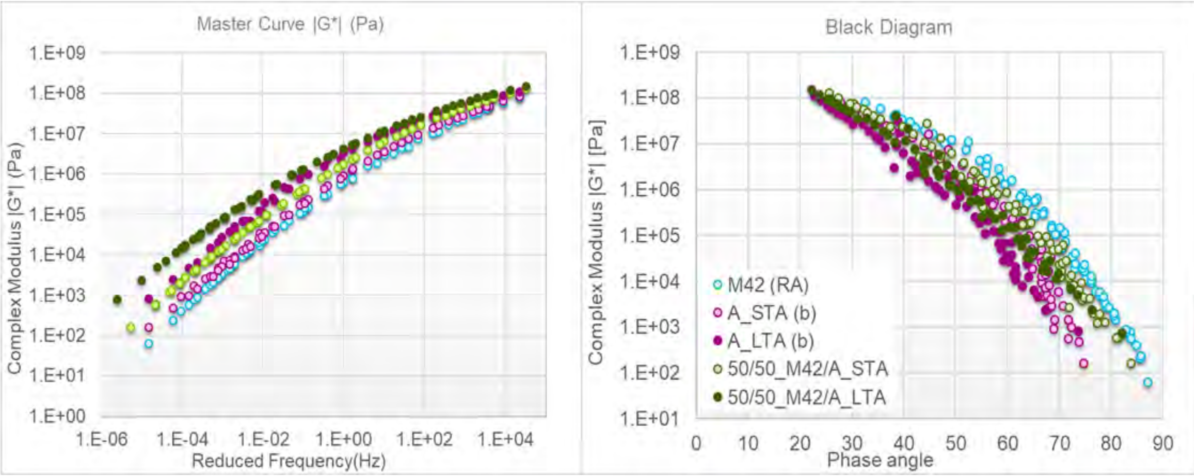


Figure 21. Master Curve at 25 °C and a black diagram for the PMB 'A' and RA 'M42' blends

The G* master curves for the RA 'M42' recovered bitumen, as shown in Figure 22, increased after blending with PMB 'B', particularly at low frequencies and/or high temperatures. The black diagrams for the 50/50 and 25/75 blends, shown in Figure 22, took a polymeric typical 3-shape and fell between the virgin PMB (more elastic) and the RA bitumen (more viscous) performances. Figure 22 shows that there was a slight difference in terms of master curves and black diagrams between the 50/50 and 25/75 blends.

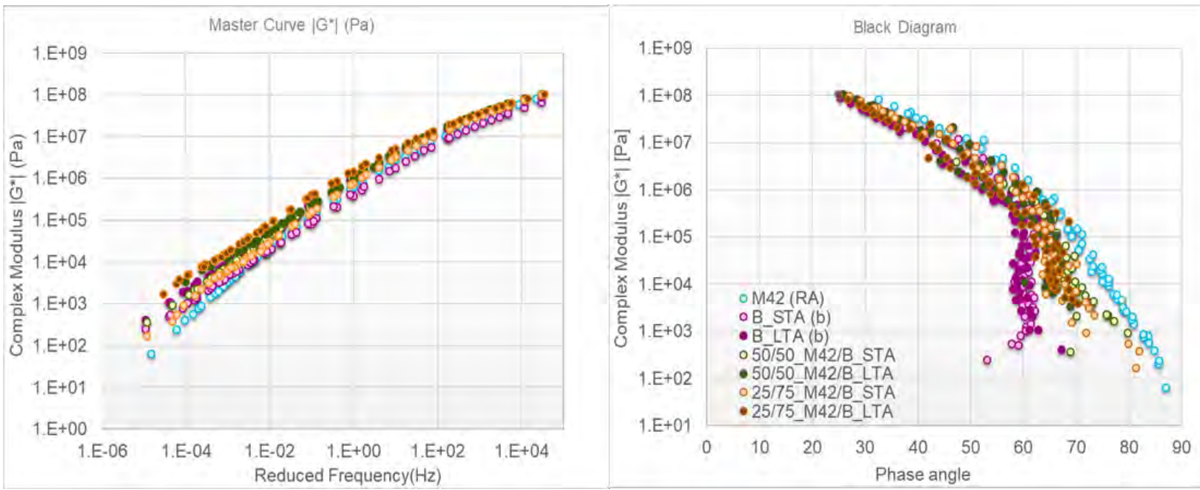


Figure 22. Master Curve at 25 °C and a black diagram for the PMB 'B' and RA 'M42' blends

Figure 23 shows a noticeable increase in the G* values for the 50/50 M42/PMB 'C' compared to the 100% RA M42 and the virgin PMBs over the frequency domain. The black diagrams, in Figure 23, for 50/50 blends resemble more the RA 'M42' (smooth shape) than the PMB 'C' (polymeric 3-shape).

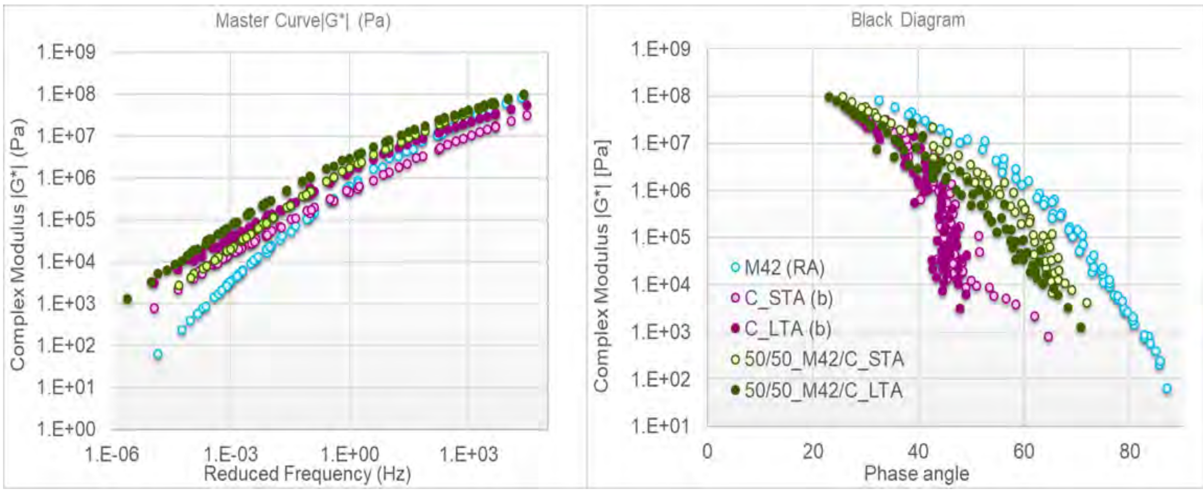


Figure 23. Master Curve at 25 °C and black diagram for the PMB ‘C’ and RA ‘M42’ blends

Figure 24 shows the results for the G-R parameters for the different RAs recovered bitumens and the blends of RA ‘M42’ recovered bitumens with other virgin PMBs. The G-R value of the RA ‘M42’ recovered bitumen was interestingly comparable to the virgin PMBs shown in Figure 11. The results of the RAs agree with the penetration results in Table 5 where the RA ‘M42’ recovered bitumen was the softest followed by ‘M1’ and then ‘A5’. The effect of adding the RA ‘M42’ recovered bitumen combined with the effect of MRTFOT ageing conditions led to a considerable increase in the G-R values indicating the hardening of the materials. The G-R values, as expected, increased with ageing for the PMBs and RA ‘M42’ blends. The G-R values decreased when reducing the percentage of RA for the M42/B blends. The results of the G-R values for the PMBs and RA ‘M42’ blends agree with the G-R results in Figure 11 for the virgin PMBs where bitumen ‘C’ exhibited the lowest G-R values followed by PMB ‘B’ and then PMB ‘A’.

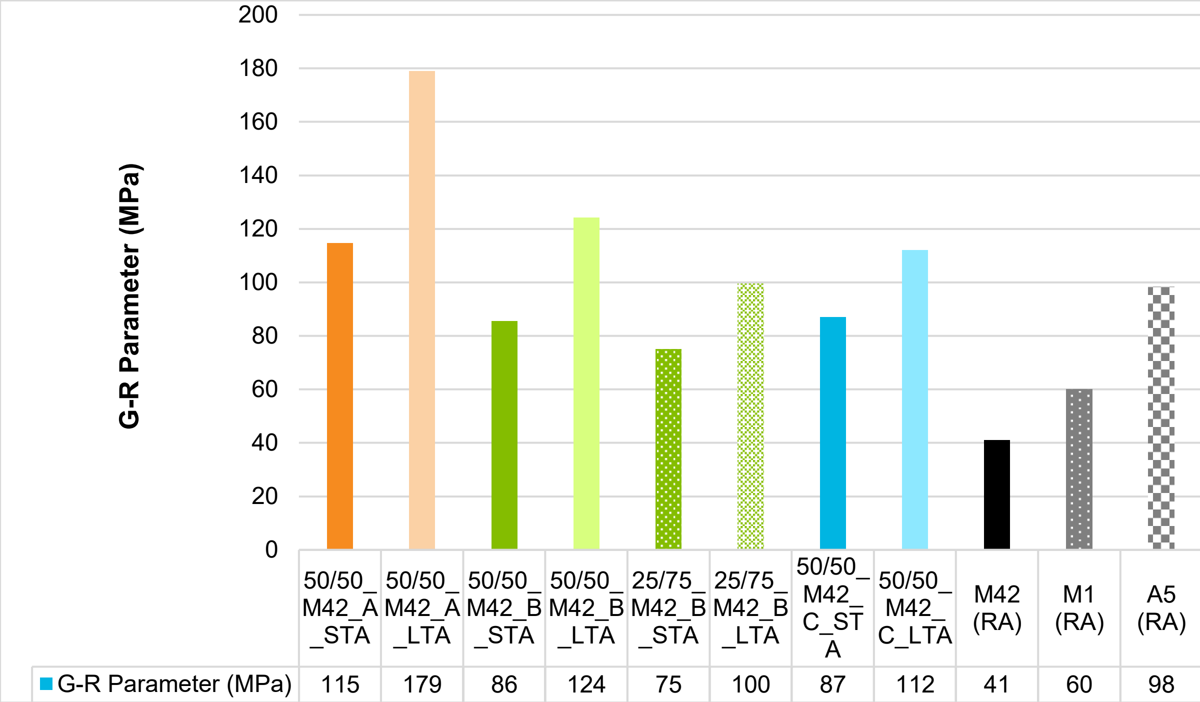


Figure 24. G-R parameter computed at 0 °C and 1.59 Hz

4.2.3 Multiple Stress Creep and Recovery (MSCR)

Figure 25 shows that all the recovered bitumens from RAs 'M42, M1 and A5' fell below the line indicating "insufficient" elastomeric polymer recovery. The recoveries of the RAs were slightly higher than the 40/60 paving grade bitumen, although their Jnr values were higher than the 40/60 paving grade bitumen. These results may be indicative of the presence of very low polymer modification (or the effects of polymer degradation).

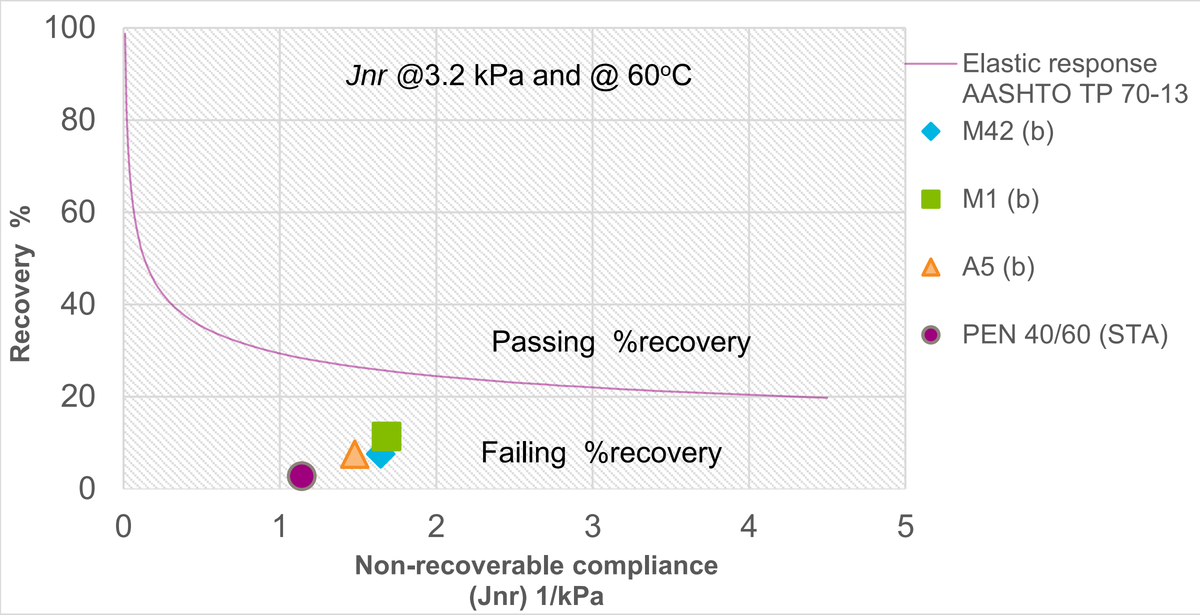


Figure 25. The elastic response of RA bitumens

The results in Figure 26 showed that blending 50% of PMB ‘A’ with the RA ‘M42’ did not provide “acceptable” polymer modification in accordance with AASHTO TP 70-13. The effect of ageing, as expected, increased the recovery percentages and decreased the Jnr values.

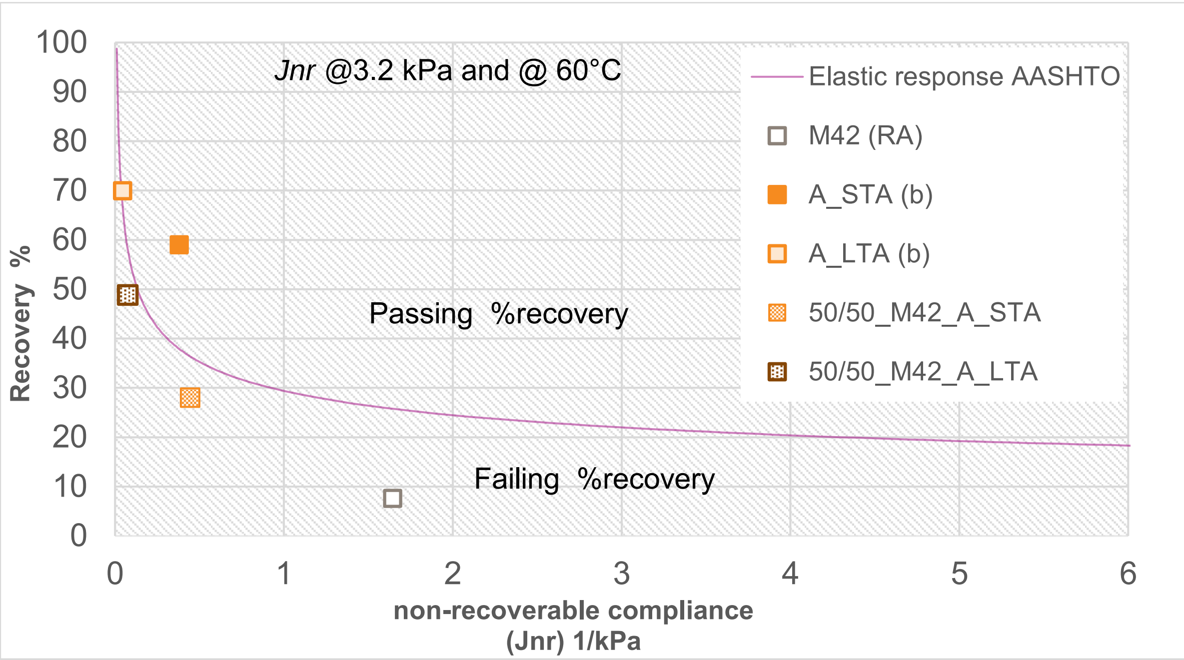


Figure 26. The elastic response for the PMB ‘A’ and RA ‘M42’ blends

The blended bitumens of RA ‘M42’ with PMB ‘B’ at 25% and 50% fell above the line, as shown in Figure 27, indicating an “acceptable” elastomeric polymer behaviour except for the 50/50 M42/B blend after STA. The results of recoveries of the blends increased with ageing, whilst the Jnr values decreased with ageing.

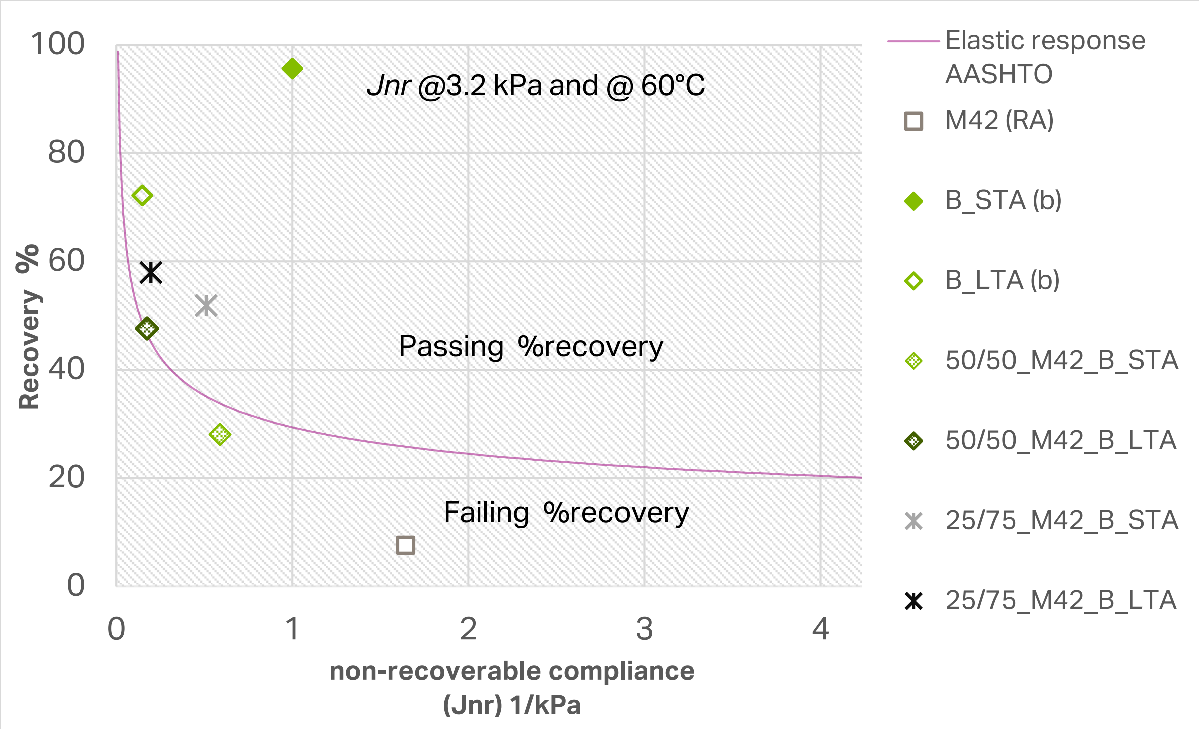


Figure 27. The elastic response for the PMB 'B' and RA 'M42' blends

Figure 28 shows that the blended 50% of PMB 'C' with RA 'M42' was adequate to provide "acceptable" elastomeric polymer in accordance with AASHTO TP 70-13. The effect of ageing on the 50/50 blends increased the recoveries and decreased the Jnr values.

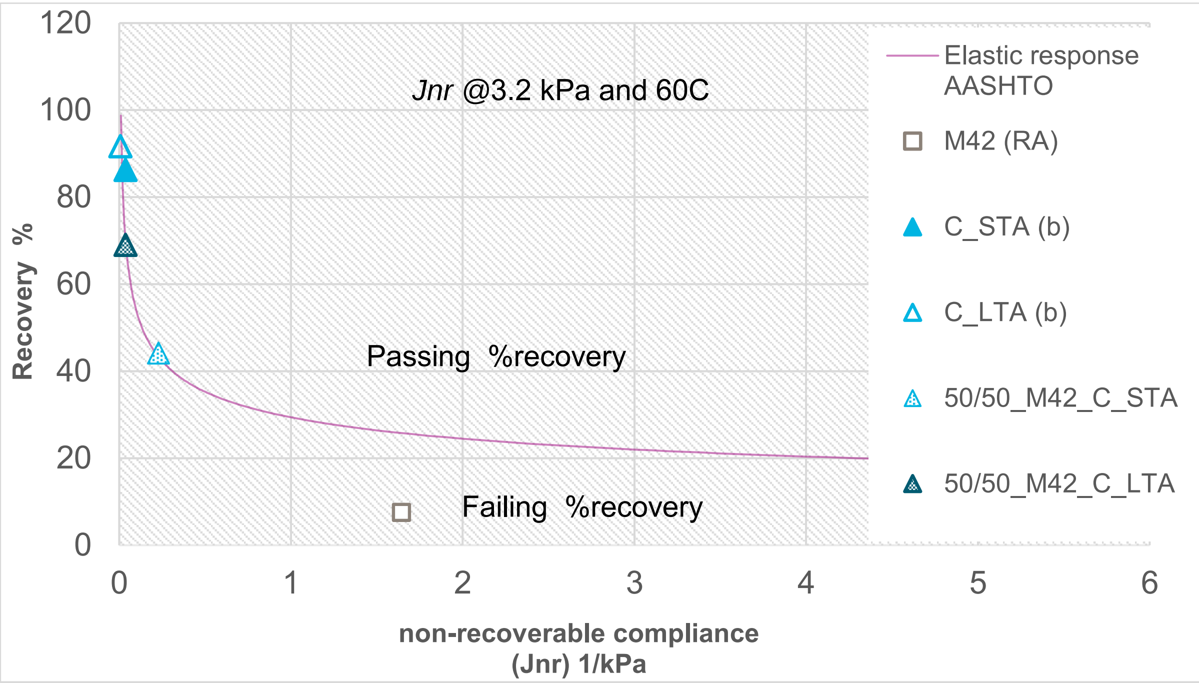


Figure 28. The elastic response for the PMB 'C' and RA 'M42' blends

4.2.4 Fourier Transform Infrared Spectroscopy (FTIR)

Figure 29 presents the analysis of total peaks for the sulfoxide for the blends of RA ‘M42’ with different PMBs. As expected, the total peaks increased with ageing for all the blends. The peak results of 75% PMB ‘B’ blends were, as expected, lower than for the 50% blends. The results for the different RAs ‘M42, M1, and A5’ were relatively higher than the PMBs and their blends. The S=O peaks for the RA ‘M42’ were slightly lower than other RAs ‘M1 and A5’

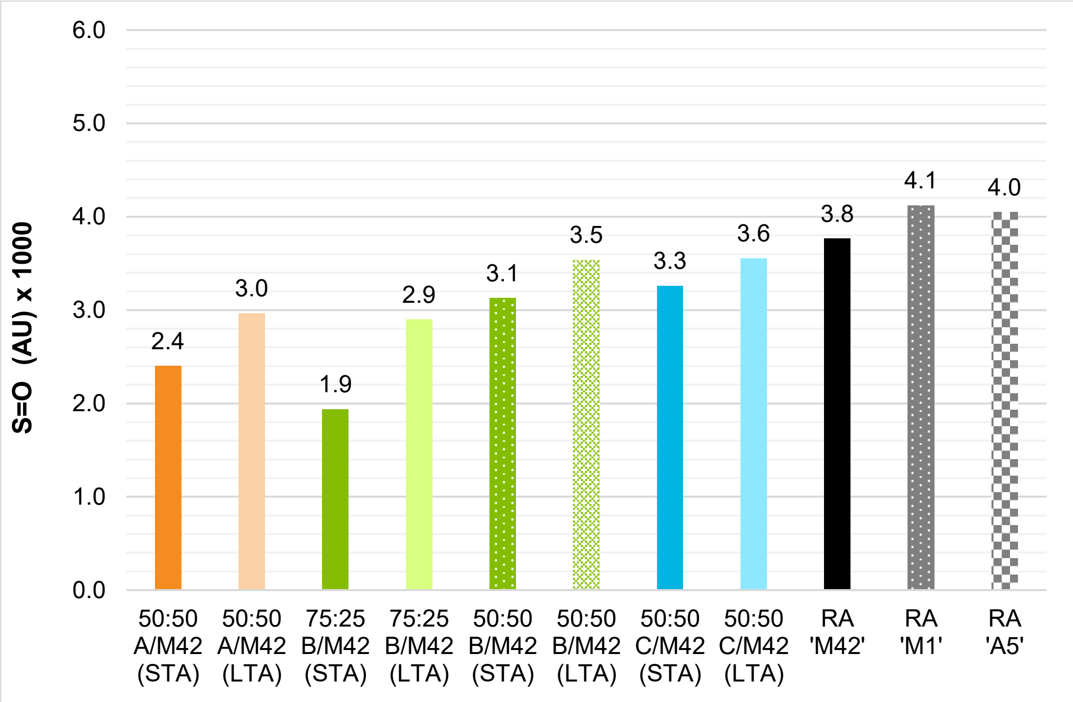


Figure 29. The sulfoxide peaks for the different blended bitumens and bitumen recovered from the RA

4.2.5 Saturates, Aromatics, Resins, and Asphaltenes (SARA)

Figure 30 presents the percentages of the four SARA fractions for different RAs ‘M42, M1 and A5’ in addition to the blends of RA ‘M42’ with the PMBs under different ageing conditions. The four SARA fractions varied from one RA to another, as shown in Figure 30, with the RA ‘M42’ having the greatest asphaltenes percentage. The effect of ageing on the blends was generally associated with an increasing percentage of asphaltenes and resins, and a decreasing percentage of aromatics and saturates. The addition of RA ‘M42’ to the virgin PMBs increased the percentage of asphaltene and decreased the percentage of aromatics. Figure 31 showed that all the RAs and the blends of RA ‘M42’ exhibited a sol-type behaviour <0.7 CI. The results in Figure 31 also showed that the blends of RA ‘M42’ changed towards a more gel-like colloidal structure with ageing. The blends of RA ‘M42’ with PMB ‘B’ showed the most sol-type behaviour (lowest CI values) in comparison to other blends which agreed with previous SARA results for the PMB ‘B’. The CI values were slightly increased by the addition of RA ‘M42’.

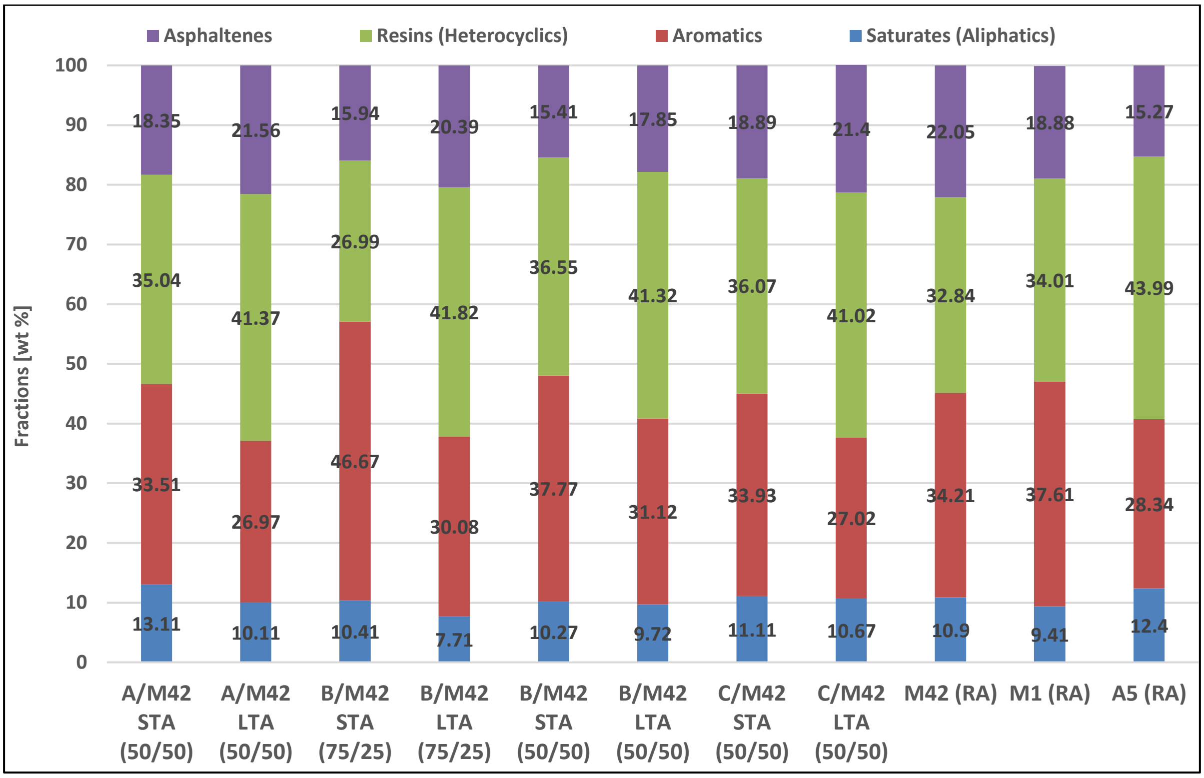
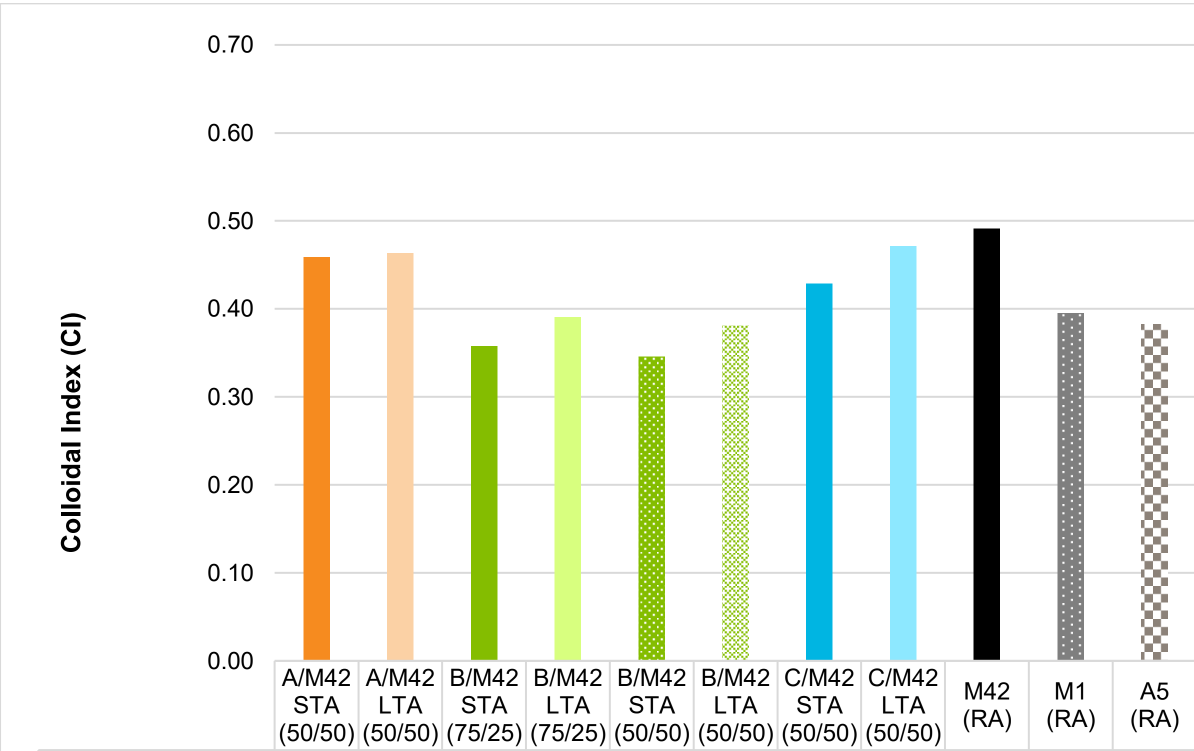


Figure 30. The SARA (saturate, aromatic, resins and asphaltenes) compositions



	A/M42 STA (50/50)	A/M42 LTA (50/50)	B/M42 STA (75/25)	B/M42 LTA (75/25)	B/M42 STA (50/50)	B/M42 LTA (50/50)	C/M42 STA (50/50)	C/M42 LTA (50/50)	M42 (RA)	M1 (RA)	A5 (RA)
Colloidal Index (CI)	0.46	0.46	0.36	0.39	0.35	0.38	0.43	0.47	0.49	0.40	0.38

Figure 31. Colloidal index (CI) for RA blends under different ageing conditions

4.3 Key Findings

The MRTFOT, in accordance with MCHW Clause 955, was used to mechanically blend the virgin PMBs with the recovered RA bitumen under STA and LTA conditions. The resultant bitumens were characterised for their physical and chemical properties. This section also presented the properties of the RAs. Given the results presented in this section, the following summary and conclusions are drawn:

1. The physical and chemical properties of the different RAs considered in this study suggested that the RAs had not been significantly aged. The test results on bitumens recovered from the RAs did not indicate a clear presence for polymer modification.

2. The combined effect of adding RAs and MRTFOT ageing can lead to significant hardening of the virgin PMBs.

3. The computed G-R parameters at low temperature (0°C) provided a consistent evaluation of the combined effect of MRTFOT ageing and the addition of RAs. The analysis of the G-R parameter showed that the addition of RAs to virgin PMBs with MRTFOT ageing can reduce the ability of materials to accommodate thermal and traffic-induced stresses.

4. An “acceptable” elastomeric polymer response, in accordance with AASHTO TP 70-13, can still be maintained by adding up to 50% of RAs to a highly polymer modified bitumen such as PMB ‘C’. The addition of 50% of RA did not maintain an “acceptable” elastomeric polymer response when blended with a lower polymer modified bitumen such as PMB ‘A’.

5. The chemical analysis using FTIR and SARA agreed with the physical analysis with regard to the combined effect of MRTFOT ageing and the addition of RAs. The FTIR analysis showed a consistent increase in S=O peaks with the addition of RAs and the MRTFOT ageing. The SARA analysis showed that the addition of RAs to the virgin PMBs and the MRFTOT ageing increased the percentage of asphaltene and decreased the percentage of aromatics indicating the hardening of the materials.

5. Stage 3: Asphalt Mixtures Containing RA

5.1 Introduction

The analysis presented in previous sections (Stage 1 and 2) provided an in-depth understanding of the properties of virgin PMBs and the combined effect of adding recovered RA bitumen and ageing on these properties. This section focuses on the properties of asphalt mixtures containing one virgin PMB and one RA source in addition to using various proportions of virgin aggregate materials. This section also presents the chemical and physical properties of the bitumens recovered from the mixtures. This section aims to provide an understanding of the interaction and performance of asphalt mixtures and the resultant bitumens (blends of virgin and RA bitumens). This section also provides a comparison between the properties of the resultant bitumens obtained from Stage 2 (directly blended using MRTFOT) and from the current stage (recovered from asphalt mixtures).

5.2 Properties of the Recovered Bitumens

The properties of the recovered bitumens from different asphalt mixtures (0% RA, 25% and 50% RA) are presented in this section. Following the analysis and findings from Stages 1 and 2, the selected RA was taken from planings from the ‘M42’ and the selected virgin bitumen was PMB ‘B’. The analysis of Stage 1 (Bitumen Testing) has shown that the PMB ‘B’ was more susceptible to polymer degradation during the laboratory ageing process. It was, therefore, decided to select this PMB for further analysis under the asphalt mixture conditions. The process of blending the virgin PMB to the asphalt mixture containing RA can provide a more realistic picture of the interaction between the virgin PMB and the RA bitumen, where it can be anticipated that not all bitumen in the RA will be available to interact with the added virgin PMB. The bitumen content found in the RA ‘M42’ was less than the designed bitumen content, which may not be surprising considering the action of traffic, environmental effects, and planing processes.

5.2.1 Penetration and Softening point:

The penetration and softening point results for the recovered bitumens are shown in Table 7 (Stage 3 column). Table 7 also includes the penetration and softening point results of PMB ‘B’ and the RA ‘M42’ blends from Stage 1 and Stage 2 to provide comparisons on the effect of different conditions associated with each stage, i.e. blending, interaction, ageing conditions, asphalt manufacture and recovery process. The results in Table 7 for stages 1 to 3 provided consistent results with minor differences that can be attributed to the different conditions associated with each stage. For example, the penetration and softening point for the recovered bitumen from 0% RA were very close to their corresponding values in Stage 1, e.g. PMB ‘B’ after STA. These results suggested that the recovery process had a negligible effect on degrading the crosslinked polymer network.

Table 7. Stages 1 to 3 Penetration and Softening Point Test Results

Test	Stage 1: PMB 'B' (75-130/75)			Stage 2 (MRTFOT blending) 25% RA 'M42'/ 75% PMB 'B'		Stage 2 (MRTFOT blending) 50% RA 'M42'/ 50% PMB 'B'		Stage 3: Asphalt Mixtures Recovered binders		
	NA	STA	LTA	STA	LTA	STA	LTA	0% RAP (Control)	25% RAP 'M42'	50% RAP 'M42'
Penetration (dmm)	94	66	40	52	33	36	25	69	58	44
Softening Point (°C)	90.5	88	67.6	73.6	65.6	61.6	66	85.5	63.6	72.6

5.2.2 Dynamic Mechanical Analysis (DMA)

Figure 32 shows the master curves and black diagrams of the bitumens recovered from asphalt mixtures made using different contents of RA 'M42', e.g. (0%, 25% and 50% of 'RA 'M42'). Increasing the RA content has increased the complex modulus over the frequency domain. The results of G* values agree with the penetration relationship between different RA contents shown in Table 7. The black diagrams for the recovered bitumens showed a typical 3-shape with phase angle values < 70 degrees for all the recovered bitumens indicating that a good crosslinked polymer network can still be established with up to 50% RA. This confirmed previous penetration and softening point values that the recovery process had a negligible effect on degrading the crosslinked polymer network. The results at this stage align in general with the corresponding bitumens in Stage 2.

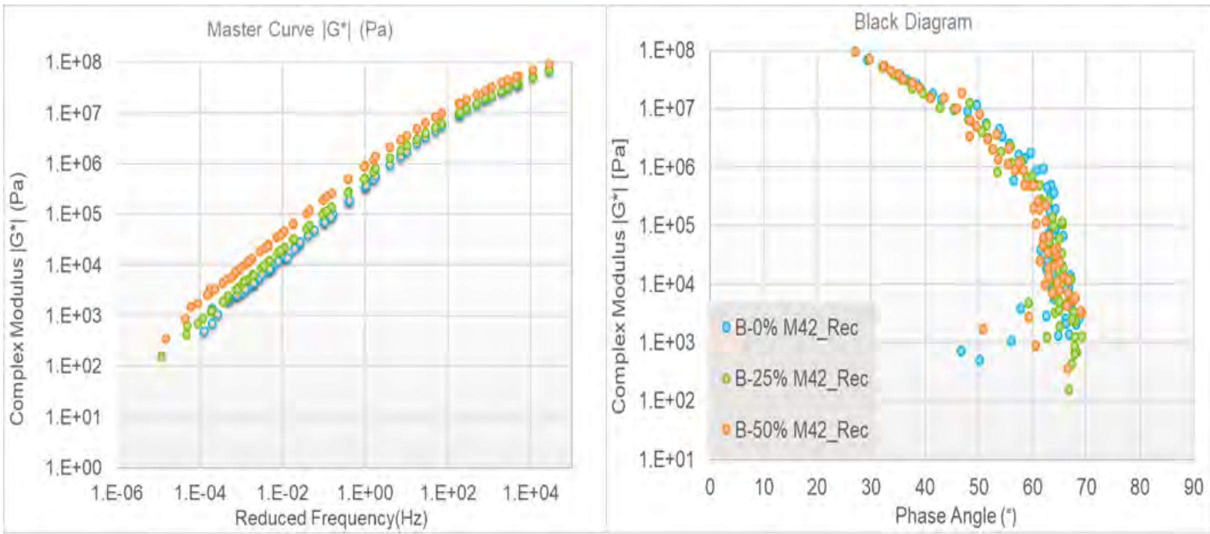
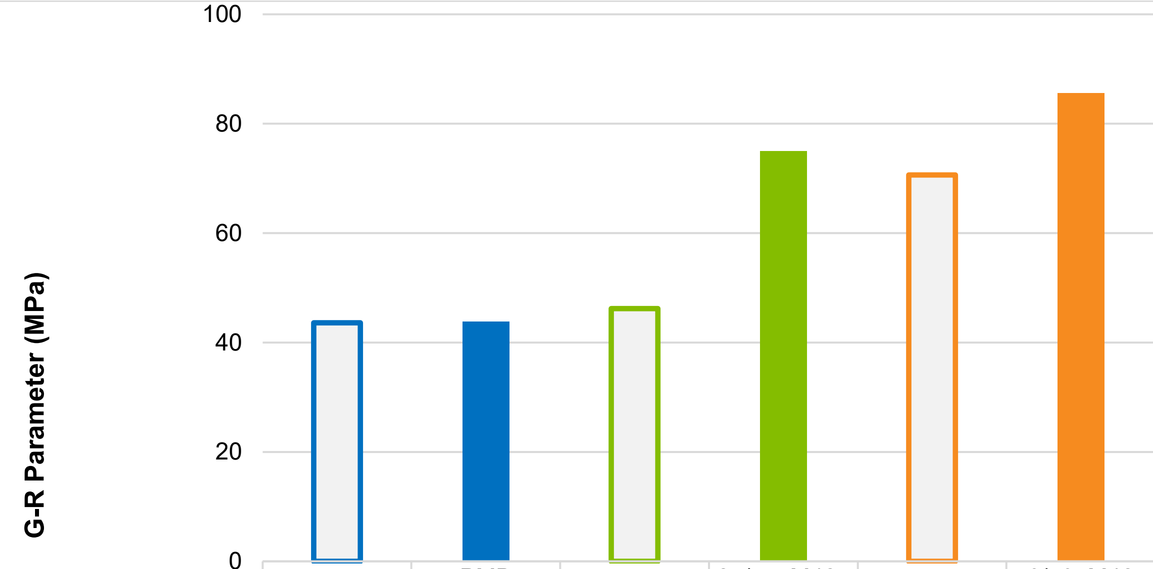


Figure 32: Master Curve at 25 °C and a black diagram for the recovered bitumens (Stage 3)

Figure 33 shows the results for the G-R parameters computed at 0 °C for bitumens recovered from mixtures (Stage 3) alongside their corresponding bitumens from Stage 1 and Stage 2. The G-R values for the PMB showed similar values under Stage 1 and Stage 3 conditions confirming the penetration and softening results in Table 7. The G-R values, as expected, increased with increasing the RA content. The addition of 25% RA resulted in only a slight increase in the G-R value. The G-R values for the bitumens recovered from asphalt mixtures containing RA (Stage 3) were generally smaller than their corresponding values in Stage 2.

The recovered bitumens (Stage 3) from asphalt mixtures made using 25% RA and 50% RA contained a larger amount of PMB ‘B’ than their corresponding bitumens in (Stage 2), Figure 24. The larger amount of the virgin PMB of (Stage 3) contributed to good rheological properties compared to their corresponding bitumens in (Stage 2).



	0%RA_rec (Stage 3)	PMB 'B'_STA (Stage 1)	25%RA_rec (Stage 3)	25/75_M42_B_STA (Stage 2)	50%RA_rec (Stage 3)	50/50_M42_B_STA (Stage 2)
G-R Parameter (MPa)	44	44	46	75	71	86

Figure 33. G-R parameter computed at 0 °C and 1.59 Hz

5.2.3 Multiple Stress Creep and Recovery (MSCR)

The average percentage recovery at 3.2 kPa stress, versus the average non-recoverable creep compliance at 3.2 kPa stress is shown in Figure 34. The PMB ‘B’ from Stage 1 and the blended bitumens (25% and 50% RA ‘M42’) after STA from Stage 2 were also introduced alongside the recovered bitumens (Stage 3) for comparison. For example, the recovered bitumens from asphalt mixtures made using 0% RA, 25% RA and 50% RA corresponding to the bitumens in Stage 1 and Stage 2 of PMB ‘B’ STA, 25/75 M42/B STA and 50/50 M42/B STA, respectively. Figure 34 shows that as RA content increases, the relative difference between the corresponding bitumens also increases. All the recovered bitumens fell above the line indicating an “acceptable” elastomeric polymer response which confirmed previous results. The results of recoveries, as expected, decreased with the increase of RA content.

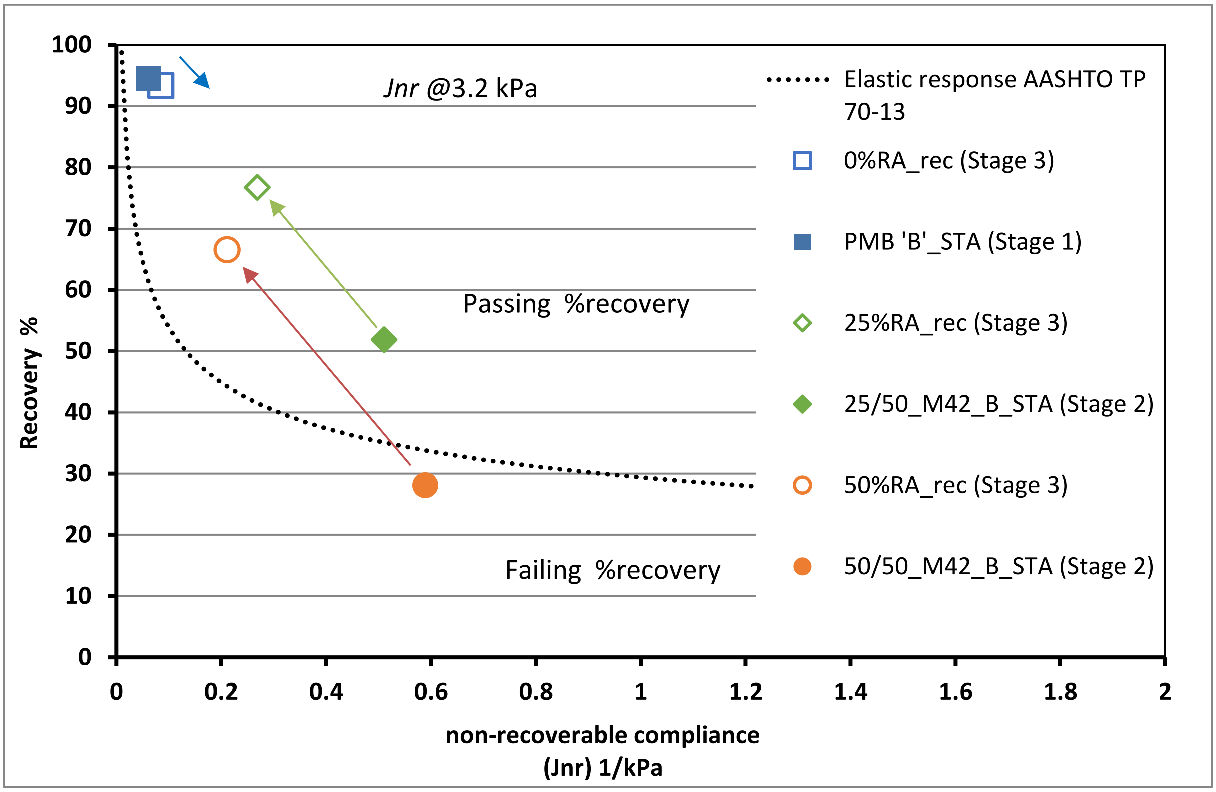


Figure 34. The elastic response of the RA bitumens and their corresponded blends from Stage 2

5.2.4 Fourier Transform Infrared Spectroscopy (FTIR)

Figure 35 shows the S=O peaks for the recovered bitumens from asphalt mixtures made using 0% RA, 25% RA ‘M42’ and 50% RA ‘M42’, in addition to the results of PMB ‘B’ and the RA ‘M42’ blends from Stage 1 and Stage 2, to evaluate the effect of different conditions associated with each stage. The results in Figure 35 showed an almost consistent trend with regards to the impact of RA content where the S=O peaks increased with increasing the RA content. However, the S=O peaks of the recovered bitumens from the asphalt mixtures were almost three times greater than those of their corresponding bitumens from Stage 1 and Stage 2. It should be mentioned that the FTIR measurements are very sensitive to any trace of contamination that could have remained during the recovery process.

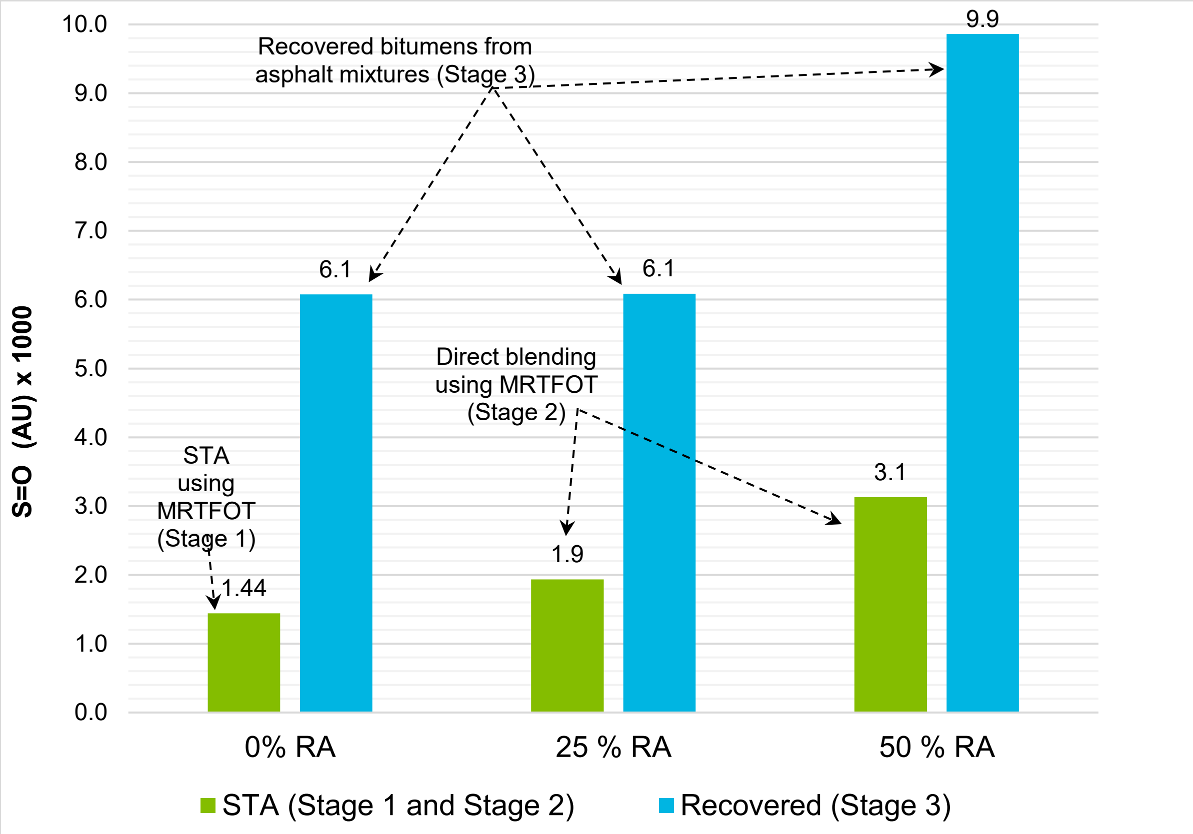


Figure 35. The sulfoxide peaks for the blended and recovered bitumens

5.2.5 Saturates, Aromatics, Resins, and Asphaltenes (SARA)

Figure 36 shows the percentages of the four SARA fractions for the recovered bitumens from asphalt mixtures made using 0% RA, 25% RA ‘M42’ and 50% RA ‘M42’ in addition to their corresponding bitumens from Stage 1 and Stage 2. The recovered bitumens showed higher asphaltenes and lower aromatics compared to their corresponding bitumens from Stage 1 and 2, The recovered bitumens showed an increase in the asphaltenes and a decrease in the aromatics with increasing RA content. The colloidal indices, as shown in Figure 37 exhibited an increase for the recovered bitumens in comparison to their corresponding bitumens. The bitumens changed towards a more gel-like colloidal structure with increasing RA, indicating that the bitumens would be more susceptible to thermal and traffic-induced stresses. However, all the bitumens are considered to have sol-type behaviour <0.7 CI. The chemical results for the recovered bitumens (Stage 3) seemed to contradict the G-R results in Figure 33, i.e. higher CI values would indicate lower thermal and traffic cracking resistance. However, the larger amount of virgin PMB in the recovered bitumens of (Stage 3) are suggested to have a dominant effect in lowering the G-R values over the SARA fractions.

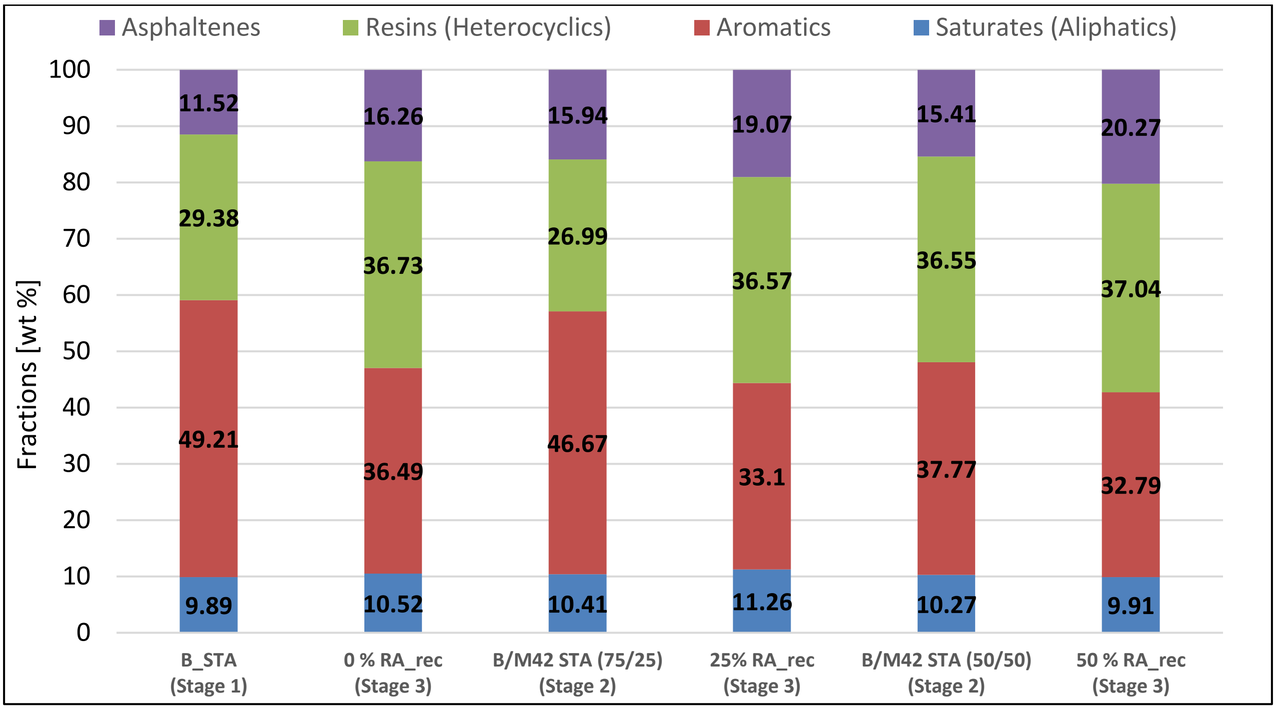


Figure 36. The SARA (saturate, aromatic, resins and asphaltenes) compositions

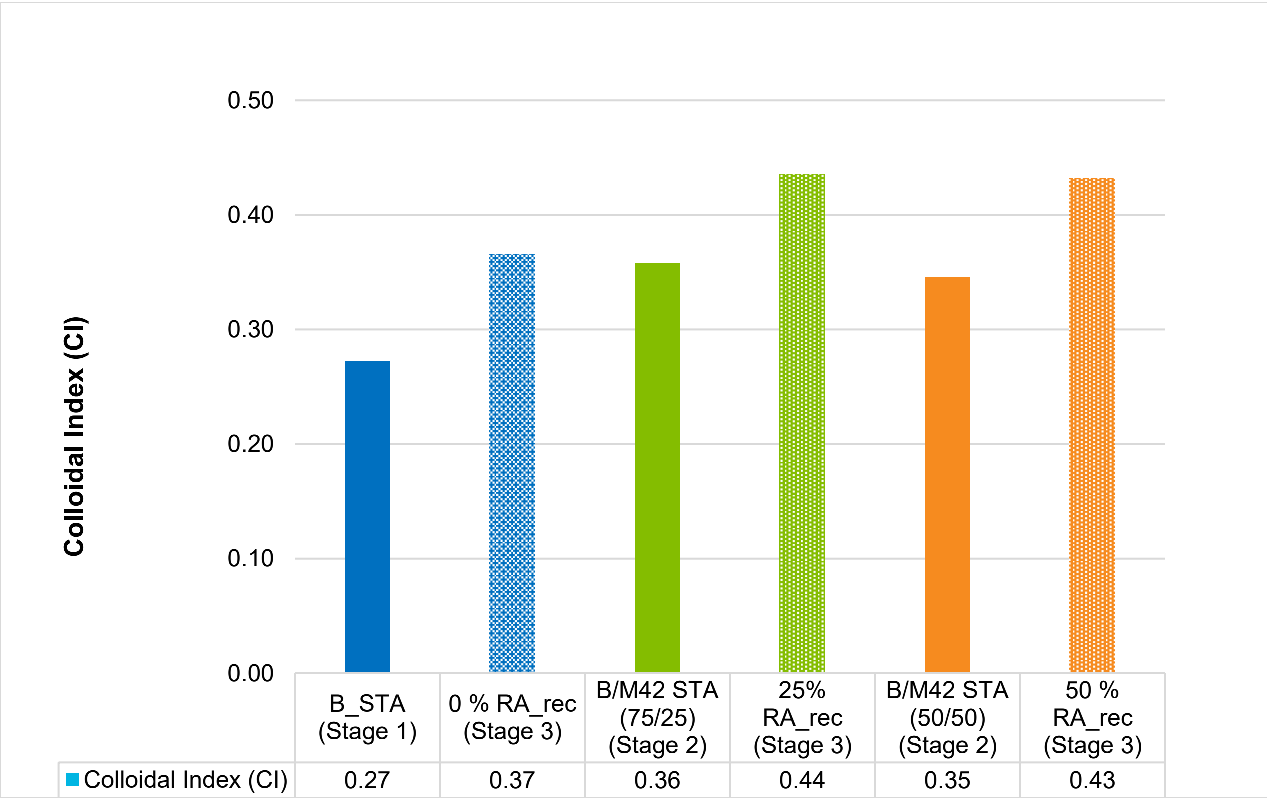


Figure 37. Colloidal index (CI) for the blended and recovered bitumens

5.3 Asphalt Mixture Testing

The asphalt mixtures were produced at 0% RA with 100% virgin aggregate (Control) mixes, 25% RA with 75% virgin aggregates and 50% RA with 50% virgin aggregates. The next sections present the results of mechanical testing conducted on those mixtures,

5.3.1 Composition Analysis

The test results of the compositional analysis are graphically shown in Figure 38. The grading analysis of 0% RA and 25% RA comply with specification requirements. However, the bitumen content of 25% RA and 50% RA were found to be below the designed bitumen content. The results of bitumen content for the 25% RA and 50% RA mixes suggested that the active bitumen availability from the RA is less than 90%.

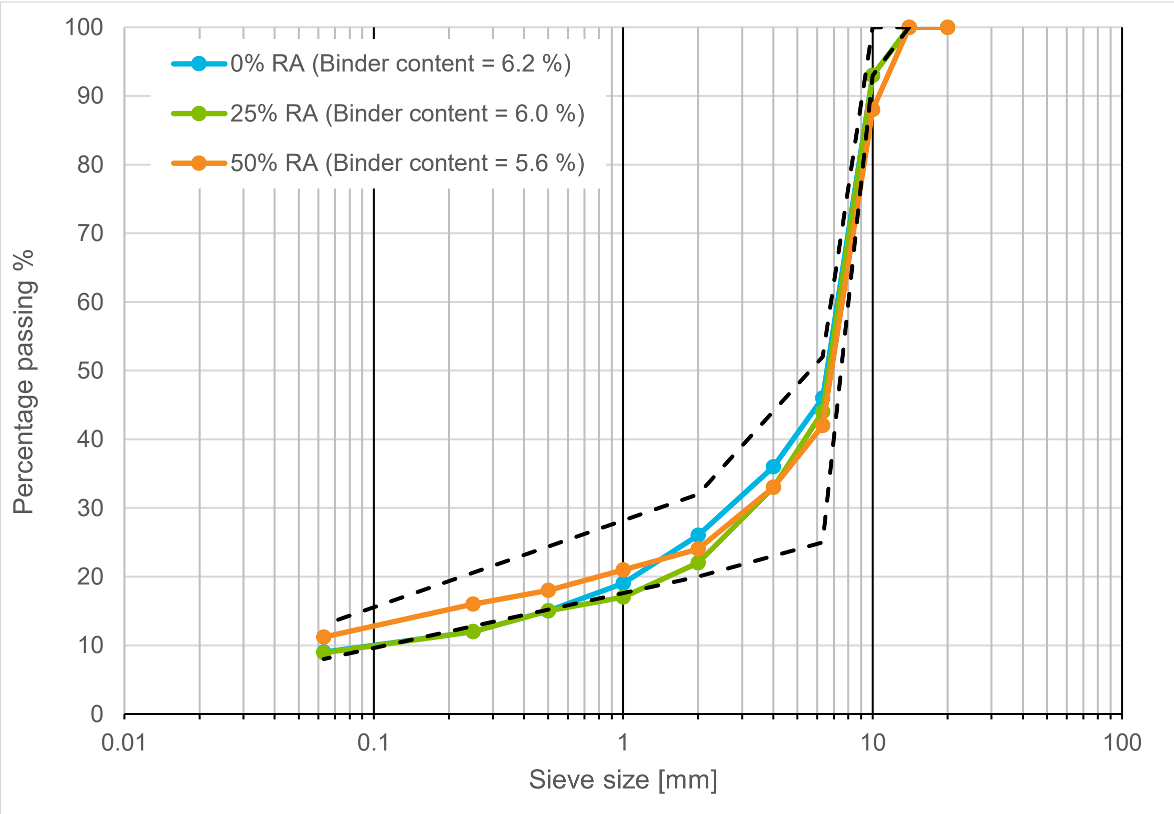


Figure 38. Compositional analysis of 0% RA, 25% RA and 50% RA mixtures.

Despite the reduced found bitumen content, it did not present any adverse impact on compliance with specification requirements for compactability, the volumetric properties and the mechanical properties of these manufactured asphalt mixtures. Details are presented in the following sections.

5.3.2 Volumetric Properties of Mixtures

The bulk densities of the asphalt mixtures were measured in accordance with BS EN 12697-6:2020 - Method B. The findings shown in Figure 39, represent the average values of at least 8 samples for each mixture with the range bars representing the maximum and minimum values. The maximum density of the control mixture (0% RA) was slightly lower than the 25% RA and the 50% RA. The 0% RA mixture had air void contents of 4.6% while those with 25% RA had air void contents of 5.8% and 50% RA had air void contents of 3.2%. The results of air voids complied with requirements (mean- 2 to 6%), although there is a variation between the minimum and maximum values.

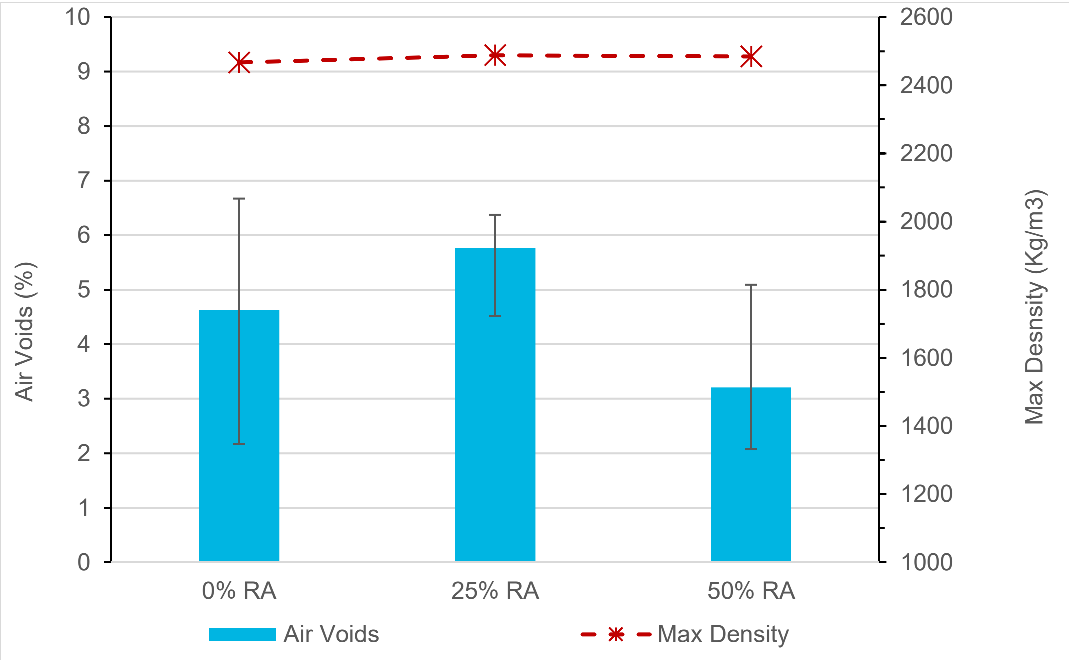


Figure 39. Volumetric Properties of 0% RA, 25% RA and 50% RA mixtures.

5.3.3 Indirect Tensile Stiffness Modulus (ITSM)

ITSM testing was conducted at 20°C in accordance with BS EN 12697-26: 2004 (Annex C). The ITSM test results for the asphalt mixtures are illustrated in Figure 40. The ITSM test results were all in the range of 1,000 to 2,000 MPa with the ITSM increasing with the increase of RA content. These results agree with the rheological properties of the recovered bitumens presented in Table 7 and Figure 32. Published 20°C ITSM data for thin surfacings (Stone Mastic Asphalt (SMA)) are reported to have a typical stiffness value in the range of 1,000 MPa to 3,500 MPa when new [27]. This can increase by a factor of 1.5 to 2.0 when aged in service.

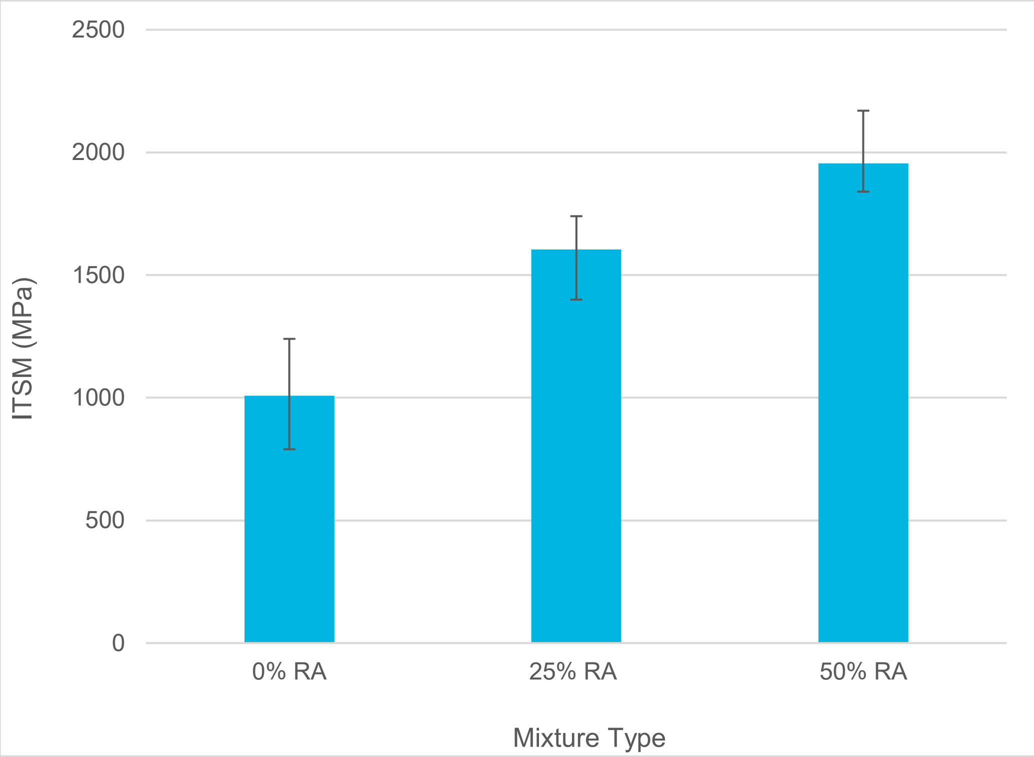


Figure 40. ITSM results of 0% RA, 25% RA and 50% RA.

5.3.4 Wheel Tracking Tests (WTT)

Resistance to deformation was measured using Wheel Tracking Tests (WTT) in accordance with EN 12697-22:2020 using the small device to 10,000 cycles. Each result presented for WTT is the mean value of the 2 comparable samples tested. Figure 41 shows the rut depth at 10,000 cycles and the wheel track slope (WTS). The values of the measured rut depth ranged from 2.4 mm for the 25% RA to 2.8 mm for the 50% RA. The wheel track slope (WTS) in air measurements were in the range of 0.039 mm/1000 cycles - 0.059 mm/1000 cycles and comply with requirements defined in PD 6691:2015+A1:2016. The results of WTT indicated that all mixtures have excellent rutting resistance.

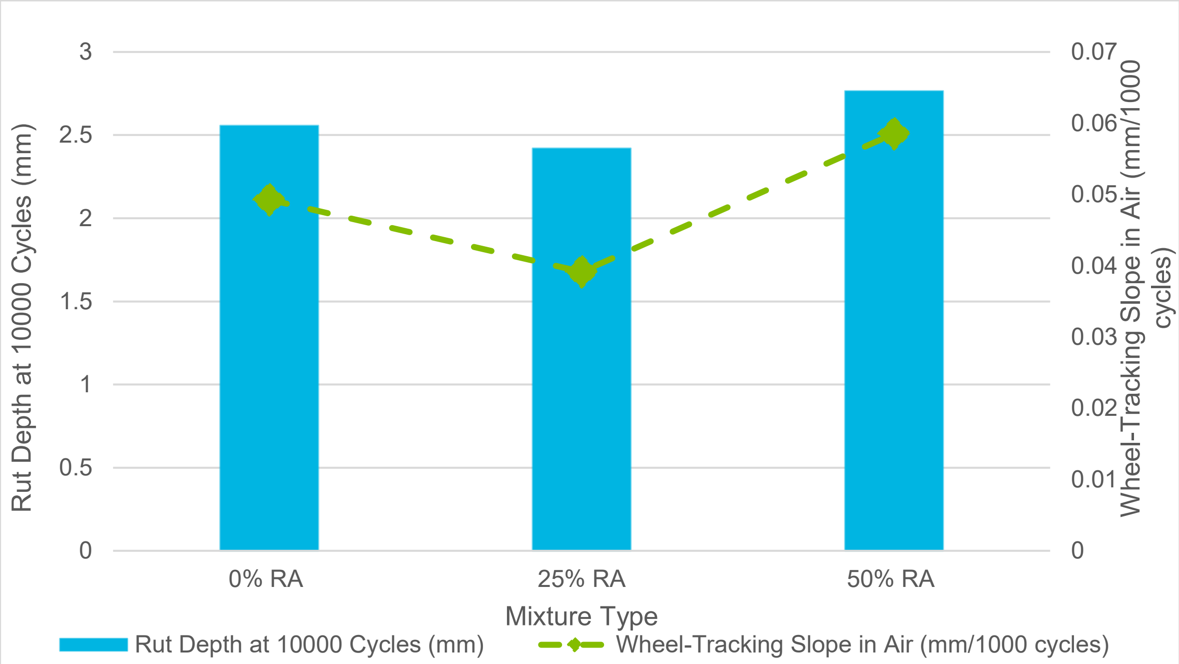


Figure 41. WTT results for 0%RA, 25% RA, and 50% RA

5.3.5 Resistance to Moisture Damage

Resistance to moisture damage of the asphalt mixtures was measured by conducting water sensitivity testing in accordance with EN 12697-12:2018 – Method A.

Figure 42 shows the average Indirect Tensile Strength of the wet group (ITSw) and the dry group (ITSd) of samples, in kPa. The values of both the ITSw and the ITSd increase as the percentage of RA increases.

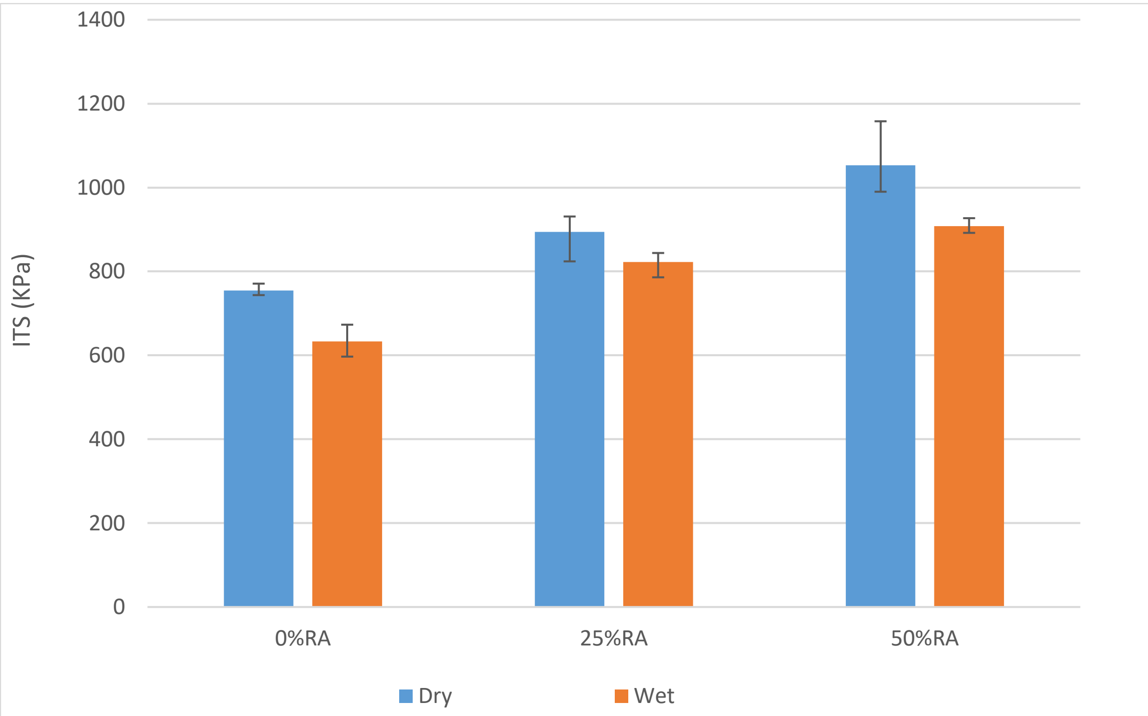


Figure 42. Indirect Tensile Strength (ITS) of 0% RA, 25% RA and 50% RA

Figure 43 shows the Indirect Tensile Strength Ratio (ITSR) of all three mixtures exceeding 80%.

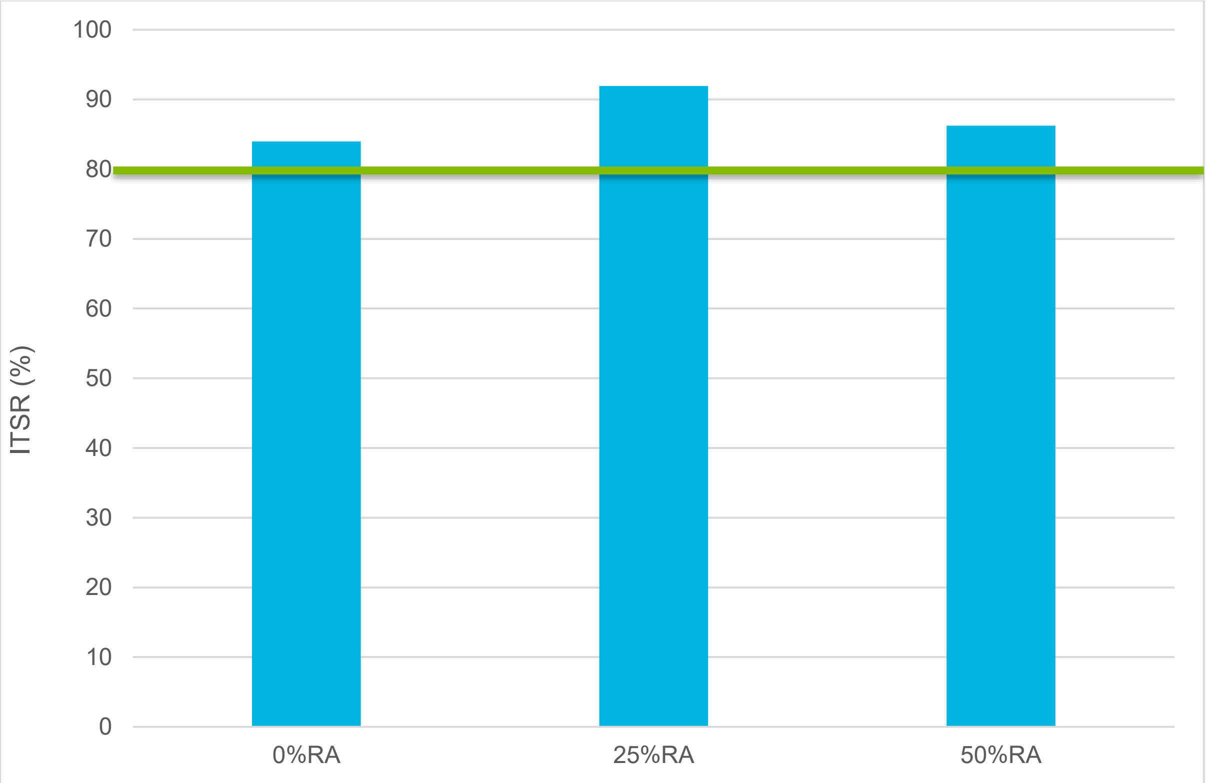


Figure 43. Indirect Tensile Strength Ratio (ITSR) of 0% RA, 25% RA and 50% RA

5.4 Key Findings

This section presents the results of recovered bitumens alongside the asphalt mixtures made using 0% RA with 100% virgin aggregate (Control) mixes, 25% RA with 75% virgin aggregates and 50% RA with 50% virgin aggregates. The bitumen testing included rheological and chemical evaluation. The asphalt mixture testing included compositional analysis, volumetric properties, ITSM, WTT and water sensitivity testing. Given the results presented in this section, the following findings are drawn:

1. There were negligible differences between the physical properties of the bitumen recovered from the control asphalt mixture (containing 100% PMB 'B') and the same bitumen obtained after STA using MRTFOT. Such results indicate that the bitumen recovery process had minimal impact on the polymer crosslinked network.

2. The SARA analysis indicated that the process of asphalt mixture manufacturing and/or the bitumen recovery process had more effects on increasing the colloidal index (CI) than the conditions of the STA using MRTFOT.

3. Increasing the RA content to 50% can reduce the resistance of bitumens to resist thermal and traffic-induced stresses. On the other hand, the addition of 25% RA showed comparable performance to the control materials (100% PMB) in terms of low temperature properties.

4. The MSCR analysis of bitumen recovered from mixtures showed that up to 50% of RA can be added to asphalt mixtures made with highly modified PMB without compromising the elastic recovery properties of the recovered bitumens.

5. The bitumen content found from asphalt mixtures made using 25% RA and 50% RA was lower than the designed bitumen content. Whilst this might suggest that the assumed active bitumen availability of 90% may be overestimated, the test results did not show any adverse impact on compliance with the volumetric, mechanical properties and compactability requirements of these mixtures.

6. The ITSM values were higher for asphalt mixtures containing higher addition of RA materials.

7. The asphalt mixtures produced with 25% and 50% RA showed comparable mechanical properties to the mixtures containing 0% RA, when tested for rutting resistance and water sensitivity in the laboratory.

6. Stage 4: Recyclability Assessments

6.1 Introduction

This section focuses on the end of life and recyclability assessment of aged RA subjected to multiple recycling. The asphalt mixtures produced in Stage 3 from 50% RA were further processed (crushed and lab-aged at 85°C for 120 hours) to produce laboratory aged RA (labelled RA1). This section presents the performance and mechanical results of asphalt mixtures produced using 50% laboratory aged RA and one PMB bitumen (PMB 'B') plus virgin aggregate. Figure 44 presents the methodology of Stage 4. The rheological properties of the recovered bitumens from the laboratory aged RA and the asphalt mixture containing 50% of laboratory aged RA are also indicated. This section also provides a recommended framework and guidance for the design factors that need to be considered when assessing RA incorporating PMB.

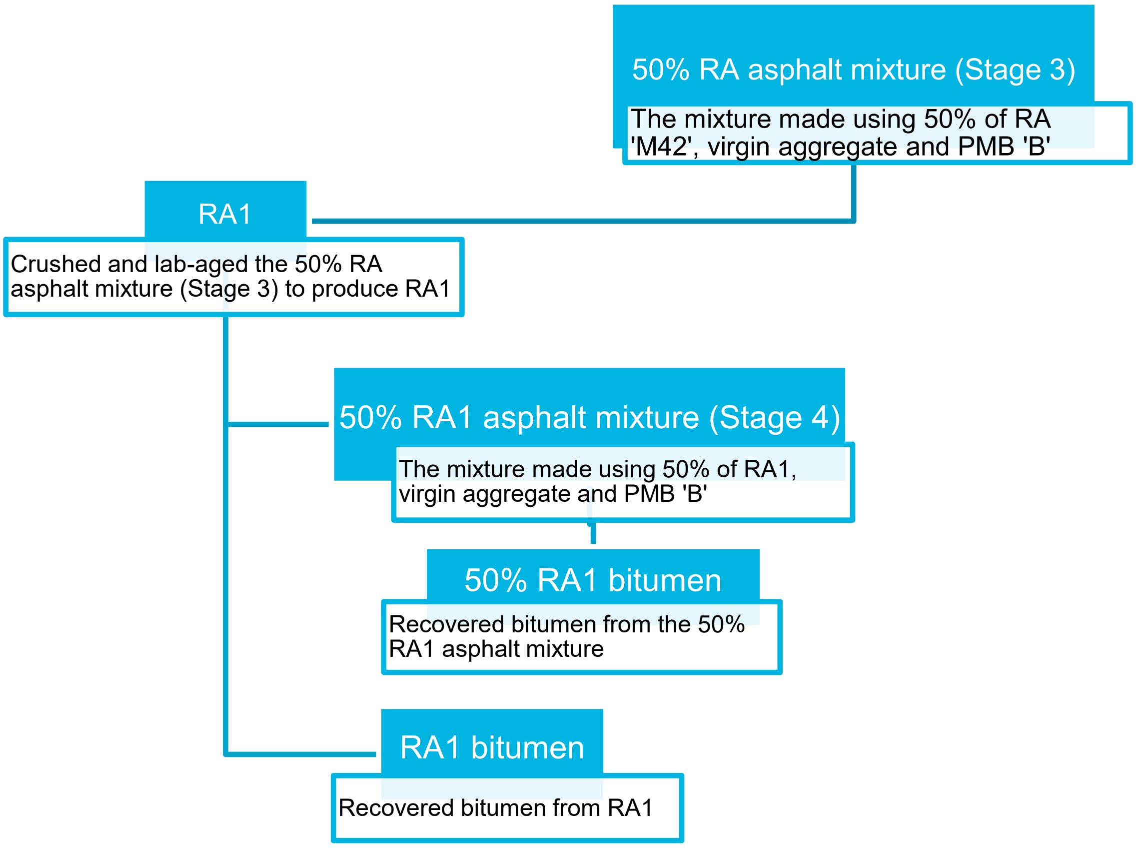


Figure 44. Stage 4 methodology

6.2 Properties of the Recovered Bitumens

6.2.1 Penetration and Softening Point

The penetration and softening point results for the bitumens recovered from RA1 and 50% RA1 mixtures are shown in Table 8 (Stage 4 column) – denoted as RA1 and 50% RA1. Table 8 also includes these results for recovered bitumen from asphalt mixture made using 50% RA in Stage 3 and before additional ageing to provide comparisons on the effect of the laboratory ageing process. The recovered bitumen from RA1 had a lower penetration and softening point in comparison with the recovered bitumens from mixtures containing 50% RA and 50% RA1 in Stage 3 and Stage 4, respectively. The decrease in the penetration values for the RA1 bitumen can be attributed to the ageing effect while the decrease of softening point for the RA1 might be attributed to partial breakdown of the crosslinked polymer network. The penetration and softening point for the recovered bitumen from 50% RA1 in Stage 4 were very close to the penetration and softening point values in Stage 3.

Table 8. Stages 3 to 4 Penetration and Softening Point Test Results

Test	Stage 3 Recovered bitumen	Stage 4 Recovered bitumens	
	50% RA 'M42'	RA1	50% RA1
Penetration (dmm)	44	33	44
Softening Point (℃)	72.6	66.8	71

6.2.2 Dynamic Mechanical Analysis (DMA)

Figure 45 shows the master curves and black diagrams of the recovered bitumens from (Stage 4), i.e. bitumens recovered the RA1 and asphalt mixtures containing 50% of RA1. The results of the bitumen recovered from an asphalt mixture containing 50% RA (Stage 3) are included for comparisons. The RA1 bitumen has increased complex modulus over the frequency domain compared to other bitumens. The G* master curves for the bitumens recovered from 50% RA (Stage 3) and 50% RA1 (Stage 4) were very similar.

The results of G* values agree with the penetration relationship between different recovered bitumens (Stage 3 and Stage 4) shown in Table 8. The black diagrams for the 50% RA1 recovered bitumen had phase angle values < 70 degrees indicating a good polymer crosslinked network.

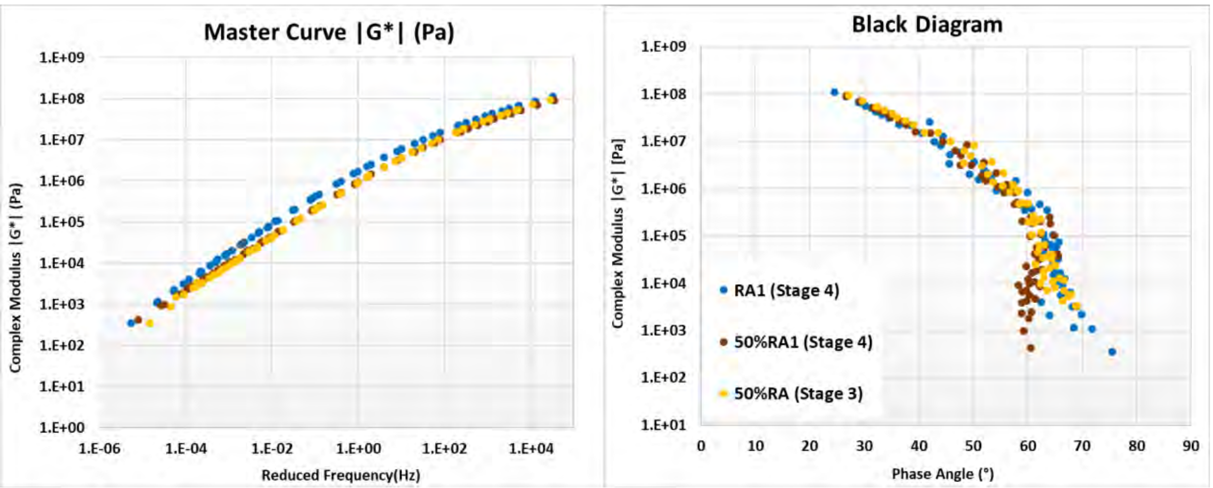


Figure 45. Master Curve at 25 °C reference temperature and a black diagram for the recovered bitumens (Stage 3 and Stage 4)

Figure 46 shows the results for the G-R parameters computed at 0 °C for the recovered bitumens (Stage 4) alongside the recovered bitumen from 50% RA (Stage 3). The G-R value for the bitumen recovered from RA1 was the highest among other bitumens indicating the least thermal and cracking resistance. The bitumens recovered from 50% RA1 (Stage 4) had comparable low temperature performance to the bitumen recovered from 50% RA (Stage 3).

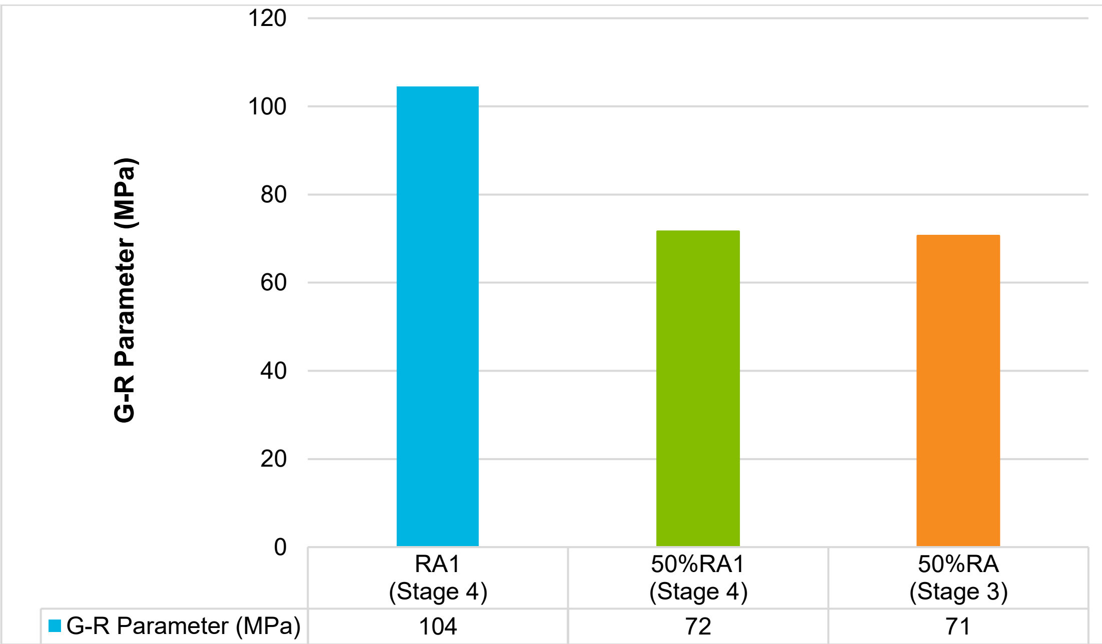


Figure 46. G-R parameter computed at 0 °C and 1.59 Hz

6.2.3 Multiple Stress Creep and Recovery (MSCR)

Figure 47 presents the average percentage recovery at 3.2 kPa stress, versus the average non-recoverable creep compliance at 3.2 kPa stress. All the recovered bitumens (from Stage 4 and the 50% RA from Stage 3) fell above the line indicating an “acceptable” elastomeric polymer. The results for the 50% RA1 (Stage 4) had the highest recovery and the lowest Jn value indicating excellent rutting performance. These results highlighted the desirable properties imparted by the PMB already available in the RA1 mixtures in addition to the added virgin PMB.

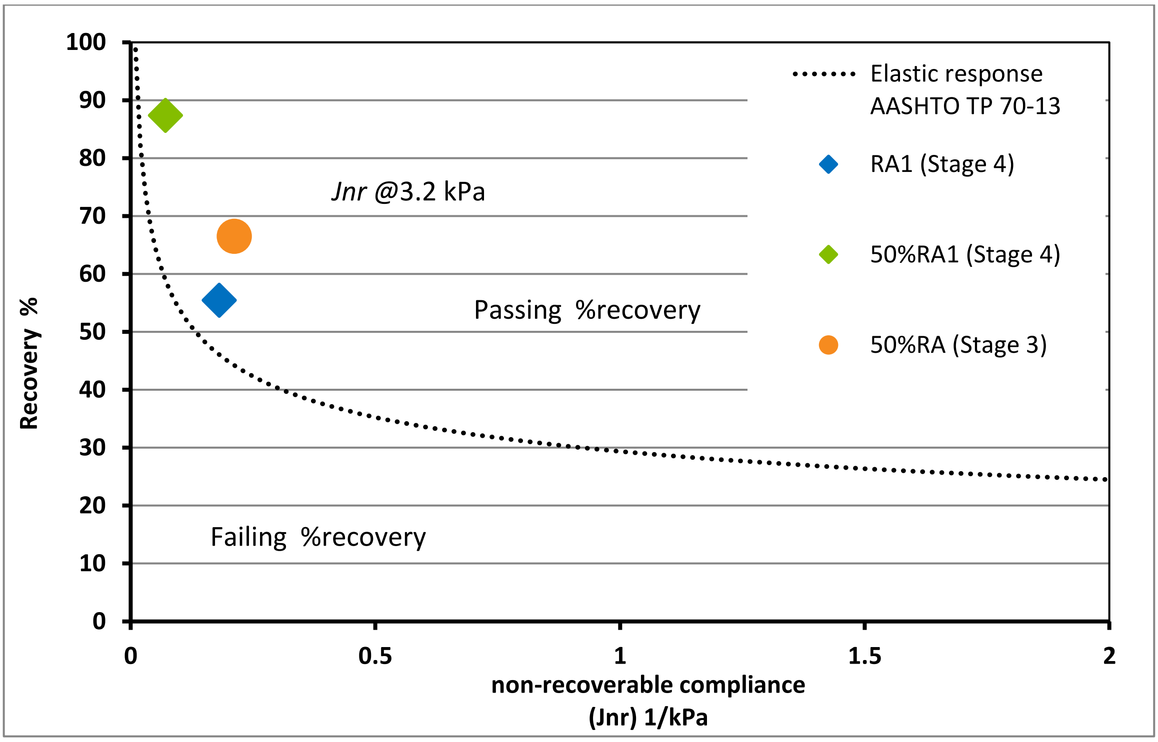


Figure 47. The elastic response of the recovered bitumens from Stage 3 and 4

6.3 Asphalt Mixture Testing

This section presents the mechanical testing for asphalt mixtures produced using 50% of RA1 (lab-aged RA mixture) with virgin PMB and virgin aggregate materials. The results of the original control asphalt mixture (containing 0% RA) was also included to provide comparisons on the effect of multiple recycling.

6.3.1 Volumetric Properties of the Mixtures

The findings shown in Figure 48 represents the average values of at least 8 samples for each mixture with the range bars representing the maximum and minimum values. The 0% RA had air void contents of 4.6% while the 50% RA1 had air void contents of 4.8%. The results of air voids complied with the target air voids requirements (2% to 6%), although there is a variation between the minimum and maximum values.

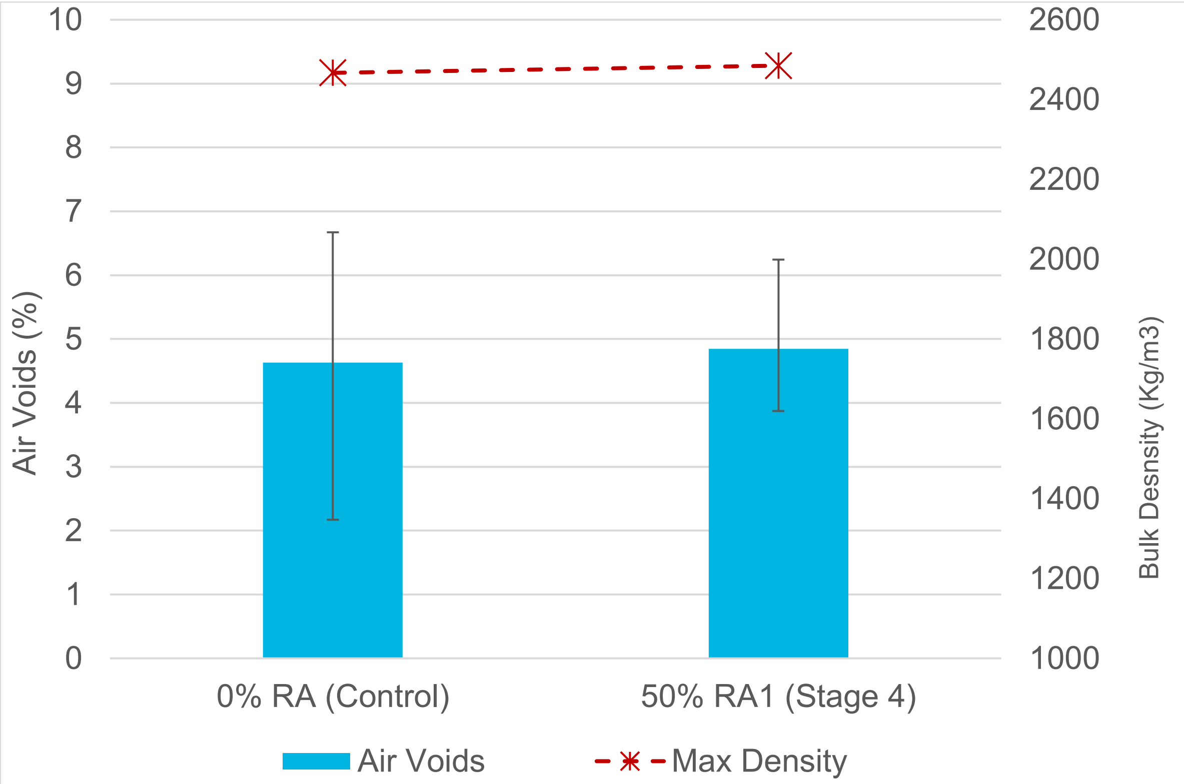


Figure 48. Volumetric Properties of 0% RA (Control) and 50% RA1 (Stage 4.2) mixtures.

6.3.2 Indirect Tensile Stiffness Modulus (ITSM)

The ITSM test results for the asphalt mixtures are shown in Figure 49. The ITSM test results were all in the range of 1,000 to 2,000 MPa with the ITSM increased from 1010 MPa to 1580 MPa with the addition of 50% RA1.

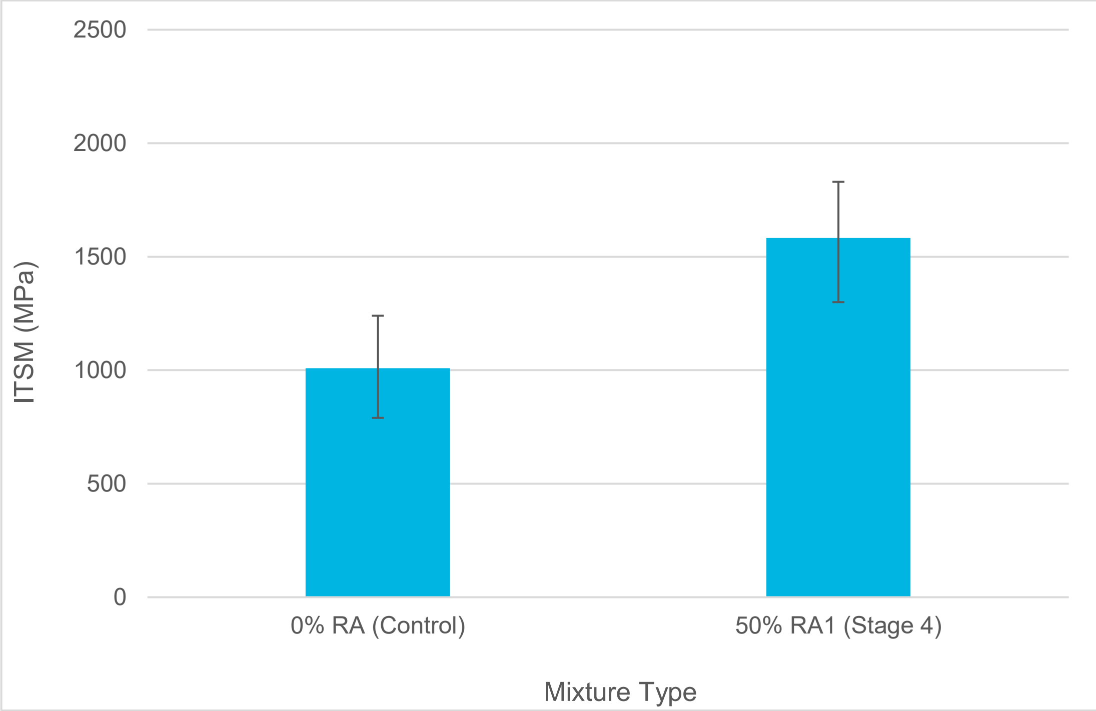


Figure 49. ITSM results of 0% RA (Control) and 50% RA1 (Stage 4).

6.3.3 Wheel Tracking Tests (WTT)

Figure 50 shows the results of Wheel Tracking Tests (WTT) in accordance with EN 12697-22:2020 using the small device to 10,000 cycles. Each result presented for WTT is the mean value of 2 comparable samples tested. Figure 50 shows the rut depth at 10,000 cycles and the wheel track slope (WTS). The value of the measured rut depth for the 0% RA (Control) was 2.55 mm while the rut depth value for the 50% RA1 was 2.11 mm The wheel track slope (WTS) in air measurements were in the range of 0.049 mm/1000 cycles - 0.052 mm/1000 cycles which comply with the requirements defined in PD 6691:2015+A1:2016. The results of WTT indicated that both mixtures have excellent rutting resistance.

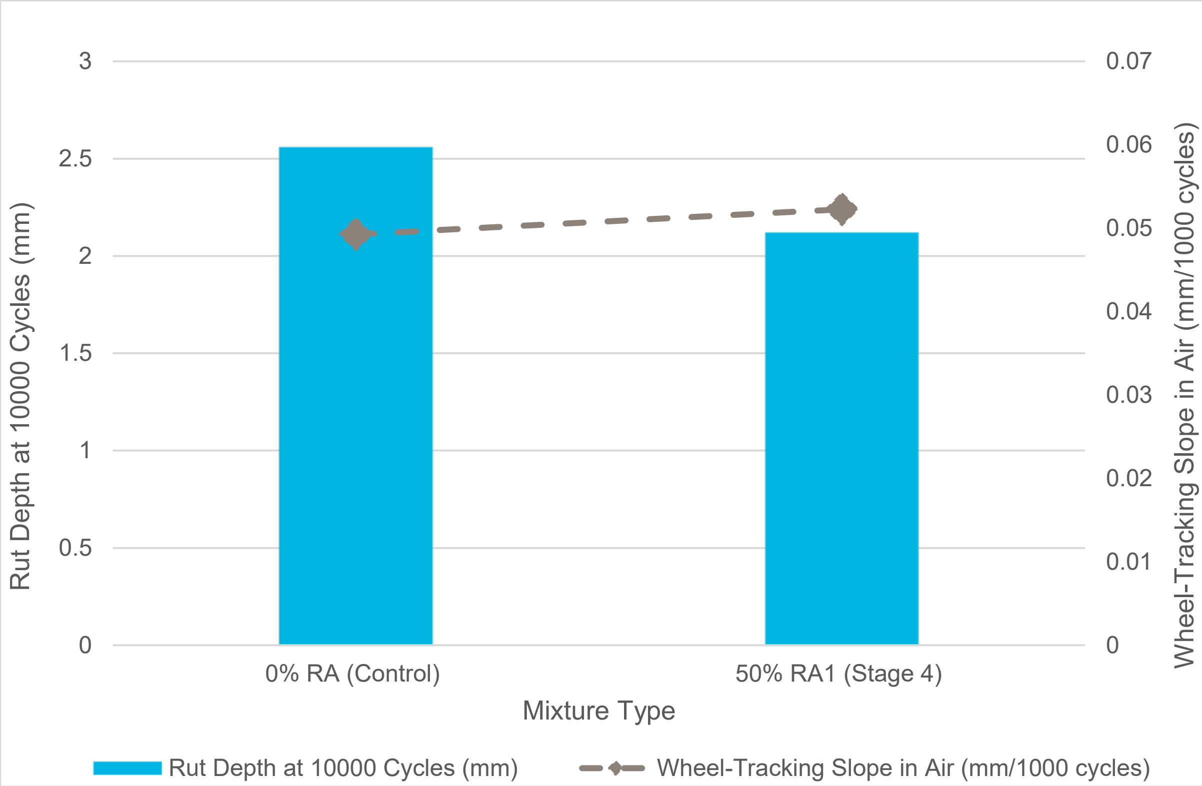


Figure 50. WTT results for 0%RA (Control) and 50% RA1 (Stage 4)

6.3.4 Resistance to Moisture Damage

Resistance to moisture damage of the asphalt mixtures was assessed by conducting water sensitivity testing in accordance with EN 12697-12:2018 – Method A. Figure 51 shows the average Indirect Tensile Strength of the wet group (ITSw) and the dry group (ITSd) of samples, in kPa. The values of both the ITSw and the ITSd increased with the addition of 50% RA1. Figure 52 shows the Indirect Tensile Strength Ratio (ITSR) of the asphalt mixture containing 50% RA1 had a higher ITSR ratio than the control mixture indicating a good resistance to moisture damage.

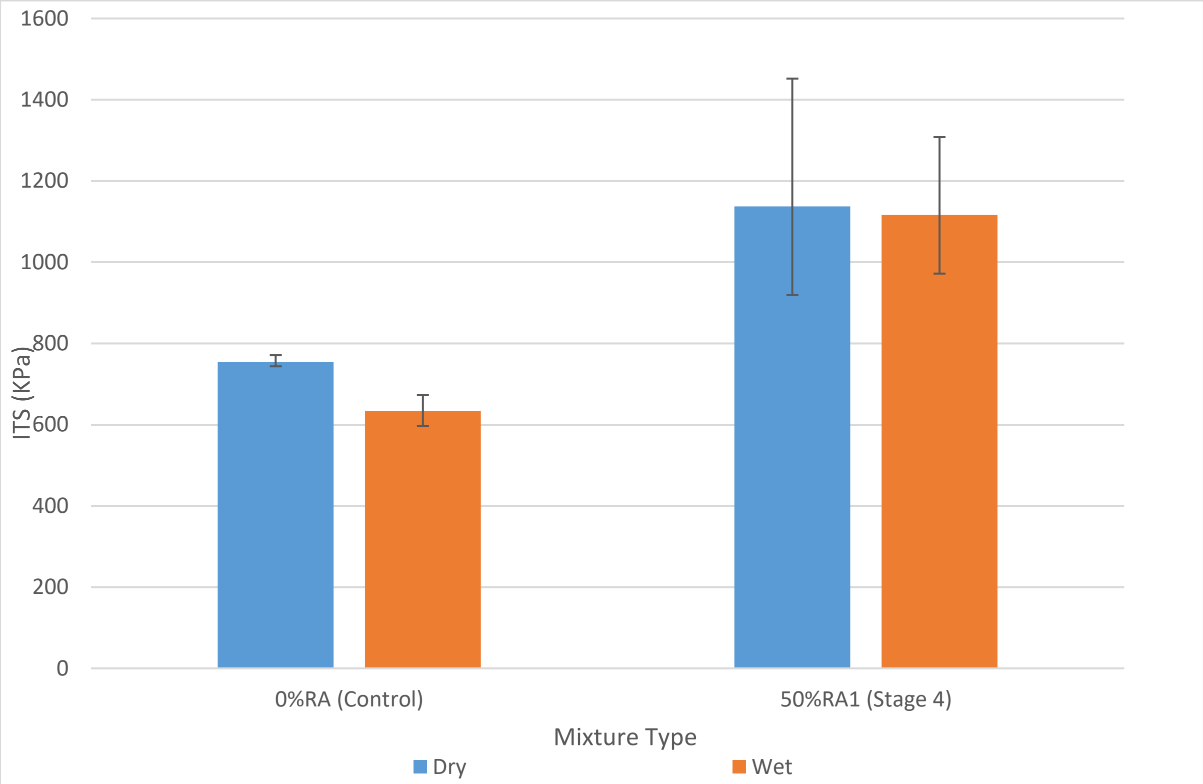


Figure 51. Indirect Tensile Strength (ITS) of 0% RA (Control) and 50% RA1 (Stage 4)

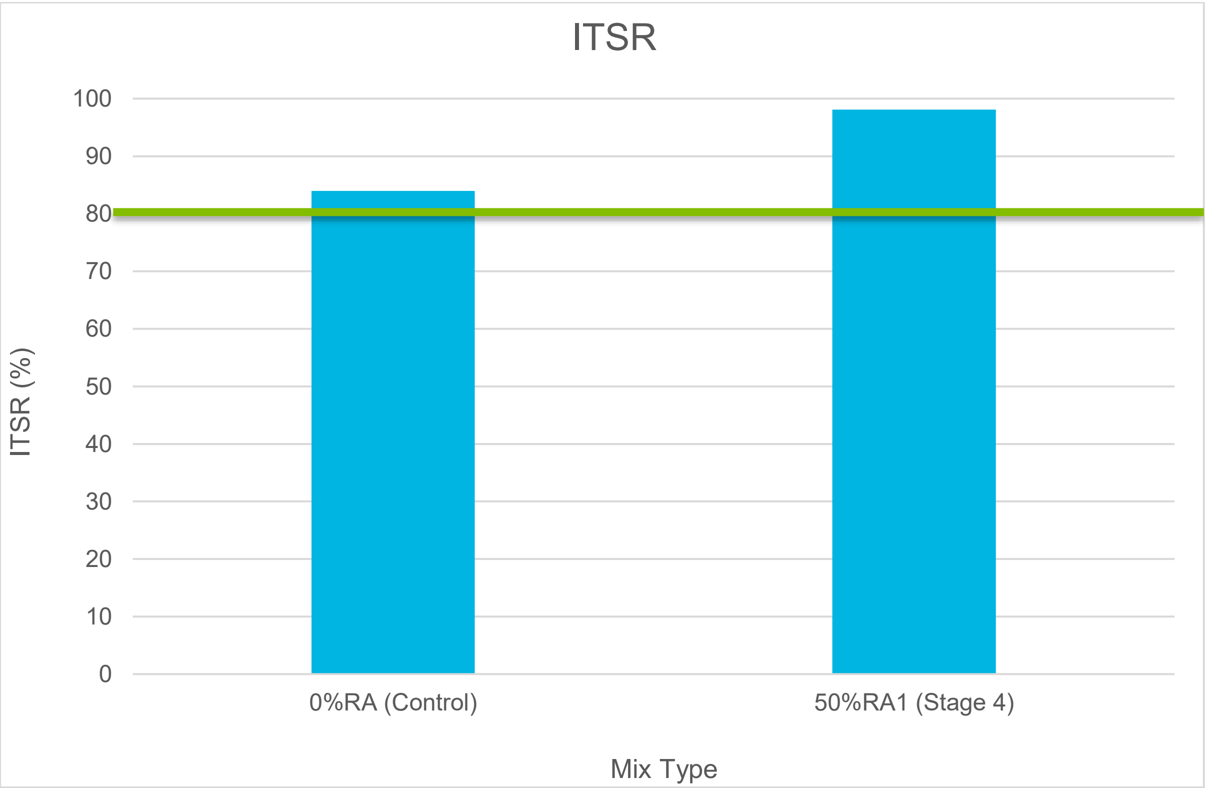


Figure 52. Indirect Tensile Strength Ratio (ITSR) of 0% RA (Control) and 50% RA1 (Stage 4)

6.4 Guidance for Assessment Framework

Given the analysis and results presented in this work, Figure 53 provides proposed guidance for the assessment of bitumen recovered from asphalt mixtures made using RA (incorporating reclaimed PMB), virgin aggregate and virgin PMB. Other key design factors and considerations when recycling asphalts into thin surfacings are provided by Road Note 43 [28], and the guidance illustrated in Figure 53 can be used in conjunction with the protocol shown in Figure 2.2 (Flow chart for design of thin surfacing system with RA) of Road Note 43. For completeness, an extract from the Road Note 43 design process for incorporating RA in thin surfacings is reproduced in Appendix B.

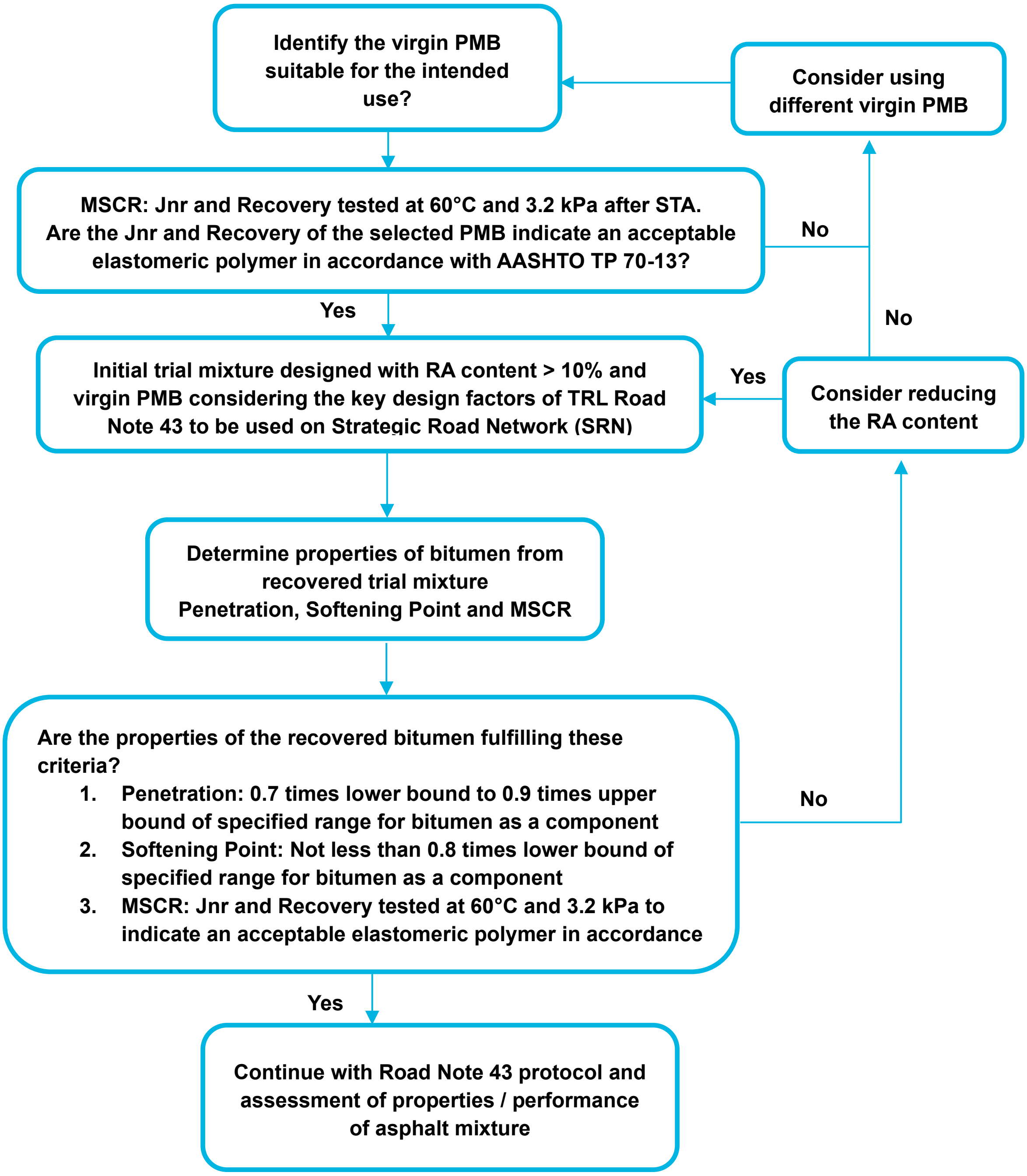


Figure 53. Guidance for recovered bitumen assessment of asphalt mixtures containing reclaimed PMB

6.5 Key Findings

The impact of multiple recycling for asphalt mixtures containing PMB was evaluated in this section. The assessments included asphalt mixture testing in addition to characterisation of the recovered bitumens from the asphalt mixtures. Given the results presented in this section, the following findings are drawn:

1. The effect of multiple recycling is generally associated with the hardening of the bitumens, reducing their ability to resist thermal and traffic-induced cracking. The bitumen recovered from the asphalt mixture containing 50% multiple recycled materials (RA1) had comparable low temperature performance to the bitumen recovered from an asphalt mixture containing 50% ‘M42’ RA.

2. The desirable rheological properties of the PMB were largely maintained after 2 cycles of recycling for asphalt mixtures containing a highly modified PMB. The bitumen recovered from the multiple recycled asphalt mixture still showed an acceptable elastomeric polymer response in accordance with AASHTO TP 70-13.

3. The addition of 50% RA1 had increased the ITSM values of the asphalt mixtures containing 0% RA1 materials by about 1.5 times.

4. The asphalt mixtures produced using 50% RA1 showed mechanical properties at least comparable to, or better than, those of the control mixtures when tested for rutting resistance and water sensitivity in the laboratory.

7. Conclusions and Recommendations

The work outlined in this report was a collaborative project between Highways England (HE), Mineral Products Association (MPA) and Eurobitume UK. The overall objective of this project was to improve the assessment of Reclaimed Asphalt (RA) materials and end-of-life assessment for mixtures containing RA, specifically RA containing Polymer Modified Bitumen (PMB) and into mixtures containing PMB. The methodology of this work comprises four main stages involving an extensive laboratory investigation into PMBs and RAs in addition to their asphalt mixtures.

The objectives of this research have been delivered through four work stages:

- Objective 1: Identify the properties of the PMB in RA; taking into consideration the age, polymer content, RA composition, the rheological and mechanical characteristics of RA.
- Objective 2: Develop a method for recovery of PMBs from asphalt samples and a protocol for blending RA with added new PMBs.
- Objective 3: Assess the physical and chemical properties of recovered bitumens blended with PMBs, taking into consideration the RA content, type/grade of a new bitumen and long-term ageing potential.
- Objective 4: Evaluate the impact of using a high content of RA into asphalt mixtures, taking into consideration the effect of ageing and moisture-induced damage.
- Objective 5: Assess the effect of multiple recycling on the mechanical properties of asphalt mixtures containing RA including the effect on the rheological properties of the recovered bitumens.

The main findings and conclusions are presented below. Given the results and analysis presented in this work:

1. The methodology developed for this project introduced effective and efficient scientific protocols to distinguish between the rheological and chemical properties of different RA containing PMBs. The PMB recovery process developed during this project (extracting the bitumens from asphalt mixtures) was shown to have minimal impact on the crosslinked polymer network of the PMB. The Modified Rolling Thin Film Oven Test (MRTFOT) in accordance with MCHW Clause 955 provided a feasible protocol for blending bitumen recovered from RA with virgin bitumens, whilst at the same time providing a means to accelerate ageing in the laboratory.

2. The results of bitumen characterisation highlighted the importance of using more fundamental and performance related testing to characterise the PMBs. The DMA and MSCR analysis have provided effective tools to distinguish between the level of polymer modification of different PMBs. The addition of up to 50% of recovered RA bitumen to a highly modified PMB can still result in having blended bitumen with “acceptable” elastic recovery properties, in accordance with AASHTO TP 70-13.

3. The G-R parameter derived from the DMA testing at low temperature (0°C) and SARA analysis provided consistent results about the combined effect of ageing and incorporating RAs on the cracking resistance of bitumens. The G-R parameter and the SARA analysis have shown that the combined effect of ageing and incorporating RAs can make materials more susceptible to thermal and induced cracking. The addition of 25% RA had, however, comparable performance to the control materials (100% virgin PMB) in terms of low temperature properties.

4. Assuming up to 90% availability of the bitumen in the RA was found to be appropriate to meet the required volumetric, mechanical properties and compactibility of asphalt mixtures incorporating up to 50% RA.

5. The addition of 50% RA materials can increase the stiffness of asphalt mixtures by a factor of 1.5 to 2 depending on the properties of RA materials.

6. The asphalt mixtures produced using 50% RA showed comparable or enhanced mechanical properties to those of the mixtures containing 0% RA, when tested for rutting resistance and water sensitivity.

7. The desirable rheological properties of the PMB were largely maintained after multiple (second round) recycling for asphalt mixtures containing a highly modified PMB. The re-recycled asphalt mixtures produced (using 50% lab-aged RA) showed mechanical properties at least comparable to those of the control mixtures containing 0% RA, when tested for rutting resistance and water sensitivity.

The following recommendations are proposed to obtain a better understanding of RA materials containing PMBs, particularly when used on large scale production and installation, and to validate the laboratory test results:

- The laboratory-based findings from this work show the probable feasibility of using high levels of RA containing PMBs (up to 50% RA), and into mixtures containing PMBs. It is strongly recommended to extend this work to full- scale asphalt plant and road trials.

- The performance-related characteristics for asphalt mixtures in this study were limited to rutting and water sensitivity. Further work incorporating long-term durability assessments, including evaluating the low temperature cracking characteristics, is recommended.

- Detailed analyses of the whole-life cost benefit from using a highly modified PMB is also recommended to promote longevity and assess the sustainability of the recycled asphalt mixtures.

Acknowledgements

The work detailed in this report was undertaken by AECOM's Pavements and Materials team on behalf of the Collaborative Research group comprised of Highways England, Mineral Products Association (MPA) and Eurobitume UK. The Collaborative Research group and authors are grateful to MPA and Eurobitume UK members for advising on current best practice and their experience.

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Appendix A: Bitumen Recovery from Reclaimed Asphalt containing PMB

Binder recovery of bituminous materials using dichloromethane followed by rotary evaporator carried out in accordance with BS EN 12697-1 and BS EN 12697-3, modified according to this Operating Procedure. This method is based on a Dichloromethane extraction followed by centrifugation to remove any suspended solids and finally rotary evaporation to remove the solvents.

Apparatus

1. Calibrated balance
2. Bottle roller
3. Centrifuge
4. Rotary evaporator
5. Metal rolling barrel with rubber bung
6. Various glassware
7. Stopwatch
8. Thermometer
9. 0207 - FORM

Chemicals

1. Dichloromethane (DCM)
2. Paraffin Oil
3. Silica Gel (not passing a 0.063mm sieve)
4. Petroleum Jelly
5. White Spirits for cleaning

Safety

Ensure the fume cupboard is used when pouring/using DCM.
For use and disposing of chemical reagents, all relevant safety operations must be followed.

MSDS (Material Safety Data Sheet) for each substance is supplied by manufacturers
COSHH sheets are created or amended and are accessible to all Laboratory staff members.

Sample Storage and Sample Preparation

This analysis is undertaken on an ‘as received’ portion of the sample.
The ‘as received’ samples should be stored in sealed containers at least until the analysis is completed and results checked/approved.
Samples should be stored at room temperature

Sample Extraction

If the sample requires breaking down (i.e. site cores) the sample may be heated gently to a maximum of 120 ˚C for about an hour. Add the sample reference (label). Cool the sample down and break down using a pallet knife (smaller the better).

1. *Bottle Roller*
   a) Clean the sample barrel with the bottle brush provided and place on balance and zero reading.

Note - For the smaller samples (less than 1000g) use the small scale with serial number 1005. If the sample is bigger use the other scale referenced as 1583.

b) Weigh out an appropriate amount of sample, so that it will contain between 120g and 150g of recoverable binder. However, if the percentage binder is not known use at least 800g of the sample and record the mass on to 0207 – FORM.

Note: If the sample weigh less than 1000g use the small barrel (black), if the sample weigh more than 1000g use the bigger barrel (blue).

c) Carry the barrel to the fume cupboard and add DCM and sufficient silica gel* to absorb any water and firmly insert the rubber bung. (It has been found by AECOM operatives that to yield sufficient test sample, 800g of asphalt to 400ml of DCM is required).

*Note: Silica gel is not always required as most of our samples are pre-heated up to 120°C

d) Shake the barrel well away from the face, leave to stand for an initial 2-3 minutes, release the bung and repeat until all residual pressure is released.
e) Place the barrel on the bottle roller and turn on for 60± 5 minutes and verify that the binder is well dissolved.
f) Allow the bitumen solution to stand for a minimum of 10 minutes.
g) Aggregate remaining in the bottle after the solution is poured out for filtration should be shaken with a further quantity of solvent. This process is repeated until no discolouration of the solvent is visible and the washings are visibly free from material in suspension. This usually takes between 3 to 4 cycles.
h) Slowly decant the liquid phase into a suitable container/flask (multiple containers may be needed depending on the size of the sample)

Note: Binder must not exceed 24 hours in a solvent solution.

2. *Centrifugation*
   a) Turn on the centrifuge and set the flow so the handle points out at 90°.
   b) Place a collection vessel under the outflow tube of the centrifuge.
   c) Slowly pour the extracts into the top sieve and check flow, increase if needed by turning handle to the left.
   d) Ensure the collection vessel does not overflow, replace if necessary. Outflow should not exceed more than 150ml/min and must be monitored by the test operator.
   e) Transfer the extract into a suitable glass container.

3. *Rotary Evaporation*

Note: Temperatures and pressures can be found in table 1.

a) Turn on the rotary evaporator and set the rotation to 75 ± 15 r/min and the temperature to T1 ± 5˚C ensuring this is checked against the calibrated thermometer.
b) Turn on the pump and cold water tap in the sink to ensure that there is cold water flowing through the condensation column.
c) Start the rotation and heater.
d) Turn the black tap on the pump (beside the inlet) to reduce the pressure in the apparatus to P1 ± 5kPa
e) Transfer the extract from the container to a conical flask in the fume cupboard and securely attach bung and place conical flask on a tripod.

f) Remove tripod and lower the evaporating flask into the oil bath.

Note: Do not lower sample into the oil bath if temperature is above 85˚C (set evaporator to 88/89˚C)

g) Open the induction stopcock clockwise to allow the bitumen solution to be drawn from the conical flask into the evaporating flask.
h) Use the stopcock to adjust the flow of the bitumen to a constant flow, such that a constant flow of droplets entering the evaporating flask.

Note: Do not allow the volume of bitumen solution in the evaporating flask to exceed 400ml or the pressure to be lower than 80kPa.

i) When all the bitumen solution has been transferred into the evaporating flask, raise the temperature of the oil bath to T2 ± 5˚C (set to 155˚C to reach 150˚C - ensuring this is checked against the calibrated thermometer).
j) Isolate the vacuum pump by turning the black tap on the side of the pump, allow the pressure to gradually increase to atmospheric pressure or to pressure P2 (2kPa ± 0.5), over a period of 3 min ± 30 s.
k) For soft bitumen's, it is advised not to exceed 120ºC for T2 regardless of the solvent used.
l) If the bitumen appears to be bubbling after 10 minutes, raise the temperature to T3 ± 5˚C (175°C - ensuring this is checked against the calibrated thermometer) and continue to evaporate until the bubbling ceases.
m) For soft bitumen's, it is advised not to exceed 120ºC for T3 regardless of the solvent used.
n) Raise the flask from the oil bath and wipe of excess oil from the surface of the flask (caution, the flask will be hot).
o) Stop the rotation and vacuum pump and release the pressure from the auxiliary tap.
p) Remove the flask and clean inside of neck with a clean tissue; gently move the liquid in a circular motion before pouring the contents into a penetration tin.
q) Cover and allow the sample to cool to room temperature.

Table 1:

		Phase 1		Phase 2		Extra
Solvent	Boiling point (˚C)	Temperature T_1 (˚C)	Pressure P_1 kPa	Temperature T_2 (˚C)	Pressure P_2 kPa	Temperature T_3 (˚C)
Dichloromethane	40.0	85	85	150	2.0	175

Note: Complete process from start to finish must be completed within 20 hours.

It is advisable to keep the vacuum pump running for about 30 minutes after completion of recovery to ensure the removal of any dichloromethane (or other suitable solvent) vapour from the oil.

Cleaning Equipment

Manually clean neck of round bottomed flask within the fume cabinet with a hot knife, and then DCM or white spirits using a clean cloth.

To clean the Evaporator machine, place the used but cleaned round bottomed flask back as before, turn on the pump and reduce the pressure. Draw the solvent from the beaker into the flask by opening the stopcock - this will clean the in-flow tube.

Then turn off the pump and vent the pressure by turning the gas port.
Clean the sample barrel by adding 200-300ml of DCM. Roll for 5-10 minutes.
Clean the round bottomed flask again within the fume cabinet with DCM/White Spirits.
The Centrifuge can be disassembled, parts cleaned in the fume cabinet with DCM/White Spirits and reassembled.

Environmental Considerations

The equipment used is not affected by changes in temperature or humidity, therefore environmental control is only necessary for operator comfort and to ensure that volumetric changes to glassware and aqueous solutions are kept to a minimum. A target temperature range is 20 ± 5° C.

Waste Disposal

Excess samples are stored on site for a suitable period to allow for re-analysis if necessary. Samples for disposal and solid waste are collected in a skip and removed by a specialist carrier.

Excess and waste solvents are separated into chlorinated and non-chlorinated solvents. They are collected in suitably labelled containers in the laboratory. These containers are emptied into the appropriate waste safe located in the compound at the side the building. A specialist company collects the waste on request.

Appendix B: Road Note 43 Flow Chart for Incorporating RA

Source: I. Carswell, J.C. Nicholls, I. Widyatmoko, J. Harris, R. Taylor, Best practice guide for recycling into surface course, Road Note 43, IHS, 2010.

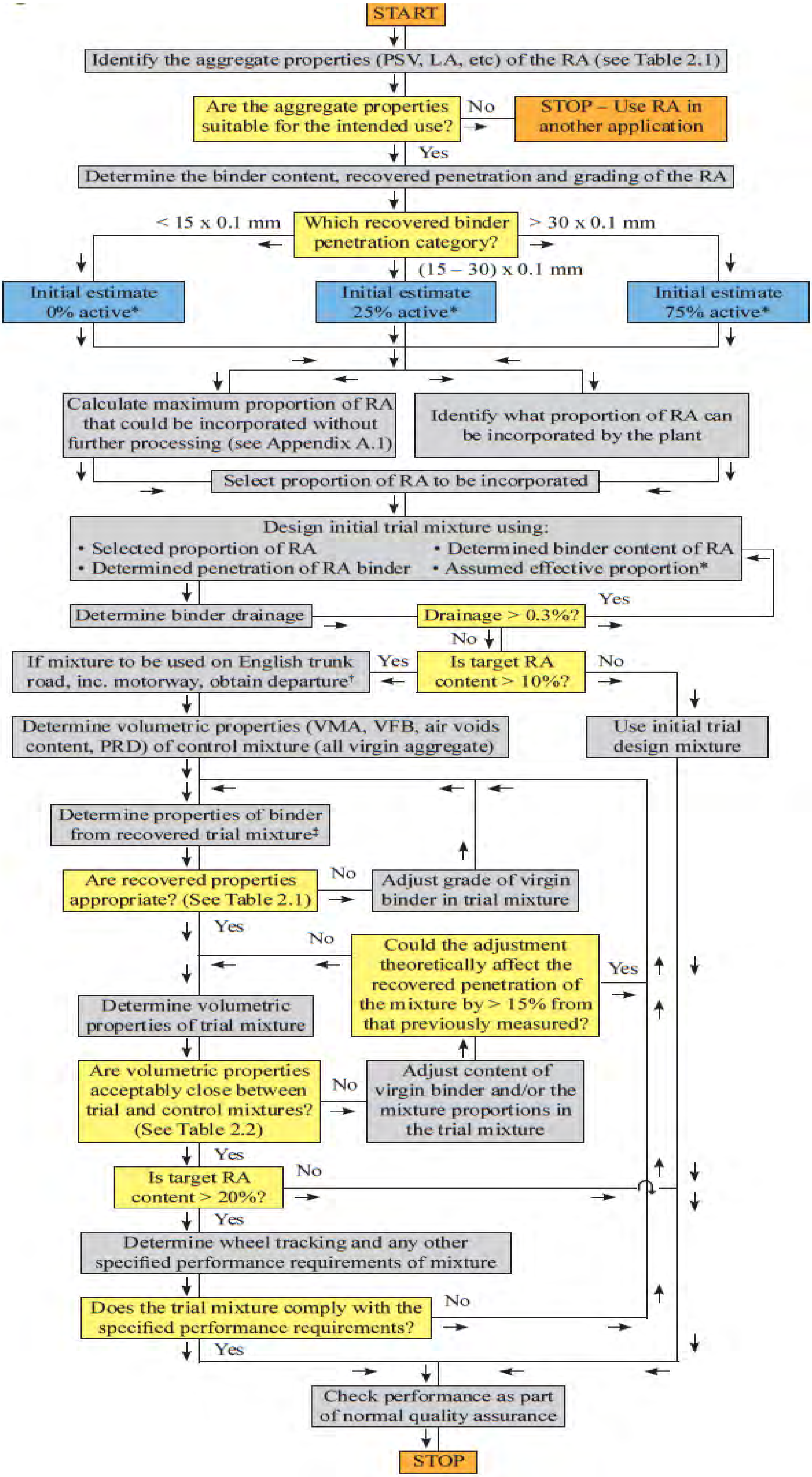

Figure 2.2 Design Factors for Incorporating RA (Carswell et al., 2010 - TRL Road Note 43)

Appendix C: Laboratory Results

Files related to the test results of this study can be requested from Dr Ayad Subhy or Dr Iswandaru Widyatmoko by emails at:

ayad.subhy@aecom.com or daru.widyatmoko@aecom.com

The files include the following results:

1. The DSR raw data (Frequency sweep and MSCR results)
2. Penetration and Softening Point results
3. FTIR results
4. SARA results
5. Compositional Analysis results
6. ITSM results
7. Water Sensitivity results
8. WTT results
9. Volumetric Properties of Asphalt Mixtures (Bulk Density, Max Density and Air Voids) results